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INFLUENCE DES FRACTIONS DE MOUTURE DE BLÉ TENDRE (FARINES PATENTE, DE-COUPURE ET BASSE) SUR LES PROPRIÉTÉS RHÉOLOGIQUES DES PÂTES ET CARACTÉRISTIQUES DES BISCUITS

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Résumé

Pour tenir compte de la variation de la valeur technologique des farines de blés tendres pour la biscuiterie, les critères physico-chimiques liés à la composition des farines et les paramètres rhéologiques permettant de prévoir leur valeur biscuitière ont été évalués. Des courants de mouture référés comme les farines patente, de coupure et farines basses recueillies à partir d'un diagramme de mouture industriel ont été étudiés individuellement ou en combinaison, afin déterminer leur impact sur le comportement rhéologique des pâtes et caractéristiques des biscuits secs « semi-sucrés», de type Petit beurre et aussi sur des cookies typiques des procédés coupe-fil et à la rotative.

Une procédure de fractionnement/reconstitution a été développée afin d'accéder à la contribution des fractions gluten, amidon de refus, amidon principal et fraction hydrosolubles incorporées en différentes concentrations, dans des « farines modèles», à l'aide d'un plan de mélange, sur les attributs rhéologiques des pâtes (viscoélasticité, consistance, viscosité élongationnelle, dureté, intensité de cohésion), dimensionnelles et texturales des biscuits secs (densité, fermeté, résistance aux bris, friabilité) des biscuits. Ces données ont permis de déterminer par quel mécanisme la variation de la concentration des polymères protéines, pentosanes et amidon ainsi que les lipides explique celles des attributs rhéologiques des pâtes et caractéristiques des biscuits.

Une bonne compréhension des effets quantitatifs et qualitatifs des constituants endogènes aux fractions gluten, amidon et hydrosolubles permet de manipuler les caractéristiques des biscuits, et cela sans modifier les proportions de sucres et gras d'une recette biscuitière. La fortification d'une farine commerciale problématique à l'usine (pâte collante, biscuits aux dimensions démesurées et/ou trop fragiles avec des surfaces rugueuses) avec les fractions isolées de ces courants de mouture permet de pallier à certains de ces problèmes.

Abstract

To take account of variations in the technological value of soft wheat flours used in biscuit making, we identified and evaluated physicochemical criteria linked to flour composition, as well as rheological parameters allowing the value of flours in biscuit making to be predicted. The patent, middle-cut and low-grade milling streams, obtained from an industrial milling flow, were studied to determine their impact on the rheological behaviour of biscuit doughs and the characteristics of dry, semi-sweet biscuits (*petit beurre* biscuit type) and typical wire-cut and rotary-molded cookies. A fractionation and reconstitution process was developed to determine the role of gluten, starch tailings, prime starch and soluble fractions on the rheological characteristics of doughs (viscoelasticity, consistency, elongational viscosity, hardness and cohesion) and the dimensional and textural attributes of dry biscuits (density, hardness, resistance to breakage and friability). These fractions were incorporated into flour in different concentrations to yield « flour's models systems» using a mixing plan design. The data obtained allowed us to determine the mechanisms by which the variations in concentrations of protein, pentosans and starches macropolymers, explain the variations in the rheological attributes of doughs and products characteristics.

The understanding of the quantitative and qualitative effects of the constituents of glutens, starches and the hydrosolubles fraction allows biscuit characteristics to be manipulated, without modifying the proportions of sugar and fat in biscuit recipes. The fortification of problematic commercial flours (difficulties encountered include sticky dough and biscuits that are brittle or with inappropriate dimensions and top grain) with fractions isolated from the mill streams can provide a solution to some of these problems.

Résumé long

Des courants de mouture industrielle (les farines patentes, de coupure basse) ont été étudiés, individuellement ou en combinaison, pour déterminer leur impact sur le comportement rhéologique des pâtes et propriétés dimensionnelles et texturales des biscuits secs et des cookies à la rotative et coupe fil.

L'ajout de la fraction de farine basse, portion périphérique de l'endosperme, dans les trois recettes biscuitière et cookies provoque une augmentation de la consistance de la pâte et une forte augmentation de la fermeté et densité des biscuits. Par contre, on observe une corrélation inverse avec ces mêmes attributs avec l'utilisation de la patente, portion du cœur de l'endosperme. Toute combinaison de courants de mouture, induisant de larges variations dans la composition minérale, polysacharrides non amylacés et protéique, provoque des changements majeurs sur les caractéristiques des pâtes et produits finis. L'étude des régressions « PLS» décrit bien les relations qui prévalent entre les farines individuelles et recombinées ainsi que leurs interactions sur les attributs rhéologiques des pâtes et caractéristiques des produits, selon la contribution des ingrédients des recettes. De plus, certains paramètres physico-chimiques des farines (protéine, granulométrie et valeurs alvéographiques) permettent d'expliquer la consistance de pâtes.

Une procédure de fractionnement et de reconstitution des farine a été validée sur ces trois courants de mouture pour accéder à la contribution des fractions gluten, amidon de refus, principal et hydrosolubles incorporées, dans les mêmes proportions que les farines originales correspondantes. Cette procédure a permis de produire des biscuits secs, aux surfaces lisses et longueurs très similaires aux biscuits des farines originales équivalentes. Cependant, une diminution assez substantielle des attributs viscosité élongationnelle, consistance et vitesse de relaxation a été notée sur les pâtes reconstituées, suggérant que d'autres facteurs peuvent aussi intervenir dans la mise en forme des biscuits secs. De plus, la procédure favorise le brunissement des biscuits secs.

Un plan de mélange utilisant les fractions gluten, amidon et hydrosoluble isolées des trois courants de mouture a permis d'induire des variations majeures dans la composition

biochimique de « farines modèles». Cette étude a démontré que la fonctionnalité des farines de blé ne peut être expliquée par la simple sommation des effets individuels de ces fractions. En effet, même si chaque fraction gluten, amidon et fraction soluble possède des caractéristiques biochimiques spécifiques qui varient selon le courant de mouture, la valeur technologique de la farine dépend essentiellement des relations structure/fonction des protéines, pentosanes, amidon et lipides, de leur concentration et aussi des nombreuses interactions qui prévalent entre eux. Des modèles de régression et les diagrammes de contour ont permis de déterminer comment ces interactions qui existent entre le gluten, amidon et fraction hydrosoluble altèrent les caractéristiques des pâtes et des biscuits secs.

La consistance, viscosité élongationnelle et dureté des pâtes produites par la farine basse, étaient surtout expliquées par les pentosanes (corrélation négative). Par contre, ces mêmes attributs rhéologiques démontraient une corrélation positive avec les protéines de la farine patente. La qualité et non la quantité des protéines de la portion centrale de l'endosperme semblent jouer un rôle crucial sur ces attributs rhéologiques.

La fortification d'une farine industrielle problématique (pâte collante, biscuits fragiles et rugueux, aux dimensions démesurées) avec les fractions gluten, amidon de refus et principal et fraction soluble de la farine basse et patente a permis de modifier considérablement les caractéristiques des biscuits. Ainsi, toute augmentation de la teneur en gluten de la farine commerciale induit une augmentation prononcée de la consistance et viscoélasticité des pâtes, tout en minimisant les problèmes de fêle des biscuits. Les niveaux d'addition des fractions solubles dans la farine commerciale ont été contrôlés (< 5.0%), à cause de leur contribution au collant de la pâte. A cause de sa teneur en pentosanes, cette fraction exerce un effet notable sur la fermeté des biscuits. L'amidon principal ne modifiait ni les attributs rhéologiques ni les caractéristiques des biscuits de la farine industrielle. En revanche, à la même concentration d'amidon de refus, une augmentation excessive de la dureté et consistance des pâtes, et fermeté et résistance aux bris des biscuits a été observée. L'utilisation de cette fraction est, de loin, la plus déterminante pour modifier la qualité des biscuits à cause de sa teneur en pentosanes.

Long abstract

Three mill streams (patent, middle-cut and low-grade flours) obtained from the industrial milling flow were studied, alone and in combinations, to determine their impact on the rheological behaviour of doughs and the dimensional and textural properties of dry, semi semi-sweet biscuits (*petit beurre* or butter biscuit type) and wire-cut and rotary-molded cookies

The addition of the low-grade fraction (which comes from the peripheral portion of the endosperm) in the three recipes resulted in greater dough consistency and an increase in the finished products' firmness and density. When patent flour (which comes from the heart of the endosperm) was used, an inverse correlation was found for the same attributes. The use of patent flour resulted in softer, stickier doughs with lower consistency but longer biscuits with a crispy texture and a tendency towards breakage. Various combinations of these fractions, which result in significant variations in the mineral, pentosan and protein content of the flour, produced major differences in the characteristics of both doughs and finished products. The PLS regressions provides a good description of the relations and interactions between individual and recombined flours as well as their effects on the characteristics of doughs and finished products, owing to the contribution of recipe ingredients such as fat and sugars. Certains physico-chemical flour's parameters (protein, alveograph values, particle size) allow the prediction of dough's consistency.

A fractionation and reconstitution procedure was validated using the three mill streams to determine the contributions of the gluten, starch tailings, prime starch and soluble fractions, which were incorporated in the same proportions as in the corresponding original flours. This procedure allowed the dry biscuits to be produced with a smooth surface and lengths very similar to those in biscuits made from the equivalent parent flours. However, a fairly substantial decrease in elongational viscosity, consistency and relaxation rate attributes was found in reconstituted doughs, suggesting that other factors may also play a role in setting in dry biscuits. Moreover, this procedure favours the browning reactions.

A mixing plan using isolated fractions of the flours resulting from the various mill streams was used to produce major variations in the biochemical composition of «model flours» in order to more accurately determine the "raw material" effect. The study showed that wheat flour functionality cannot be explained simply by the summation of these components' individual effects. Even if the gluten, starch and soluble fractions each have specific biochemical characteristics, a flour's technological value is determined essentially by the structure–function relations of proteins, pentosans, starch and lipids, their concentrations and the many interactions occurring among them. Regression models and contour plots diagrams allowed us to elucidate how these interactions among the three isolated fractions alter the characteristics of doughs and dry biscuits. The consistency, elongational viscosity and hardness of doughs made from low-grade flours can be explained mainly by pentosan content (negative correlation). A positive correlation, however, was found between these rheological parameters and proteins in patent flour; the quality rather than the quantity of the proteins in the central part of the endosperm seems to play a crucial role here

The fortification of a problematic industrial flour (difficulties encountered include overly sticky dough and rough, brittle biscuits with disproportionate dimensions) with gluten, starch tailings, prime starch and the soluble fraction of low-grade or patent flours allowed us to modify the quality of the biscuits. Increased gluten content resulted in a pronounced increase in the consistency and viscoelasticity of doughs. Furthermore, given the same protein content, gluten isolated from patent flour behaved differently from that derived from low-grade flour, due to the quality of the proteins. Gluten-fortified flour produces machinable doughs and biscuits that are smooth, without apparent cracks. The addition of soluble fractions to commercial flour was kept below 5.0%, due to their contribution to increased dough stickiness. Owing to its pentosan content, this fraction has a determining effect on biscuit firmness. At concentrations of 9%, prime starch does not modify the rheological characteristics or quality of biscuits made from industrial flour. On the other hand, increasing the content of starch tailings, which are rich in pentosans, resulted in an unacceptable increase in dough hardness and consistency (4.5 X of its initial value) and biscuit firmness (2.3 X of its initial value) and resistance to breakage. This fraction had a much more pronounced effect by far on dry biscuit quality than the gluten fraction.

Avant propos

Cette thèse compte six chapitres dont quatre sont écrits sous forme de manuscrits scientifiques qui seront publiés dans des revues scientifiques.

Le premier chapitre intitulé « Introduction Générale» inclut une revue de littérature portant sur la description d'un diagramme de mouture de blé tendre et sur les critères qui expliquent la valeur technologique d'une farine pour la biscuiterie. Plusieurs procédures de fractionnement et reconstitution des farines y sont décrites et l'impact des fractions isolées comme le gluten, l'amidon principal et de refus et aussi la fraction hydrosoluble sur les caractéristiques dimensionnelles et d'apparence de surface des cookies sugar-snap sont discutés. L'impact des paramètres physico-chimiques (protéines, lipides, pentosanes et absorption d'eau, granulométrie,….) impliqués dans la fonctionnalité des farines pour les cookies et biscuits ainsi que les attributs rhéologiques d'importance pour expliquer les caractéristiques dimensionnelles et texturales des produits finis sont abordés.

Le deuxième chapitre intitulé « Influence of soft wheat flour patent, middle-cut and clear flours streams and combinations on dough rheology and cookie characteristics of three recipes» vise à appréhender la fonctionnalité des farines issues de trois courants de mouture, qui ont été mélangés dans des proportions potentiellement rencontrées dans la minoterie sur certaines propriétés rhéologiques des pâtes préparées à partir d'un procédé représentatif des cookies coupe-fil et à la rotative et les biscuits secs laminés de type « petits beurre» et sur les caractéristiques des produits finis. Cette étude nous a permis de déterminer que ces fractions de mouture, incorporées individuellement ou en combinaisons présentent de larges variations physico-chimiques qui expliquent les différences notées dans les caractéristiques des produits finis. De plus, ces dernières caractéristiques répondent différemment aux variations biochimiques, à cause de la différence à cause de la différence dans les proportions de sucre et gras des recettes qui est loin d'être négligeable.

Il ressort de cette étude que certains paramètres des produits finis peuvent être expliqués par certains paramètres physico-chimiques (granulométrie, protéines,..) et aussi par des paramètres rhéologiques (viscoélasticité). Cette étude nous a aussi permis de sélectionner la recette biscuit secs, laminés comme étant la plus sensible aux variations des courants de mouture et ainsi fournit d'avantage d'attributs prédictifs expliquant les variations de la composition de la farine comme matière première. Un poster intitulé «Influence of soft wheat fraction on cookie characteristics » couvrant une partie de ces résultats a été présenté à la réunion annuelle IFT à Dallas en 2000, poster 39C-7.

Le troisième chapitre intitulé « semi sweet biscuit making potential of soft wheat flour patent, middle-cut and clear mill streams made with native and reconstituted flours» vise à déterminer l'impact d'une procédure de fractionation/reconstitution (typique aux procédés commerciaux de glutennerie et amidonnerie) des trois courants de mouture sur plusieurs attributs rhéologiques d'une pâte et caractéristiques des biscuits secs, laminés de type-beurre. Les résultats obtenus ont permis de démontrer que la procédure de fractionnement utilisée pour isoler le gluten, l'amidon et la fraction soluble et autres sous fractions pour ensuite les reconstituer sous la forme de la farine originale produisait des biscuits aux dimensions et apparence de surface assez similaires aux farines natives correspondantes, et cela, malgré le fait que la procédure modifiait modérément certains paramètres rhéologiques. Une partie de ce travail intitulé «Les effets interactifs des constituants des patentes et farines basses sur la rhéologie des pâtes» a été présenté au AACC, Food Network à Kentville, Août 2003.

Le quatrième chapitre intitulé «Flour constituent interactions and their influence on dough rheology and quality of semi-sweet biscuits : a mixture design approach with reconstituted blends of gluten, water-solubles and starch fractions isolated from different soft wheat flour grades» a permis de quantifier les rendements des fractions amidon principal, de refus, le gluten, la fraction soluble isolées des trois courants de mouture ainsi que leurs teneurs respectives en protéines et pentosanes solubles et insolubles et lipides. En faisant varier les proportions de gluten, amidon et fraction soluble incorporées à des «farines modèles» à l'aide d'un plan de mélange, on a pu déterminer les relations structure/fonction des protéines, pentosanes, amidon et lipides sur les propriétés des pâtes et biscuits. Même si cette approche présente le désavantage que, selon certaines circonstances, des effets masquants (ex. nouvelle distribution de l'eau entre les constituants) peuvent réduire la sensibilité, surtout à cause des manipulations physiques, elle a permis d'établir l'effet de la variation de ces fractions et de leurs constituants endogènes sur les attributs rhéologiques de la pâte biscuitière et aussi sur les caractéristiques dimensionnelles et texturales des biscuits. Certains résultats de ces travaux ont été présentés par un poster au CIFST/AAFC à Guelph en 2004 «The role of gluten, starch and water soluble fractions isolated from a patent and clear flour on the dough rheological behavior» et aussi à la réunion annuelle du IFT à Las Vegas en 2004, papier 30987 intitulé « Effect of gluten, starch and water soluble fractions on dough and biscuit quality».

Le cinquième chapitre s'intitule « Incorporation of patent and clear flour streams and their isolated gluten, water soluble, prime starch and starch tailing into a commercial soft wheat flour: impact on the dough rheology and semi-sweet biscuit». Ce travail a été présenté à la réunion annuelle au IFT à New Orleans en 2005 et CIFST à Montréal en 2006. Cette étude a permis de confirmer que la substitution partielle d'une farine commerciale problématique pour la production (pâte trop collante, biscuits trop fragiles et apparence rugueuse) par des fractions de mouture natives modifie le comportement rhéologique des pâtes en les rendant plus machinables et aussi améliore les dimensions et propriétés mécaniques des biscuits. L'addition de l'amidon de refus isolés de ces deux courants de mouture à cette farine provoque des changements majeurs dans les attributs rhéologiques des pâtes et de plus augmente considérablement les attributs mécaniques des biscuits, à cause de leur haute teneur en pentosanes. L'incorporation du gluten quant à elle, altère considérablement les propriétés rhéologiques des pâtes tout en exerçant un effet plus restreint sur les attributs dimensionnels et texturaux des biscuits, alors que l'amidon principal et fraction solubles comparativement à la fraction gluten et amidon de refus, exercent plutôt un effet minime sur les caractéristiques des biscuits.

Finalement une « Conclusion Générale» est présentée au chapitre 6. Ce chapitre comporte une discussion globale de l'ensemble des résultats obtenus dans ce projet, leur importance pratique dans le domaine d'études et pour l'industrie et conclut avec la présentation des perspectives d'application pour l'industrie et de recherche pour des travaux futurs.

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INTRODUCTION GÉNÉRALE

Problématique

Il est reconnu depuis de nombreuses années que les farines de blé de provenances différentes se comportent différemment à la cuisson et ainsi on peut obtenir des produits finis de bonne ou de mauvaise qualité. Ces écarts sont dus à des différences qualitatives et quantitatives des constituants de la farine. Il est cependant très difficile d'établir des corrélations entre, d'une part, la composition chimique et la structure de la farine de blé et, d'autre part, ses propriétés biscuitières. Ces difficultés sont accrues par plusieurs facteurs : le grand nombre de constituants; un poids moléculaire élevé conjugué à une solubilité limitée; la difficulté d'isoler ces constituants à l'aide de solvants sans altérer leur fonctionnalité; enfin les nombreuses interactions des constituants entre eux au cours du pétrissage et cuisson.

La farine de blé tendre est une matrice biologique complexe. Cette complexité s'explique par les nombreuses variétés de blés homologuées pour l'utilisation en biscuiterie. Chaque variété possède des caractéristiques qui varient en fonction des conditions climatiques, des lieux de récolte, de l'ajout d'engrais. De plus, la composition de certains constituants de la farine tels l'amidon, protéines et pentosanes peut dépendre des caractéristiques héréditaires du cultivar et aussi du degré de mouture du grain. Ces constituants, en plus de leur fonctionnalité propre, entrent en interaction de manière très complexe et altèrent à différents degrés l'aptitude de la farine à la biscuiterie. Les caractéristiques de la farine sont donc la résultante d'une multitude de facteurs qui interagissent. L'utilisateur les subit et il doit adapter son procédé à la qualité de la farine qu'il reçoit.

Qualité de la farine est synonyme de valeur technologique. La valeur technologique se juge d'après son aptitude à donner une pâte machinable, c'est à dire une pâte qui ne doit pas coller, mais doit résister à un certain degré de brisure et pouvoir s'étendre en couches minces sans se briser, sans craqueler à la surface, ni se rétrécir, ni se crêper et aussi donner un biscuit de qualité. Qualité du biscuit, quant à elle, se traduit par une maîtrise rigoureuse des caractéristiques physiques (dimensions, couleur, humidité), apparence de la surface et de la texture (densité, dureté, résistance aux bris)

Les industries de la biscuiterie notent des disparités dans la fonctionnalité des farines de blé tendre selon les journées de production, le type de procédés et de biscuits. Ces disparités peuvent être causées par de nombreux facteurs inhérents à la farine ou externes qui demeurent difficiles à appréhender ; mentionnons, une farine de qualité industrielle médiocre, un entreposage inadéquat de la farine, un problème de juxtaposition de la farine dans le silo, un pétrissage inadéquat, un temps de repos de la pâte trop prolongé, des conditions hygrométriques durant le pétrissage inappropriées, etc....

Quelque soit la ou les causes des problèmes rencontrés durant la production, cette variabilité est bien réelle. Elle se répercute sur la qualité des biscuits. Parmi les fluctuations qui peuvent exister durant la fabrication des biscuits, mentionnons des pâtes biscuitières qui collent à la découpeuse et au moule ou sont parfois trop consistantes, une variation non contrôlée de l'épaisseur entraînant un nombre moyen de biscuits inférieurs ou supérieur à la norme, des écarts dimensionnels trop prononcés pouvant engendrer un arrêt de production, une variation dans la texture entraînant parfois leur fragilisation « la fêle des biscuits ». Les biscuitiers se doivent donc d'obtenir des farines performantes et conformes à des normes physico-chimiques pour assurer le succès de leur production et la qualité du produit fini.

Il est reconnu que les fiches techniques des minotiers qui incluent les critères teneur en protéines et cendres des farines et valeurs alvéographiques sur de simples pâtes ne suffisent pas à caractériser les farines et prédire leur comportement durant la fabrication des biscuits, ces 3 critères sont donc souvent remis en cause par la profession, quant à leur adéquation avec la qualité des biscuits. Quant au bilan des tests du farinographe et amylographe et test biscuitier sugar-snap AACC 10-52, fourni par les rapports annuels de la Commission Canadienne des Grains (CCG) sur les farines de blé tendre de l'Est versus Ouest, l'expérience industrielle démontre que ce bilan ne permet pas, lui non plus, de prédire le comportement technologique des farines pour la biscuiterie. D'autres critères susceptibles d'influencer les caractéristiques dimensionnelles et texturales des biscuits telles la granulométrie, la teneur en pentosanes, la proportion glutenine/gliadine, la capacité d'absorption d'eau et autres ne sont nullement adressés dans le cahier de charges de la CCG. Reste, finalement, le test biscuitier d'une recette usine, le seul vrai indicateur de la valeur technologique d'une farine pour une recette et un procédé donnés. L'entreprise ne peut se permettre de réaliser ce test à cause de la cadence accélérée de la production couplée avec la variabilité de la farine d'un silo à l'autre et d'une journée à l'autre ou même à l'intérieur d'un batch. Le biscuitier expérimenté ajuste alors objectivement la consistance de sa pâte ou subjectivement, à l'aide d'une unité de mesure placée sur une opération en continue pour satisfaire les besoins de la production et prier pour que les produits biscuitiers, crackers et biscottes répondent aux normes dimensionnelles et texturales souhaitées.

Le système biscuit est plutôt complexe. Ceci fait en sorte qu'il est difficile d'évaluer quelles composantes de la farine altère sa fonctionnalité. Même si les pâtes biscuitières sont découpées de la même taille, les dimensions finales des biscuits diffèrent après cuisson pour un même lot de farine. Toutes les étapes de fabrication incluant pétrissage, temps de repos de la pâte, cuisson interviennent donc dans la qualité du produit fini. Les protéines du blé sont principalement responsables des différences de la qualité des produits de panification; la quantité et qualité des protéines étant en relation avec le volume du pain. Cependant avec des recettes biscuitières contenant de fortes teneurs en sucres et gras et peu d'eau, l'effet des protéines et autres constituants tels les pentosans , lipides inhérents à la farine demeure difficile à appréhender. Cela s'explique aussi par les résultats contradictoires rapportés dans la littérature sur le rôle de ces constituants pour la biscuiterie; et de plus, certains travaux réalisés sur les produits de panification servent encore de références pour expliquer leur fonction. A cet égard, mentionnons aussi que la plupart des résultats publiés ont été acquis avec des anciennes normes AACC 10-50 pour le «sugar-snap» cookie et AACC10-53 pour le «wire-cut» cookie, riches en sucre et gras. L'emploi ultérieur dans le texte du terme cookie fait implicitement références à ces recettes, sauf mention particulière pour le terme biscuit qui réfère essentiellement, dans notre travail, aux biscuit secs (semi-sucrés de type petits beurre).

Pour atteindre l'objectif performance et conformité, le biscuitier dispose de trois solutions bien distinctes : la première approche consiste à modifier l'aptitude de la farine de blé tendre par adjonction d'autre farines, comme par exemple, la farine de soya, de blé dur. Cette approche permet de pallier à certains problèmes de l'usine (le collant de la pâte, consistance trop ou pas assez élevée, dimensions et fêle des biscuits inappropriées, etc.).Une approche qui est souvent privilégiée par l'industriel. Il est important de souligner, à cet effet, que l'usage des courants de mouture comme substitution ou fortification d'une farine commerciale pour pallier à ces problèmes demeure encore peu répandu, à cause du manque de connaissance des propriétés qu'elle confère aux produits biscuitiers. La seconde approche correspond à l'adjonction d'additifs chimiques (agents réducteurs et oxydants) et enzymatiques (comme la papaïne, pentosanase) pour modifier les propriétés viscoélastiques des pâtes biscuitières. Cette solution est loin d'être pratique lorsque la production s'effectue sur des lignes de grande cadence; elle entraîne des coûts additionnels et, elle est loin de satisfaire un consommateur soucieux de sa santé et de plus, l'efficacité de cette approche reste encore à démontrer dans le secteur biscuitier. La troisième approche consiste à agir, en amont, par une meilleure sélection de la matière première, la farine. Une meilleure connaissance de celle-ci devrait permettre de pallier possiblement aux inconvénients mentionnés précédemment. Cette approche constitue un défi réel pour l'industrie soucieuse d'améliorer la qualité et l'image de son produit. Dans ce cas, on réfère à son aptitude, donc aux constituants intrinsèques: l'amidon, les protéines, les pentosanes et lipides qui peuvent jouer un impact majeur sur la qualité du produit.

Tout procédé de transformation correspond à un changement d'état, physique et/ou chimique, de la matière. Dans le cas de molécules à poids moléculaire élevé, telles les biopolymères (amidon, pentosans, gluten), l'aptitude technologique du mélange de départ dépend des interactions physico-chimiques (covalentes, hydrophobes, hydrogène, Van der walls) qui existent entre les constituants. Il s'agit d'une physico-chimie très complexe, que les moyens analytiques actuels ne permettent pas de décrire avec précision. Le procédé biscuitier n'échappe pas à cette règle. La farine et les ingrédients de la recette sont mélangés en phase aqueuse concentrée; le mélange obtenu subit un traitement mécanique précis qui détermine l'arrangement conformationnel des biopolymères et par conséquent, le comportement rhéologique de la pâte. Ainsi la connaissance de ce comportement peut servir à mesurer l'aptitude technologique de la farine et permettre d'affiner la sélection de variétés de blé adaptées au procédé, le choix de leur mélange éventuel, ou la mise au point de formules prévisionnelles. Les approches physico-chimiques et rhéologiques constituent des voies privilégiées pour comprendre, ou du moins prédire la variabilité de la valeur biscuitière des farines.

Il y a donc un défi technologique intéressant qui ouvre la voie à de nouvelles recherches. C'est donc dans ce contexte que nous avons réalisé notre étude qui s'inscrit en amont du procédé de fabrication. Il s'agit précisément d'identifier les critères analytiques susceptibles de prédire les caractéristiques dimensionnelles et texturales des biscuits. Pour cela nous nous intéresserons plus particulièrement aux propriétés rhéologiques des pâtes biscuitières fabriquées à partir de trois courants de mouture d'un diagramme de mouture industriel, soit les fractions de mouture appelées les farines patente, de coupure et basse (référées comme patente, middle-cut et clear flour dans la terminologie anglo-saxonne) lesquelles lorsque mélangées dans des proportions bien définies constituent la farine commerciale ou «straight grade flour».

General Problem

It has been known for many years that wheat flours of different origins behave differently when baked, resulting in finished products of varying quality. These variations are caused by qualitative and quantitative differences in flour components. It is very difficult to establish correlations between the chemical composition and structure of wheat flour, on one hand, and its biscuit-making properties, on the other. These difficulties are explained by a number of factors: the large number of components involved their high molecular weight and limited solubility, the difficulty of isolating these components with solvents without altering functionality, and, lastly, the numerous interactions between components during mixing and baking.

Soft wheat flour is a complex biological matrix. The complexity is reflected by the many licensed flour varieties used in biscuit making. Each variety has characteristics that maqy vary depending on climatic conditions, geographical origin of harvest and the use of fertilizer inputs. In addition, the composition and properties of certain flour components such as starch, proteins and pentosans may depend on the genetic characteristics of the cultivar and the degree of grain's milling. These components not only possess their own functionality but also enter into very complex interactions with one another, altering to varying degrees the flour's potential in biscuit making. Flour characteristics are therefore the result of a multitude of interacting factors. Users, who are accustomed to this, must adapt their process to the quality of the flour received.

Flour quality is synonymous of flour's technological value, which is determined by its ability to provide a machinable dough, in other words, a dough that can withstand a certain degree of breakage and that can be stretched into thin layers without breaking, shrinking (retraction), and then can produce a high-quality biscuit. Biscuit quality entails strict adherence to a rigorous set of physical characteristics (dimensions, density, colour and moisture), surface appearance and texture.

The biscuit and/or cookie manufacturing industry has observed numerous differences in the functionality of soft wheat flours depending on the production date, process used and the type of end-product. These differences may be caused by numerous factors inherent in the flour or external in nature, which remain difficult to determine; such factors could include mediocre-quality commercial flour, inadequate storage, different flours being juxtaposed in the silo, inadequate mixing, excessive resting time for dough and inappropriate relative humidity and temperature conditions during mixing. Whatever the cause or causes of the problems encountered during production, such variability is real and has an effect on overall biscuit quality. Fluctuations that may occur during biscuit manufacturing include uncontrolled variations in biscuit thickness and density, variations in texture sometimes resulting in brittleness (cracking of biscuits) and biscuit doughs that stick to the cutting machine or rotary-moulded or that are sometimes too consistent. Biscuit manufacturers must therefore obtain high-performance flours that meet physicochemical standards to ensure production success and the quality of the finished product.

It is well known that millers' flour specifications, which include criteria such as protein and ash content and alveograph values, are not adequate to accurately characterize flours and predict their behaviour during biscuit-making. Indeed, people in the industry often question the value of these three criteria in assessing the overall biscuit quality in commercial plant production. Furthermore, industry experience shows that the farinograph and amylograph test results provided in the annual reports of the Canadian Grain Commission (CGC) on canadian soft wheat flours from East to West do not allow predictions to be made on flours' behaviour in biscuit making. Other criteria likely to influence the size and textural characteristics of biscuits such as the particle size distribution, pentosan content, glutenin-gliadin ratio, water-absorbing capacity and others tests remain unaddressed in the CGC's specifications. Finally, the biscuit test, using an industry recipe, is the only true indicator of the technological value of a given flour. Firms are not able to carry out this test, however, because of the time constraints of accelerated production and the variability of flour from day to day, from silo to silo and even within the same batch. To satisfy production requirements and ensure that products meet standards for the desired size and texture, experienced biscuit makers will adjust the consistency of the dough, either subjectively or objectively, using an appropriate measurement device located in a continuous operation.

Biscuit-making systems are rather complex, so that it is difficult to assess which flour components alter functionality. Even if the biscuit dough is cut-out in the same size, the final dimensions of biscuits made from the same batch of flour may vary after baking; therefore, all manufacturing stages including mixing, dough resting time and baking play a role in the quality of the finished product. Wheat proteins are reported to be mainly responsible for differences in quality of bread-making products, the quantity and quality of proteins being related to the bread volume. However, with biscuit recipes, involving high sugar and fat contents and little water, the effects of proteins and other flour components are difficult to discern.

To meet performance and uniformity objectives, biscuit makers have three very different solutions at their disposal. The first approach consists of modifying the suitability of soft wheat flour for biscuit making by mixing in other flours such as soy flour or hard wheat flour or various milling fractions (flour streams) obtained from the milling flow and adapting the manufacturing process accordingly. This allows, for example, the stickiness of the dough to be minimized and dough of the appropriate consistency to be obtained, producing a quality biscuit, an approach often favoured by manufacturers. It is important to mention that, that the use of mill streams to substitute part of the flour or fortify a problematic flour is not a current practice of the biscuits manufacturers, since the advantages to improve the quality of their products is still not understood and difficult to predict. The second approach is to add chemical additives, for example reducing and oxidizing agents and enzymes such as papaïn and pentosanase, to modify the viscoelastic properties of the biscuit dough. This is unpractical when dealing with high-speed production lines, entails additional costs and is unappealing to health-conscious consumers due to the use of non-friendly ingredients. Furthermore, the effectiveness of this approach remains unproven in biscuit manufacturing. The third approach is to intervene further upstream, through a more careful selection of the raw material, the soft wheat flour. A better knowledge of this material should allow the disadvantages mentioned earlier to be overcome. This approach presents a real challenge to an industry that wishes to improve the quality and image of its product. In this case, more accurately determining the effect of a flour involves an assessment of its potential in biscuit making and therefore its intrinsic components, starch, proteins, pentosans and lipids, and others factors which can have a major impact on product quality.

This document present a survey of current knowledge on the use of three flour grades from the different streams in a milling flow in the manufacture of three types of cookies: wire-cut, rotary-molded and laminated dry biscuits (type butter biscuits or *petit beurre* type). More specifically, the impact of these fractions on the rheological properties of doughs and the characteristics of finished products is examined. In this context, a fractionation procedure was employed to separate the starch and gluten components and soluble fraction from these three flours, which have fairly variable protein, ash, pentosan and lipid contents. Mixing of these fractions in different proportions was adopted to formulate « flour model systems » in order to quantify and qualify their effects on dough and biscuit quality attributes.

« Imagination is more important than knowledge. Knowledge is limited. Imagination encircles the world. »

Albert Einstein (1879-1955)

CHAPTER I

LITERATURE REVIEW

1-Soft wheat flour milling streams and composition

1.1- Histology of wheat

Wheat belongs to the grass family and designates two different species:

Triticum aestivum **(soft wheat)**

Triticum durum **(hard wheat)**

The wheat grain is a fruit (caryopsis) which, botanically speaking, consists only of the external part of the grain, made up of a hull consisting of several hard, dry layers protecting the seed inside (Godon, 1991).

Peeling the layers of the hull like an onion, it contains, beginning with the outside layer and moving inward: the pericarp, seed coat (testa) and hyaline layer (which is adjacent to the aleurone layer) (Figure 1.1). The hull (~ 9–14% of the grain), which is transformed into bran in the milling process, is composed mainly of cellulose and hemicellulose and also has a high proportion of mineral matter. The aleurone layer consists mainly of proteins, lipids, minerals and pentosans.

The next layer is the endosperm, which makes up 82%–85% of the dry matter of the grain is made up mainly of starch, with roughly 10% protein and traces of minerals and vitamins (Table 1.1). The endosperm consists of a matrix of proteins which surrounds the starch granules.

Lastly, the germ or embryo makes up 3% of the grain by weight, containing most of the fats in the grain but is also rich in proteins, mineral matter and pentosans. Due to its high lipid content (~15%), it can contaminate milling products and also lead to rancidity in the flour, if not properly eliminated during the milling procedure

Knowledge of the histology of the wheat grain and the industrial milling process allows a better understanding of the separation processes that occur during milling, as well as an assessment of the proportions of the various tissues in the resulting milling fractions (Table1.2).

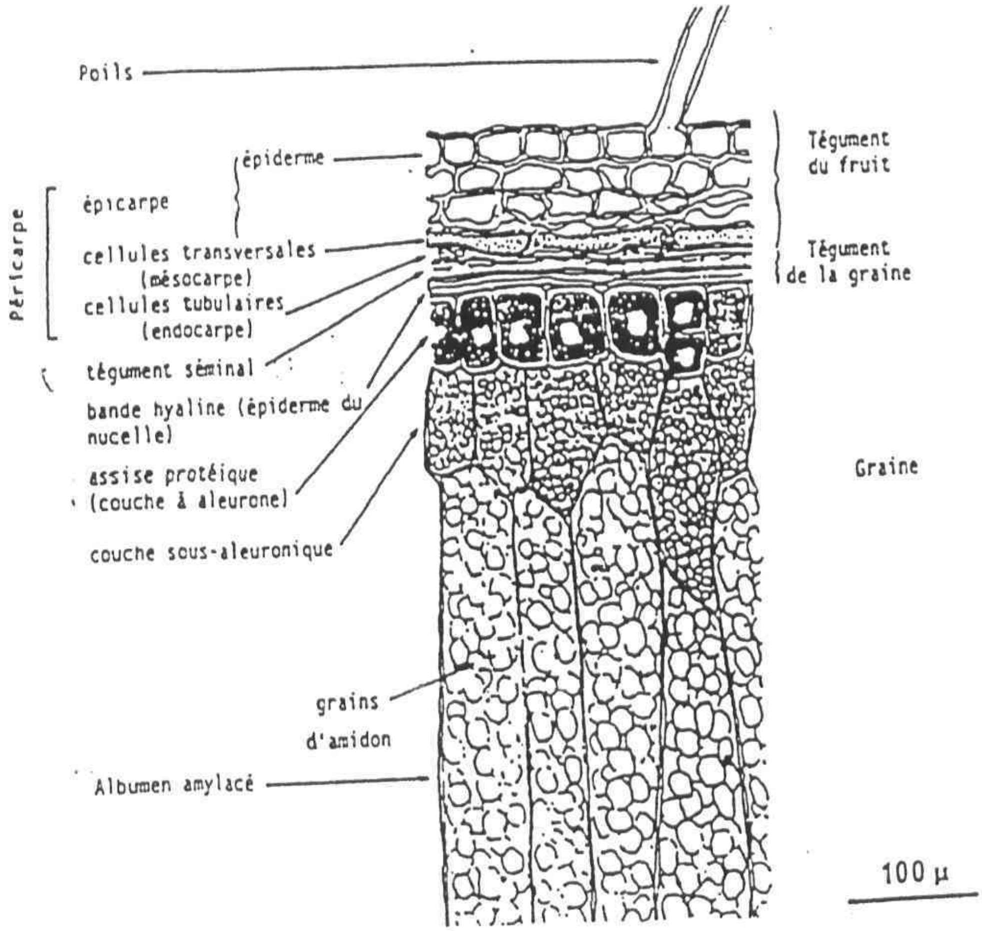


Figure 1.1: Longitudinal section of a wheat grain (Fleckinger, 1935)

Figure translation terms:

Tégument du fruit (Fruit Tegument); Tégument de la graine (Seed Tegument)
Poils (Hair); Péricarde (Pericarp); Épiderme (Epidermis); Épicarpe (Epicarp) : Mésocarpe (Mesocarp); Endocarpe (Endocarp); Tégument séminal (Seminal Tegument); Couche hyaline (Hyaline layer); épiderme du nucelle (Nucellus Epidermis); Assise protéique (Protein matrix); Couche à aleuronne (Aleuronne layer); Couche sous aleuronique (Sub-aleuronic layer); Grains d'amidon (Starch granules); Albumen amylacé (Starchy albumen)

Table 1.1: Chemical composition of the different parts of the wheat grain (Godon, 1991)

Grain Part (% of grain)	Proteins	Minerals	Lipids	Cellulosic matter	Pentosans	Starch
Pericarp (4%)	7-8	3-5	1.0	25-30	35-43	0
Seminal tegument (1%)	15-20	10-15	3-5	30-35	25-30	0
Nucellus epidermis (7-9%) Aleurone Layer	30-35	6-15	7-8	6	30-35	10
Germ (3%)	35-40	5-6	15	1	20	20
Endosperm (82-85%)	8-13	0.35-0.60	1.0	0.3	0.5-3.0	70-85
Peripheral endosperm (10%)	10-15	0.40-1.0				65-72
Central endosperm (70%)	6-9	0.3-0.4				72-88
Whole Grain (100%)	10-14	1.6-2.1	1.5-2.5	2-3	5-8	60-70

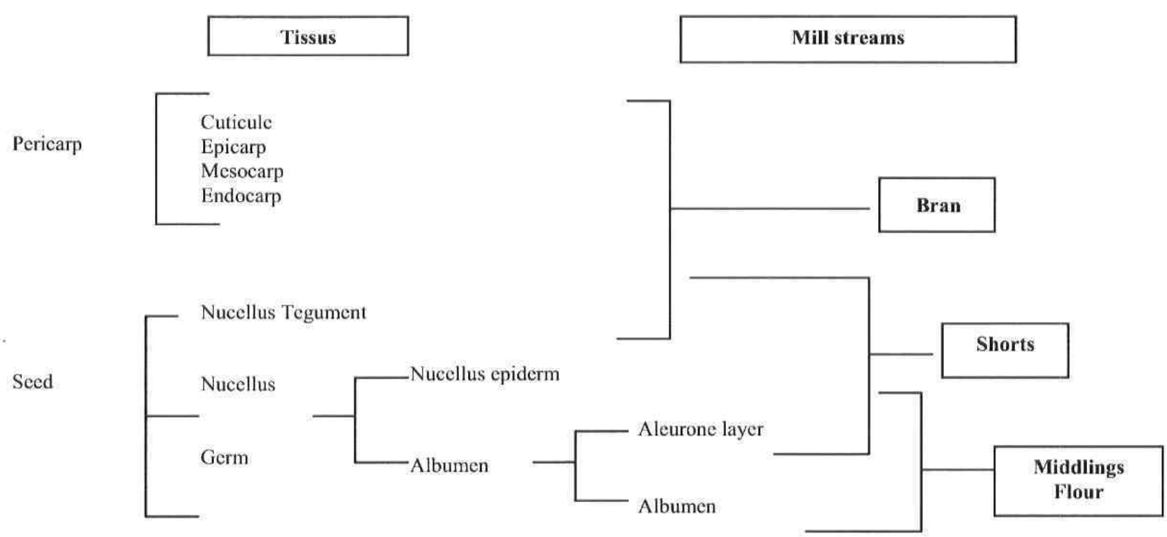


Figure 1.2: Relations between botanical components and milling fractions (Godon, 1991)

The endosperm is the most frequently studied part of the grain. It consists of the outer aleurone layer (6%–7% of the endosperm dry matter) and the starchy endosperm. The aleurone layer, which represents botanically the outermost layer of the endosperm, is partly eliminated during the milling process in the bran and shorts.

The starchy endosperm consists mainly of starch and proteins, in proportions that vary according to the position of the cell in the endosperm. The protein-starch ratio is higher in the outer cells of the endosperm (Evers, 1970). While the starch content is variable, the protein content remains constant regardless of the cells' position.

Starchy endosperm cells are classified according to their size and shape. Peripheral or subaleuronic cells, adjacent to the aleurone cells, are identical in size and cubical in shape (60 :m). The central endosperm cells are 72–144 :m long and 96–120 :m wide (Bradbury et al., 1956). Although they vary in size, all are round or polygonal in shape. The central cells are 2.6-:m thick, and a negative correlation has been found between the thickness of the cell walls and milling yield (Larkin et al., 1952).

1.2- The soft wheat milling process and flour streams

Efforts to understand and thereby control the milling quality of wheat have been made since the scientific description of the different endosperm types and began to appear in the latter years of the 19^{th} century. Many of the early descriptors were principally made in terms of the visual appearance of the endosperm, characterized as vitreous, steely or horny (such grains would generally be hard) as opposed to opaque, floury (for soft wheat). Endosperm hardness has been receiving a greater deal of attention due to its relationship with the milling properties, flour particle size and resultant water absorption, and dough characteristics, and has consequently become an additional marketing parameter (Hong et al., 1989). Wheat endosperms have been distinguished according to the mode of fissuring and crushing process of the grain (Greer and Hinton, 1950). In soft wheat, the fracture occurs at random across the endosperm to yield fine irregular particles, whilst in hard wheat, fracture tends to be along the walls of the endosperm cells, yielding large particles of more uniform shapes.

Milling consists of four major operations: receiving the grain; pre-cleaning, storage and cleaning; conditioning the grain for milling; and finally the actual milling process. Milling consists of a cycle of several separate operations making up the milling flow (Fig.1.3). These operations entail the reduction of the grain and endosperm fragments by grinding, grading by means of a sifting process and, lastly, the purification of the middlings, either hulled or with the bran still attached.

From the standpoint of the manufacture of biscuits, cookies, crackers, biscots and other starchy products, all the functional components of wheat come basically from the starchy endosperm; that is why the miller aims to recover as much endosperm as possible during the milling process, focusing on two objectives: a) to separate the floury endosperm from the hulls and germ and b) to reduce the endosperm into fine particles (Nuret, 1991).

The separation process takes advantage of differences in the mechanical properties of the three parts of the grain:

-The hull is pliable and resistant to breakage whereas the endosperm is brittle. These differences are accentuated upon the conditioning, or the tempering of the grain (17% moisture). Conditioning is accomplished by mixing the grain with water, once or several times, followed by a resting period of at least 24 hours or by other processes combining moisture, temperature and a resting period, while ensuring the uniform distribution of moisture in the batch (Willm and Joliet, 1994). The conditioning operation also facilitates subsequent sifting. Once conditioned, the grain passes through a series of rollers (Figure 1.3). First, the grain passes through the break system, consisting of two corrugated metal rollers turning in opposite directions at a controlled speed, which exert a shear force on the grain. The corrugated rollers act to separate the endosperm from the hull. The break system is designated by the letter B (B_1, B_2, B_3...) in the diagram. This operation reduces the wheat grain into different milling fractions, which are separated by sifting using plansifters with progressively finer sieves. After the first pass through the break rollers (B_1), the first sieve, which has large openings, retains the largest particles such as whole grains, large pieces of bran and large particles of endosperm with adhering bran, while the finer sieves collect smaller pieces of pure endosperm and endosperm with adhering bran (coarse and fine middlings and fines). The last sieves collect the break flour.

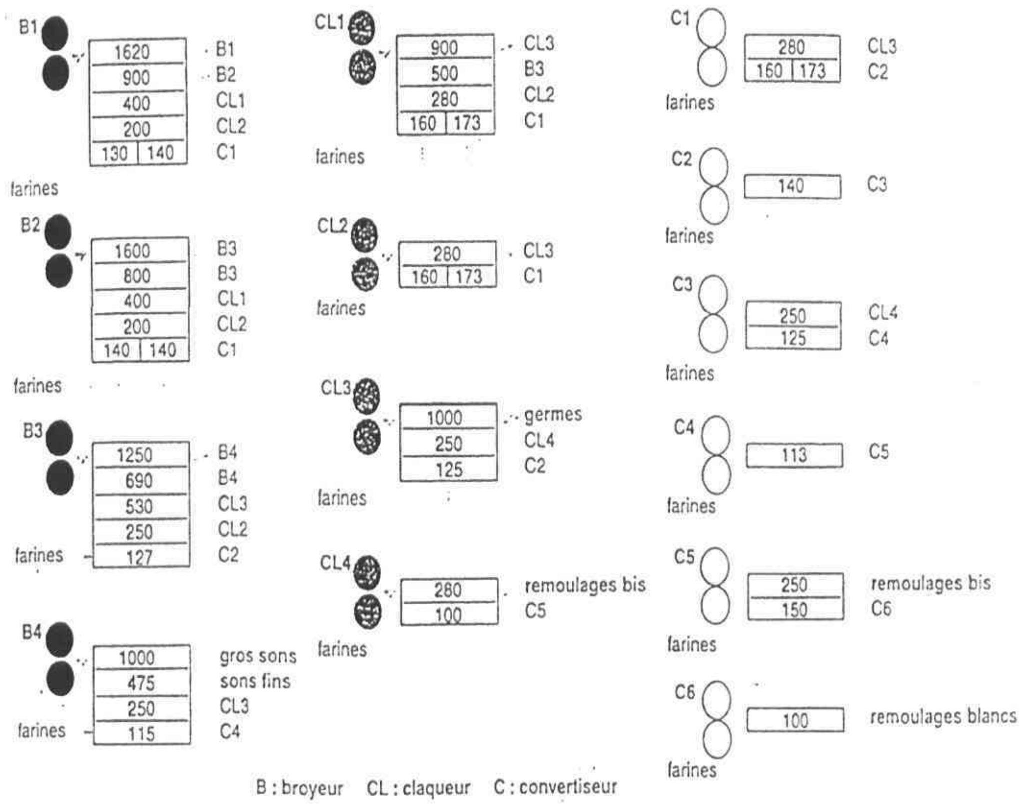


Figure 1.3: Milling flow chart for soft wheat (Willm, 1995)

Equivalence terminologique de la figure

B: broyeur (**Break**) Cl: claqueur (**Sizing rollers**) C: convertisseurs (**Middling rollers**)

The "overs" from the first break roller (what is retained in the upper sieve) go through a second series of break rollers set closer together (B_2), followed by three other sets (B_3, B_4, B_5) to maximize separation of the hull from the endosperm, while the bran (coarse and fine) is sucked out by a ventilation system. Particles retained in the intermediate sieves in the break system are sent to the sizing and middlings (reduction) rollers, representing two phases in the reduction of the particles. Sizing rollers, designated by CL (CL_1, CL_2, CL_3...), and middlings rollers, designated by C (C_1, C_2, C_3...), are smooth rather than corrugated. The purpose of these systems is to crush the pieces of endosperm into flour and eliminate any remaining particles of bran or germ. As in the break system, the products of the reduction rollers then pass through a system of sieves to separate endosperm particles of different sizes, bran fragments and lastly the flour. Overs from the first sieves pass through a series of sizing and middlings rollers to be further reduced to ensure that all the middlings and fines are transformed into flour. The flours obtained after each break, sizing and reduction operation are referred to as flour streams. The blends of these flour streams yield the straight grade flour (Prabhasankar et al., 2000; Berton et al., 2001). Moreover, during the milling process, the flour extraction started from the central to the peripheral layer of the grain to produce the patent and low grade flours as shown in Figure 1.4. This explains why each flour stream has different biochemical and physical characteristics.

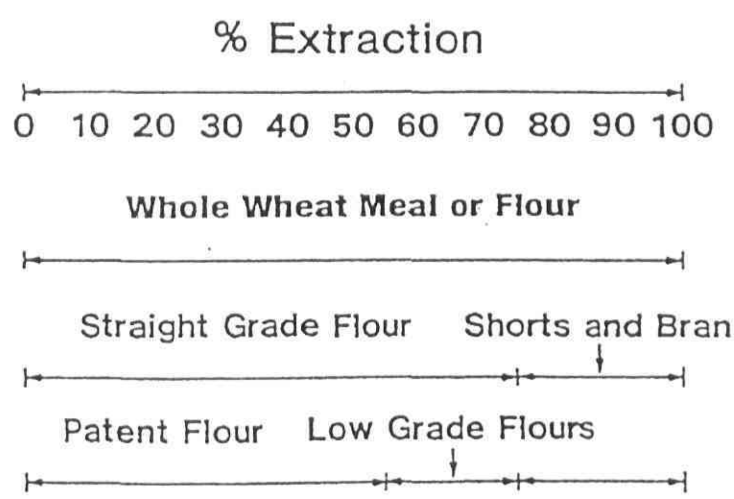


Figure 1.4- Typical grades of flour produced by milling wheat (Hoseney, 1994)

The main products obtained from milling are shown in Figures 1.2.

-Middlings (starchy endosperm) of various degrees of purity and particle size depending on the wheat used and roller setting. Two types can be distinguished: coarse middlings (hulls, endosperm fragments and coarse fractions) and fine middlings.

-Flour which has passed through a 150-:m sieve and is made up of very fine endosperm particles minus the hull.

-Non-starchy residues, which are divided into three groups depending on particle size:

a) bran (9-14% of grain by weight) made up of hulls and a certain part of the endosperm that adheres to the inner side of the hull.

b) shorts (12.5% of grain by weight) containing a mixture of hulls ground into different sizes and floury endosperm; these come from the end of the sizing and reduction operations.

c) germ (0.5% of grain by weight), the waste from the sizing and middlings rollers.

The extraction rate (amount of flour obtained from wheat after milling) for soft wheat flours is roughly 72% (Figure 1.4). This flour can be divided into patent flour (sometimes also referred as short patent flour), with a 45% extraction rate; middle-cut flour, with a 45%–65% extraction rate; and lastly, clear or low-grade flour, with a 65%–72% extraction rate (Hoseney, 1994; Atwell, 2001b).

Since milling is a progressive process, the first break rollers grind the grain into fragments that are, in turn, ground and reduced further in size. The purest products are those that come from the heart of the endosperm and are obtained from the head-end fraction or first break. At the end of the breaking process, on the other hand, it is the peripheral layer of the endosperm that is broken off as well as products with adhering hulls; in addition, the quantity of gluten increases from the centre to the periphery of the endosperm, negatively affecting quantity (Prabhasankar et al 2000., Berton et al., 2002). The same phenomenon occurs in the distribution of enzymes in the grain. The central endosperm has lower ash content than the outer part of the endosperm and the bran, which have higher ash content. The heart of the endosperm also contains the most functional proteins (Buré, 1975).

The progressive fragmentation of the endosperm during milling results in a particle size distribution that corresponds to the origin of the particles. The finest particles (< 20 :m) correspond to proteins and small starch granules, while the largest (>80 :m) represent more heterogeneous fragments (Willm, 1995). To define these differences in chemical composition, particle size distribution and other aspects according to the stage of milling, the terms patent, middle-cut and low-grade flours are used. As Figure 1.3 shows, the patent flour likely corresponds to milling fractions B_1, B_2, CL_1, CL_2, C_1 and C_2, consisting of fragments from the heart of the endosperm (flour streams obtained from the first break [B], sizing [CL] and middling [C] phases). On the other hand, the middle-cut flour corresponds to fractions B_2, B_3, CL_{13}, CL_{14}, C_3, C_4, etc., while the clear or low-grade flour is obtained at the end of the milling flow (B_5, CL_6...). The actual flour produced consists of mixtures of the flour streams from the various break, sizing and middlings rollers that the miller recombines in the desired proportions (Berton et al., 2001; Feillet 2000).

Millers control their own milling streams, and their skill is based on the preparation of the grain, the setting of the distance between break rollers, the selection of the sieve and plansifter sizes and the circulation of the materials and therefore the overall design of the milling flow. Given the same settings, differences in wheat behaviour are reflected in a larger or smaller middlings yield and greater or lesser production of the finer fractions. The miller may therefore vary the proportion of the different flour streams making up the commercial flour (patent, middle-cut or clear flour). Earlier in the process, before milling, the miller can also mix different varieties of wheats, such as red and white wheats, with useful characteristics to obtain more uniform grain characteristics for milling. For example, in 1997, Robin Hood produced commercial soft wheat flours containing 20% *Soft Red Ontario,* 30% *Soft White Spring* and 50% *Ontario Soft White.* In 1998, these flours contained 70% *White Ontario* and 30% *Soft Red White Ontario* (40% first patent, 48% middle-cut and 12% clear) and, in 2000, they contained 60% *Soft Red Winter* and 40% *Soft White Winter* (35/45/20).

The miller must obtain, from a batch of wheat, the maximum amount of flour in the appropriate qualities, at an acceptable milling cost. The yield or extraction rate, a component reflecting the intrinsic value of the wheat, indicates the quantity of flour obtained from a batch of cleaned grain arriving at the first break roller B_1 (% dry matter) (Grandvoinnet, 1991). If the extraction rate is high, the flour is "spiked, in other words, it contains a small quantity of ground bran originating from the hull fragments. The quantity of minerals found in the hull indirectly determines the extraction rate and purity of the flour.

The milling process is therefore a highly complex operation due to the different grindings of the wheat. Despite all the recent technological advances made in the milling industry, soft wheat flour produced today still contains certain botanical impurities from the wheat. As discussed previously, it is relatively easy to separate the germ during milling, since it has a high fat content which makes it to form chips, also referred as «plaquettes» ,that are easily separated in the milling operation. The bran is more difficult to separate and bran contamination increases when the peripheral areas of the endosperm are milled, due to the proximity of the bran layer. These impurities, due to the presence of bran, are non-functional from the biscuit-making point of view and therefore must be minimized in the endosperm milling, to obtain the best yield and least bran contamination. In the recent literature, milling quality is described in terms of the flour's friability (crunchiness), endosperm content and pericarp-endosperm separation. For several years, ferulic acid has been recognized as an excellent indicator of friability, while the flour's potassium content is used to predict its ash content.

1.3- Composition and functionality of the flour streams

MacMasters et al. (1971) reported the non-uniform distribution of flour components within the wheat kernel which explains the variation in composition and functional properties of different streams. During the milling process, the extraction of flour begins with the internal part of the endosperm and proceeds toward the periphery of the grain. Since these flour streams come from the different histological parts of the wheat grain, each stream has specific physicochemical and biochemical properties. Prabhasankar et al. (2000) and Berton et al. (2001) have found increasing protein, mineral matter, pentosan and fat content from the head-end (first break) streams to the tail-end streams. In addition, α-amylase activity decreases with the increase in the number of streams due to elimination of bran, that is also rich in enzymes (Kruger, 1981). A finer flour is also observed with the number of grinding treatments. In addition, the starch content decreases during the milling operation, from 80% in the polished grain to 40% in the flour. Some publications report variations in flour colour (Robinson et al., 1984a, b) and lipids (Morrisson and Barnes, 1980) among flour streams.

The rheological properties of flour-water doughs also vary depending on the flour stream. Manohar et al., 1991c showed that dough cohesion and elasticity were more pronounced in stream flours than in straight-run flours; a negative correlations have been found between total protein content and hardness ($r = -0.90$), cohesion ($r = -0.89$) and elasticity ($r = -0.91$) of doughs. A positive correlation was shown only with the adhesiveness attribute($r = 0.55$).

1.4- Sources of variability in flour quality

Sources of variability in the technological potential of flours comprise the wheat kernel itself and the milling process. The variability of wheat depends on genetic (cultivar) and environmental (soil, climate, cultivation practices) factors (Ziegler and Greer, 1978). Milling is a source of additional variation since the milling flow is different from mill to mill (Cleve and Will, 1996). The effects of these factors are not cumulative, however, but rather interact, often to a significant degree.

Genetic variability is traditionally determined via gliadin electrophoresis tests (Godon, 1991). A given variety may also provide a wide window of response, both of physicochemical and technological potential, based on environmental conditions.

There are few quality characters that are exclusively variety-dependent. The main one is the hardness of the grain, which results from the adherence of the protein matrix to the starch granules: the greater the adherence, the harder the grain. This character is hereditary and is probably coded by a gene on chromosome 5D and others minor genes (Rogers et al.,1995). The particle size index (PSI) method of determining the percentage of fine particles in a batch of grain allows hard wheats to be distinguished from soft wheats. The particle size distribution of soft wheat shows two peaks (populations) at 20 :m and 100 :m, while that of hard wheat shows only a single 100-:m peak (Willm, 1995; Hareland, 1994; Lorenz, 1986). Methods using near-infrared reflectance allow the grain hardness to be estimated (Brun and Mahaut, 1988). To distinguish between the two wheat classes, the 40-:m threshold works well (Tharrault, 1995,1997) since, in soft wheats, roughly 50% fine particles (<40 :m) are found, compared with only 25% in hard wheats. Note that hardness must not be confused with vitreousness, which involves above all the visual appearance of the endosperm, which is vitreous when translucid; this is not linked to genetic factors but is often associated with hardness since hard wheats are seen mainly as vitreous grains, as discussed previously. The particle size distribution of the flour streams also varies, that is why the first break flour is on the fine side since it is from the heart of the endosperm, which is more friable. The progressive reduction of the endosperm results in a particle sizes distribution in which the sizes of the particle are related to the particle's origin.

Since the endosperm of hard wheats breaks into larger fragments and this breakage can even occur within starch granules, a strong relationship can be observed between hardness and the level of damaged starch. However, in soft wheat, breaks generally occur along the contours of granules and, given similar milling conditions, the damaged starch content is significantly lower. Branlard et al. (1985) and Saulnier et al. (1995) also referred to the wide differences in composition, quantity and functionality of pentosans among wheat varieties. Hong et al (1989) reported that the pentosans content varied in soft white wheat from different region; moreover, geographic locations can influence the final quality of soft wheat products (Kady and Rubenthaler, 1987). Soft wheats, due to their greater friability, provide more break flours and less coarse middlings than hard wheats. White and red soft wheats also exhibit different degrees of friability.

Environmental variations can play an important role in grain quality. For example, nitrogen inputs favour protein synthesis (Martin, 1990). In addition, there is an interaction between nitrogen inputs and soil moisture conditions: dry soil and high nitrogen inputs favour protein synthesis. Wet years, on the other hand, promote starch synthesis; the quantities of starches, type A (prime starch) and B (starch tailings) are also influenced by weather conditions and an increased day length promotes the synthesis of B granules. Hot and dry temperatures increase the quantity of pentosans (Rouau and Saulnier, 1994).

Variability due to milling is far from being negligible when compared with the genetic and agronomic factors discussed earlier. Prolonged conditioning involving high moisture content decreases the hardness of grain and increases its intrinsic milling yield (Willm and Joliett, 1994). This operation may also have repercussions on the rheological characteristics of biscuit doughs (Dubois,1995; Yamazaki and Donelson, 1983).

Milling yield is influenced by a number of factors such as the speed of the rollers, the condition of their surfaces and the distance between rollers, all of which have an impact on the damaged starch content. Damaged starch content varies depending on the flour stream and increases in ascending order from the break rollers to the sizing and middlings rollers (Denantés, 1987).

Millers can combine the various flour streams from the break, sizing and reduction systems in specific proportions to improve the flours' suitability for specific or more general applications. In fact, the stream splitting method where a given group of similar or complementary streams are blended for particular type of end-products is often employed by the baking industries that require specific quality flour to manufacture different products (Dubé et al., 1987). However, it is also crucial to select the streams on their quality characteristics, which depend on the flour grades extracted in the mill (Dexter et al., 1984), the type of wheat cultivars used for milling and the flow sheet followed in the milling operation (Cleve and Will 1966). It should also be noted that wheat flour undergoes rapid alteration during the two-week period after milling due to the oxidization of fats. This modifies the dough's water absorption and rheological characteristics. Storage under conditions of high absolute humidity is also a significant factor in variations (Berger, 1997).

Due to variations in harvest yields and the cultivars sown in a given year, millers are often forced to mix wheat cultivars from different sources and locations (Eastern versus Western sources of soft wheat, Red versus White wheat) to obtain specific hardness characteristics to be more appropriate for a given milling flow in flour production. Such mixtures can also contribute to differences in dough rheology and the quality of the finished products.

2- Technological value of soft wheat flours

2.1- Biscuit, cookies and crackers doughs

The biscuit and bakery product industry offers a wide range of products, with over 1,000 known references to date. The diversity of these products is closely linked to their formulations and manufacturing processes (Figure 1.5). Three main ingredients are used in cookie and biscuit manufacturing: soft wheat flour, sugar and fat.

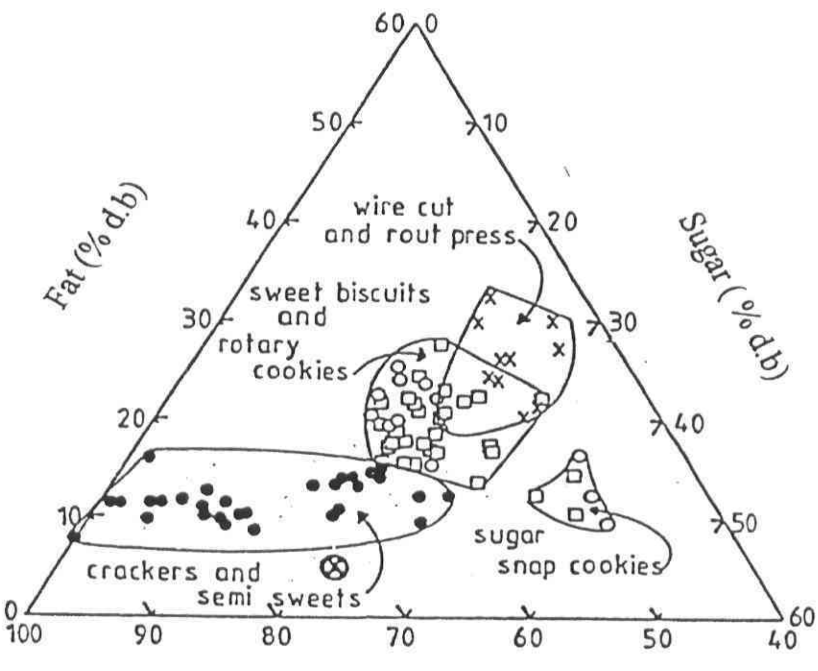


Figure 1.5: Composition of different types of biscuits, cookies and crackers according to the flour, fat and sugar content (Wade 1988)

«wire-cut and rout press»: biscuits cut by wire with the rout press process; «Sweet biscuits and rotary cookies»: dry biscuits laminated and rotary: « sugar-snap cookies»: cookies; « crackers and semi-sweet»: crackers and the dry biscuits.semi-sweet biscuit recipe corresponds to **CTUC recipe**.O

As Figure 1.5 shows, the respective proportions of these three ingredients correspond to the various types of cookies, biscuits and crackers (Wade 1988). The figure also shows the CTUC test formula «Centre Technique des Utilisateurs de Céréales de la Biscuiterie et Biscotterie», adopted in the present research project. This formula was used to introduce a new category of biscuit wheats in Europe.

Traditionally, biscuit and cookie manufacturers have used soft wheat flours with low protein (7.5%–9.0%), low ash (0.45%–0.60%) and low damaged starch contents; a pH of 4.0–6.6; and low W alveograph (flour strength) values or (W<150). However, such criteria are insufficient of assessing flour quality and the functionality of proteins and other components of flour in this context are still poorly understood (Mansour, 1982; Berger, 1997; Verel, 1996; Morris and Rose, 1996; Stauffer, 1996).

Although it is well known that certain proteins play a role in the dough network, no simple relation has been found between protein content and biscuit length; If the protein content is below 7.5%, problems in dough machinability may occur; but at 12% and over, excessive retraction occurs, and biscuits dimensions are difficult to control (Bartolucci and Launay, 2000).

Most publications on biscuit-making refer to the cookie or biscuits terms. For cookies (sugar-snap cookies), the most commonly used North American reference methods are the AACC standards 10-10 D ("Baking Quality of Cookie Flour") and 10-52 ("Baking Quality of Cookie Flour–Micro Method"). This cookie test is used by the Canadian Grain Commission (CGC) in assessing new harvests of soft wheat. However, the trend is towards the use of newer AACC methods, such as 10-53 ("Baking Quality of Cookie Flour–Macro Wire-Cut Formulation") and 10-54 ("Baking Quality of Cookie Flour–Micro Wire-Cut Formulation").

The findings on the role of flour in the dimensional characteristics of cookie are contradictory and have been qualified as anecdotal by Slade and Levine (1994b,1995). According to these authors, the AACC 10-52 cookie test method (sugar-snap cookies) contains too much sugar and fat, which probably mask or even distort the effects tested, including those that depend on flour quality. These authors recommend that the AACC 10-

52 sugar-snap cookie method, which has a flour : sugar : water : fat ratio of 100 : 60 : 15 : 30 for flour with a 14% moisture content, be replaced by a wire-cut-cookie based method such as the AACC 10-53 (flour : sugar : water : fat ratio of 100 : 42 : 22 : 40) to determine flour functionality. The latter test appears to ensure better repeatability, is more sensitive to protein content, and also positively correlated with cookie hardness (Gaines, 1993). In addition, this cookie test allows for better discrimination of wheat cultivars and the ranking obtained is closest to that observed in commercial production .

In the case of the dry, semi-sweet biscuits, type petit-beurre, the CTUC's biscuit recipe is used as a baking test in Europe to select wheats for biscuit-making (Tharrault, 1994a, b and 1997). This recipe is significantly richer in flour (70% versus 50% for AACC cookies 10-52) and the moisture content is doubled (flour: sugar: water: fat ratio of 100: 30: 26: 8). Therefore, it can be assumed that the flour effect in this recipe will have a greater impact on the rheological properties of the doughs, since these properties are likely to be more sensitive to the consequences of the formation of a viscoelastic gluten matrix. This baking test has been validated industrially by the CTPS, « Centre Technique Permanent de la Sélection des variétés» which used it since 1994 as a basic test for registering new wheat varieties in Europe, in the newly created category of biscuit wheats.

The classification of biscuits and/or cookies depends first and foremost on the production method, which is governed by moisture content, relative ingredient composition of the recipe and the rheological characteristics of the dough (Morris and Rose, 1996; Warthesen, 1995; CDAQ, 1992). Three major categories of commercial products are known in North America:

-Rotary moulded products: the dough is forced into the moulds with pressure and becomes cohesive under the effects of pressure, due to its high fat and sugar content and low moisture content (~ 20% on flour basis); this type of formulation is required since gluten development is quite limited during mixing and therefore, there is little spreading in the final product.

-Laminated processed products: the dough passes through a sheeter, which superimposes the gluten films in the dough, orienting the partially developed gluten matrix. The dough must be fairly cohesive before passing through the sheeter or laminating unit; the gluten is partially developed and mixing ensures the dough is somewhat extensible. This recipe corresponds to the CTUC recipe which is very similar to the petits beurre, often referred as semi-sweet biscuits.

-Wire-cut products: the dough must be soft enough for gravity feeding and cohesive enough to be cut with a thin wire. Spreading during baking is desirable and cookies are usually soft.

Overall, the moisture content increases from rotary to wire-cut and laminated processed biscuits, respectively. Biscuit dough development is determined mainly by gluten development. The mixing, protein oxidation and water absorption stages, as well as tolerance to retraction, play a major role in the structure and rheological properties of these doughs (Warthesen, 1995; Mansour, 1982).

The terms "short dough" (sweet cookies) and "hard dough" (semi-sweet biscuits) are often used in the United Kingdom to refer to wire-cut and rotary mold cookies (short dough) and French-made semi-sweet biscuits or the petit beurre. Gaines (1990) refers to the absence of a continuous gluten matrix in short dough due to the high sugar and fat content; in addition, the dough consistency varies greatly from batch to batch. However, according to the same author, there is sufficient water in hard doughs to hydrate partially the proteins and therefore, the mechanical energy in the mixing process allows the glutenins, gliadins, lipids and pentosans to develop into a functional composite gluten matrix. Too much water may also interfere with protein aggregation and thus alter the formation of the gluten network.

The role of water, sugar and fat, as the main ingredients in biscuit recipes will be briefly reviewed below. The different proportions of these ingredients in recipes are responsible for the wide variety of size and textures found in the finished products.

-Water: an essential element in dough formation, allowing the solubilization of ingredients, hydration of proteins and carbohydrates and the development of the gluten matrix. The role of water is complex, since it determines the conformational state of the biopolymers and affects the interactions between the various components in the recipe (Eliasson and Larsson 1993). Any increase of moisture content modifies the elastic modulus (G') and viscous modulus (G''), and reduces the viscosity. If there is too little moisture, the dough becomes brittle, due to rapid surface dehydration (Bloskma and Bushuk, 1971).

-Sugar: an excessive sugar level helps to reduce the consistency and cohesion of the dough due to the competition between sugars and water availability (Olewnik and Kulp, 1984). Since sugar retains water, sugar also acts as a hardener, causing cookies to crystallize during cooling and become crispy or crunchy (Schanot, 1981).

-Fat: contributes to the plasticity of the dough and acts as a lubricant. In large quantities, the lubricant effect of fat is so great that only a little water is required to obtain a low level of consistency. In addition, if fats are mixed with the flour before hydration, they can hinder the formation of the gluten matrix, resulting in a less elastic matrix (Faubion and Hoseney, 1990). Note that the effect of fats on dough and biscuit quality is not only a function of their composition but also that of flour and its native lipids (Kissel et al., 1971).

2.2- Dimensional and textural characteristics

Cookie diameter is determined basically by the speed at which the dough spreads during baking, as well as the speed at which the dough stops spreading. It is recognized that dough made from soft wheat flour spreads quicker because it becomes less viscous than dough made from hard wheat flour (Gaines and Finney, 1989).

Cookie quality is assessed according to dimensional characteristics: diameter, or width (W), thickness (T) and the ratio between the two, also known as the spread factor (W/T). High-quality cookies have a large diameter and a low thickness, and therefore a high a spread factor (W/T) as possible. Given doughs made with a high-quality and low-quality flours, thickness will increase during baking in both doughs but, at the end of baking, the thickness will return to its original level in the dough made with high-quality flour, and it will remain at a significantly higher level in the dough made with low-quality flour and therefore the spread factor (W/T) will be lower. Spreading is caused by the expansion of the dough under the action of leavening agents and gravity flow. The speed of spreading is controlled by the viscosity of the dough (which is determined in turn by the competition of other ingredients for water). In the case of sugar-snap cookies (60% sugar on flour basis), dough made with high-quality flour results in 50% spreading (e.g., from 6 cm to 9 cm diameter).

Both the dimensions and additionally, the density of dry, semi-sweet biscuits are also evaluated. The 6 cm x 6 cm square shape biscuits made with the CTUC recipe should present minimum shrinkage and a low density, so the rapid spreading in the oven is desirable. Certain European publications also mention eccentricity, a measurement corresponding to the ratio of the biscuit's length (direction of sheeting) to its width.

In addition, the texture of cookies and biscuits is perceived differently. While hardness is measured in cookies, crunchiness or friability is assessed in biscuits. Crunchiness refers to the number of grains and the specific tearing force for these grains. Cookie texture is fairly complex, mainly because of the relation between geometry and moisture content and the fact that mechanical properties are dependent on the water content and resulting sugar content. The ideal texture of a cookie can be summarized in two words: tender (low breaking force) but shock-resistant (strong force during impact). In the case of biscuits, where flour plays a different role, texture depends essentially on the apparent mass density of the biscuit, and therefore its dimensional characteristics (Branlard et al., 1985; Tharrault, 1997a, b). Other qualitative tests have been described, including internal structure, colour, flavour (sweetness) and surface appearance, or top grain, which refers to the presence of fractures or cracks on the cookie surface due to the recrystallization of sucrose on the product surface during baking (Clements, 1980; Maache Rezzoug, et al.,1998b,c).

A biscuit can be described as a composite matrix constituted of cavities of various sizes and shapes, which are formed during baking as the leavening gases and water vapour are released. These cavities are irregular holes formed by the expansion of air pockets trapped in the dough during mixing. The walls of cavities are formed from pieces of the endosperm linked together partly by contact with each other and partly with a sugar glass, rather like bricks and cement in a wall (Burt and Fearn, 1984; Goullieux et al., 1995; Sharp, 2001). The mechanical properties in a composite food are affected especially by the volume fraction and distribution of each phase and by the nature and extent of interactions between various phases. Additionally, the fracture pattern depends on size and distribution of defects in the material (Baltsavias et al.,1999). The mechanical characteristics of semi-sweet biscuits are usually determined with cone penetrometry (Goullieux et al., 1995; Maache Rezzoug et al., 1998 a,b,c). The analysis of the fracture pattern on the force-deformation allows the quantification of the following attribures: a) Force (Fs) required to penetrate, or tear, the particles or groups of particles in the biscuit, which provides information on the cohesion of the structure and therefore the strength of the bonds between grains or groups of grains; (b) spatial frequency (No), which reflects the number of fractures required to penetrate, or tear, particles or groups of particles by the length (mm) of penetration of the probe into the product; (c) the overall mean penetration force (Sm) and lastly (d) the overall crunchiness or friability effort of the biscuit (Fs/No). The crunchier a biscuit is, the greater the number of grains penetrated (high No) at a low tear force (low Fs). For cookies, the three-point bent test is used for quantifying their hardness as key textural attribute (the so-called cookie snap) and also the fracture stress and fracture strain (Baltsavias et al., 1999).

2.3- Baking biscuit doughs

Biscuits are baked in tunnel ovens several metres long, made up of several sections with different temperatures and humidities. Heat is transferred through conduction (between the metal conveyor and the bottom crust of the cookies), convection (through the movement of the conveyor) and infrared radiation (through the oven walls). Several physicochemical events occur during baking (Feillet, 2000; CDAQ, 1992).

- Melting of fats at 15^0C–50^0C, which is responsible for the plastic qualities of the dough and promotes the spreading of the dough under the influence of gravity.

- Dissolution of sugars: half of the sugars dissolve during mixing and the rest, in the crystalline state, dissolve during heating (1 g of sugar and 1 g of water produces 1.6 ml of solution). This also increases fluidity and allows the dough to spread out.

- Gelatinization of starch at 52^0C-95^0C, given a sufficient quantity of water. In general, the starch is only partially gelatinized in biscuits and the starch granules conserve their morphological integrity as native starch.

- Baking powders become active at $55^0C–70^0C$, releasing CO_2 and NH_3, with the dough spreading in all directions.

- Once the glass transition temperature has been exceeded (moisture-temperature dependence), the protein network can develop, and this is accompanied by an increase in viscosity. Since starch is only partially gelatinized in cookies, the increased viscosity can be attributed to the properties of the proteins.

- Dough continues to expand by production and thermal expansion of gas, until the viscosity of the dough becomes too high.

- Water loss and drying of biscuits continue until a moisture content of 3%–5% is reached, with rigidification of the network structures. Fractures (cracking) may appear on the surface when the water on the surface is lost and is replaced by water diffusing from the interior of the cookie. The sugar, which is not volatile, tends to become concentrated, resulting in crystallization on the surface of the cookies during baking. The surface dries out and cracks while the leavening agents continue to promote expansion, giving the cookies their porous structure (Hoseney et al., 1997).

- Lastly, formation of Maillard reaction derivatives, caramelization of the sugars and accompanying dextrinization of starch with the modification of the products' surface colour and flavour generation (Camire et al.,1990). At the same time, changes can be observed in the cookies' dimensions and texture due to the low moisture content (3%–5%). Due to the low moisture content, most of the components (starch and protein) are at the glassy state and are brittle, giving the cookie its "snap" when it breaks. The snap is due to the crystallisation of sucrose and it is possible to interfere with the sucrose crystallisation by adding another sugar form or others substances

3- Fractionation of soft wheat flours

3.1- Overview of fractionation techniques

As in the case of any biological material in its natural state, soft or hard wheat flour can be considered as a heterogeneous complex made up of a multitude of organic compounds. The main components of wheat flour are starch and proteins, while lipids, sugars, non-starch polysaccharides and other components are found in lesser quantities. In addition, these components are, for the most part, compounds with a high molecular weight, originally occurring in colloidal form, with dynamic physical interactions between them are prevalent. The fractionation of the major and minor components and their reconstitution in their original or different proportions are therefore difficult to achieve.

There are several different ways of studying the contributions of flour fractions to technological performance in baking and the rheological properties of doughs. The approach can be carried out via indirect correlative studies (Payne et al., 1987) or by fractionation studies (MacRitchie, 1985).

In the first approach, a population of flour samples with different physico-chemical characteristics and functionality is characterized and then relationships are established between these parameters using statistical methods. This approach has often been considered for the scoring of the high molecular weight glutenin subunits in bread –making in quality and also for the understanding of the contribution of low molecular weight glutenin subunits on flour functionality (Payne et al., 1987; Köehler et al., 1999, Uthayakumaran and Lukow, 2003). This approach has never been considered with biscuit making.

The second approach implies direct measurements on dough with modified chemical composition. This approach involves the separation and fractionation of flour components. These components can then be recombined in their original concentrations or modified to study the specific role of one or more components or by interchanging between flours of different technological value to better define the role of each fraction and also establish which fractions are contributing to the differences in flour functionality and product quality. A number of studies have dealt with the separation of the gluten, starch tailings, prime starch, soluble fraction, lipids and other components to study their individual or combined contributions to the dimensional characteristics of cookies (Yamazaki, 1955; Sollars, 1956a,b; Sollars 1958,1959 and 1966;MacRitchie, 1985; Donelson, 1988,1990). This approach is also very useful in obtaining a better understanding of the impact of adding reducing and oxidizing agents and other chemical agents into the flour, in order to propose certain hypotheses on their actions and intermolecular interactions with gluten and study their influence on the rheological properties of dough and on biscuit quality (Pomeranz, 1977). The fractionation and reconstitution of wheat flour requires that the original functionality of components is preserved and that no components are lost in the wash waters (MacRitchie, 1985, 1987). This approach allows structure-function relations of wheat proteins, for example, to be determined (Köhler et al., 1999; Uthayakumaran and Lukow, 2003). This approach has, however, the deficiency that, under some circumstances, masking effects can reduce sensitivity. Several studies have also noted the difficulty of separating individual components properly from soft wheat flours, particularly when using manual kneading. This is probably due to personal factors in manual fractionation since some studies had more success than others in separating gluten from these flours (Pomeranz, 1977).

Two very distinct approaches can be used in the fractionation of the components of soft or hard wheat flours: the use of mechanical force via ultracentrifugation (physical approach) or the sequential extraction with solvents (chemical approach).

3.2- Wheat flour separation based on physical approach

Since wheat flour contains some amounts of soluble pentosans and starchy polysaccharides from granules damaged during milling, a model system has been proposed by Tolstoguzov (1997 and 2002). Figure 1.6 shows that the concentration of gluten phases reaches 20 to 30% at equilibrium with a solution containing about 1% polysaccharides and soluble pentosans due to the thermodynamic incompatibility between gluten proteins and flour polysaccharides.

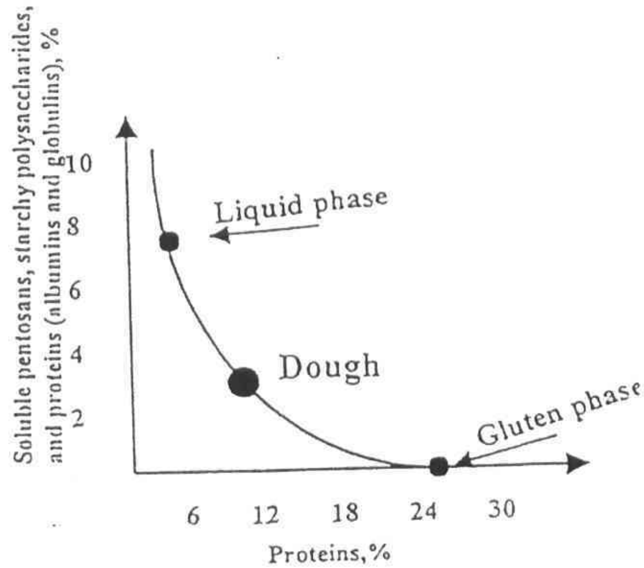


Figure 1.6: Wheat flour dough phase diagram (Tolstoguzov, 1997, 2002)

According to this model, on mixing wheat flour with water, two immiscible continuous aqueous phases must be formed under which gluten concentrations are in equilibrium with a solution containing roughly 1% polysaccharides, pentosans and soluble proteins. The first corresponds to a viscous continuous phase with a base containing soluble polysaccharides and most of the soluble proteins that are the albumins and globulins. This liquid phase, recognized to be a nonwettable medium, prompts the aggregation of the dispersed particles containing the gliadin and glutenin fractions. The aggregation of the gluten dispersed particles minimizes contacts with the nonwettable medium and lead to the second continuous phase. Both continuous dough phases are filled with several types of dispersed particles: native and/or damaged starch granules, insoluble pentosans, others food fibres, lipids droplets and air bubbles. Since starch is more hydrophilic that gluten protein, its gelatinization on baking causes dewatering of the continuous dough phases. Other factors contributing to the gelation of the gluten phase are: a) decrease in water binding capacity due to protein denaturation; b) water evaporation from the exterior loaf or biscuit layer; 3) the Maillard reaction and other cross-linking reactions of the macromolecules; and 4) glass transition phenomena in the gel network and within the exterior layer of the biscuit layer that fix its shape, structure and also retard staling in the baked products.

Baker et al. (1946) were the first to demonstrate the muliphasic nature of wheat doughs. One of the unique characteristics of wheat flour is that it separates into two continuous aqueous phases when it is transformed into dough. This separation occurs when the moisture content of the dough is fairly high, at around 48.6% (Eliasson and Larsson, 1993); two phases are obtained, the gluten phase and the liquid phase containing starch granules and soluble components.

Under the influence of the mechanical forces of ultracentrifugation, several distinct layers with highly distinct identities can be observed, namely starch, gluten gel,pentosan gel. Lipid liquid phase and some intermediates layers of damaged starch granules. These observations were confirmed by Mauritzen and Stewart (1965), MacRitchie (1976) and Larsson and Eliasson (1996a, b) using a number of different cultivars and dough composition. The charactertization of the dough fractions showed that the composition of dough phases is quite consistent for different wheat flours (MacRitchie 1976, 1984)

According to the technique described by Mauritzen and Stewart (1965), 42-g portions of simple dough (300 g of flour and 190 ml of salted water with 0.5M NaCl) are ultracentrifuged at 10,500 g for 70 minutes at 30 ^{0}C. Five or six distinct layers are obtained; one layer making up 10%–25% of the dough contains 30%–60% of the total nitrogen, or four times the original protein content of the dough.

There are several commercial processes for the physical fractionation of starch and gluten (Fellers 1973, Barr 1989, Meuser et al., 1989, VanDer Borght et al., 2005). The principles of gluten and starch industrial separation processes, such as the Martin and Raisio methods Feillet,2000), which are used to also recover prime starch and starch tailings, are shown in Figure 1.7. These two processes can be differentiated mainly by the quantity of water added to the flour for fractionation and by the techniques used to separate gluten and starch: specifically, the steps of washing with water, decanting and centrifuging. In the Martin process (solid-solid process), gluten is extracted from a flour-water dough similar to bread dough (60 litres of water to 100 kg of flour) by mixing it under a weak stream of water (kneading) and recovering the aggregated proteins (gluten) on a sieve, as well as the starches (first grade starch, or A-starch, containing large granules and second-grade starch, or B-starch, containing smaller granules mixed with pentosans) by centrifuging the filtered wash waters. In the Raisio process (liquid-solid process), the flour is suspended in water (roughly 150 litres to 100 kg of flour). After a resting stage, the first grade starch is recovered in a decanter centrifuge or hydrocyclone (Fellers, 1973). Gluten dispersion is then carried out for 40 minutes at 40 ^{0}C, which causes the gluten to clump and separate from the B-starch.

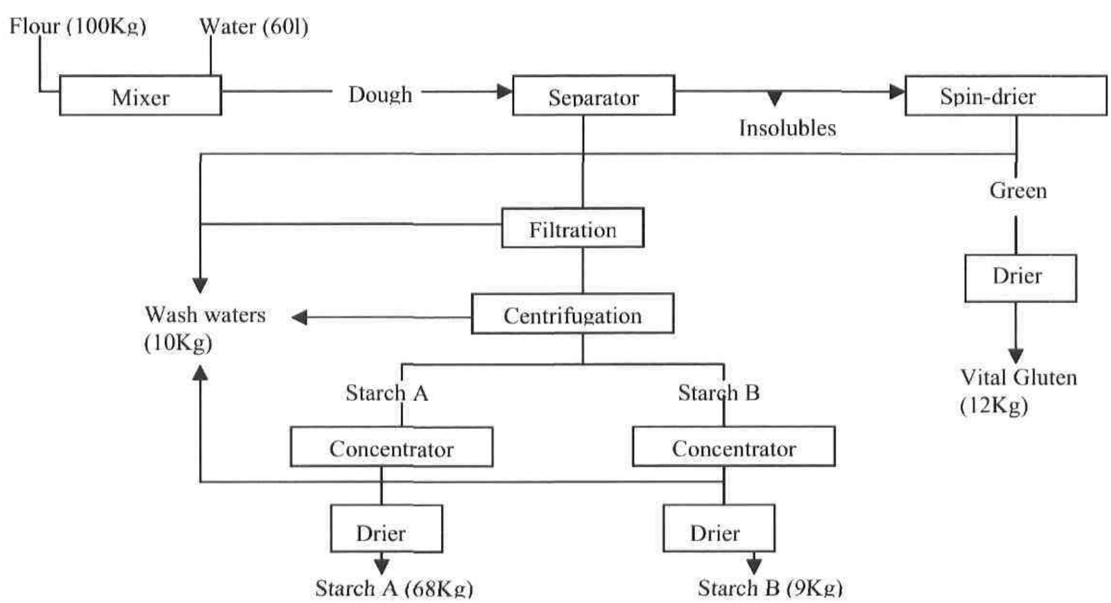


Figure 1.7: Principles of gluten and starch production (Feillet 2000)

The Martin process has certain disadvantages, including the large quantities of water required (10 m^3/tonne of flour) and large amounts of waste water generated (5-8 m^3/tonne of flour). In addition, the functional properties of the vital gluten fraction obtained from the dough are slightly modified, namely the hydration rate, water absorption and reduced gluten vitality.

In the case of liquid-solid processes such as the Raisio process, the quantity of water used and waste water generated is around 2 and 3 m^3/tonne of flour, respectively (Barr, 1989) and dough is not produced. In the Martin process, the starch is mixed with pentosans, which are recognized to alter the breadmaking properties, whereas the Raisio process allows first-grade starch of high purity to be obtained separately from the starch tailings, which contain most of the pentosans.

Czuchajowka and Pomeranz (1995) developed another fractionation technique in order to reduce the quantity of water used and waste generated. These authors prepared a simple dough (200 g of flour at a 55% absorption level for soft wheat flour mixed at 110 rpm for 2.5 minutes). The dough was left to stand at 15 ^{0}C for 40 minutes in 200 ml of water and the mixture was then transferred to a mixer with sufficient water (500 ml total including wash waters and mixing water). The dough (700 g) was vigorously agitated for three minutes and then centrifuged at 2,500 rpm for 15 minutes. Six layers were obtained: the bottom one containing the prime starch (1/3 by volume), followed by a layer of insoluble fibre, a layer of protein-rich gluten, which was easy to recover (1/8 by total weight), a layer of starch tailings (which can be washed from the protein layer with water) and lastly the water soluble layer (1/3 by volume) and a second protein layer on the surface. The dispersion stage not only prevents the development of gluten, which would interfere with subsequent separations, but also destroys the structure of the gluten. According to the authors, this process preserves gluten vitality better, while significantly decreasing the amount of water required for wheat flour constituent fractionation.

3.3: Wheat flour separation based on a chemical approach

As discussed previously, the fractionation of flour is a delicate operation in which the functionalities of the various components must be preserved (Sollars, 1956a, 1958 and 1959). Solvent-based fractionation techniques have also been used for many years. The main contribution in this area was made by Finney (1943), who was the first to recover the soluble fraction and reintegrate it during the reconstitution of flour. He used organic solvents to extract lipids from the flour, first by removing the non-starchy lipid fraction to prevent it from becoming attached to the proteins and to obtain a high extraction rate. A study by Yamazaki (1955) found that, when the fractionation of soft wheat flour is carried out with water as the only reagent, sugar-snap cookies show spreading similar to that found using the original flour, but the surface appearance or "top grain" was not acceptable. In addition, changes in the internal cellular structure can be observed (Clements and Donelson, 1981). These changes can be attributed to the collapse of air bubbles during expansion in baking and their coalescence to form larger bubbles. According to Daniels et al. (1966), when proteins are fractionated, different quantities of lipids still remain associated with the protein fractions, and are very difficult to separate afterward. This suggests that, when flour is in contact with an aqueous solution, a large quantity of lipids become bound and cannot be extracted afterwards with solvents (Olcott and Mecham, 1947). Since lipids and proteins alter baking performance, it is difficult to separate the contribution of the protein and lipid fractions. When gluten is added to the flour base, the presence of high concentrations of lipids, i.e. three to four times the normal levels in the gluten fraction, it is difficult to adequately evaluate the role of the protein fraction and/or sub-groups of proteins. According to Yamazaki and Donelson (1976), following aqueous fractionation, lipids do not perform their role as effectively in cookie baking. However, if the lipids are extracted from the flour using a solvent such as hexane to break the lipid–gluten bonds and are then restored to the flour, the baking of the cookies proceeds in the same way as with the original flour.

Finney et al. (1976) studied the effect of nine solvents on the composition and extraction of total lipids. Petroleum ether, *n*-hexane and *n*-heptane provided poor extraction rates compared with the other solvents (benzene, chloroform, acetone, butanol, methanol and 95% ethanol). The literature on the effects of various parameters on lipid extraction rates includes notably the studies of Chung et al. (1978a, b) which showed that extraction increases with the solubility of the solvent, temperature and the flour moisture content.

Sollars (1956a) separated flour into several fractions using acetic acid. First, he obtained the soluble fraction by adding 200 g of flour to 500 ml of distilled water, which he centrifuged for 10 minutes at 1,900 rpm. The supernatant was decanted, filtered and vacuum-concentrated at below 40 ^{0}C, for a final volume of 200 ml. The dough collected from the centrifuge tube was transferred into a blender, and 500 ml of an acetic acid solution (2.5 ml acetic acid and 475 ml of distilled water) was added to adjust the pH at 3.0. The mixture was agitated for one minute and centrifuged for 10 minutes and the extract was decanted. The residue was again extracted with an acetic acid solution (5 ml acetic acid and 400 ml of water) to maintain the pH at 3.0, and the mixture was centrifuged and decanted. The acid extracts were neutralized to a pH of 6.4 using a 5N NaOH solution. The mixture was centrifuged for 30 minutes at 1,900 rpm to obtain the gluten. The upper layer of residue, the starch tailings, was then removed mechanically with a spatula and the prime starch, or lower layer, was washed with distilled water. All four fractions were freeze-dried. Sollars (1956a) compared three methods of separating these four fractions: manual kneading, mechanical kneading and acetic acid extraction. The results indicated that the gluten and soluble fraction yields obtained with manual kneading were slightly better than those obtained with the other two methods. In addition, the protein content of the four fractions obtained with these three methods varied fairly significantly. Acetic acid extraction provided the best separation of the two starches while manual kneading tended to result in disintegration of the dough. The three methods produced cookies with similar diameters and colours. However, the surface appearance varied considerably, with acid extraction promoting the cracking of the cookies surface. Using acetic acid extraction, Sollars (1956a) confirmed that flours could be reconstituted from the four fractions obtained, producing cookies with diameters very similar to those of cookies made with the original flours, without the lipid extraction procedure. Moreover, air and freeze-drying of these fractions (except for the soluble fraction) provided similar results with respect to the cookie dimensions.

The most commonly used method in current studies of flour fractionation was developed by MacRitchie (1985,1987). The author obtained gluten by manual mixing of the dough in distilled water or by using a mechanical washing device after extracting the lipids with chloroform (Figure 1.8).

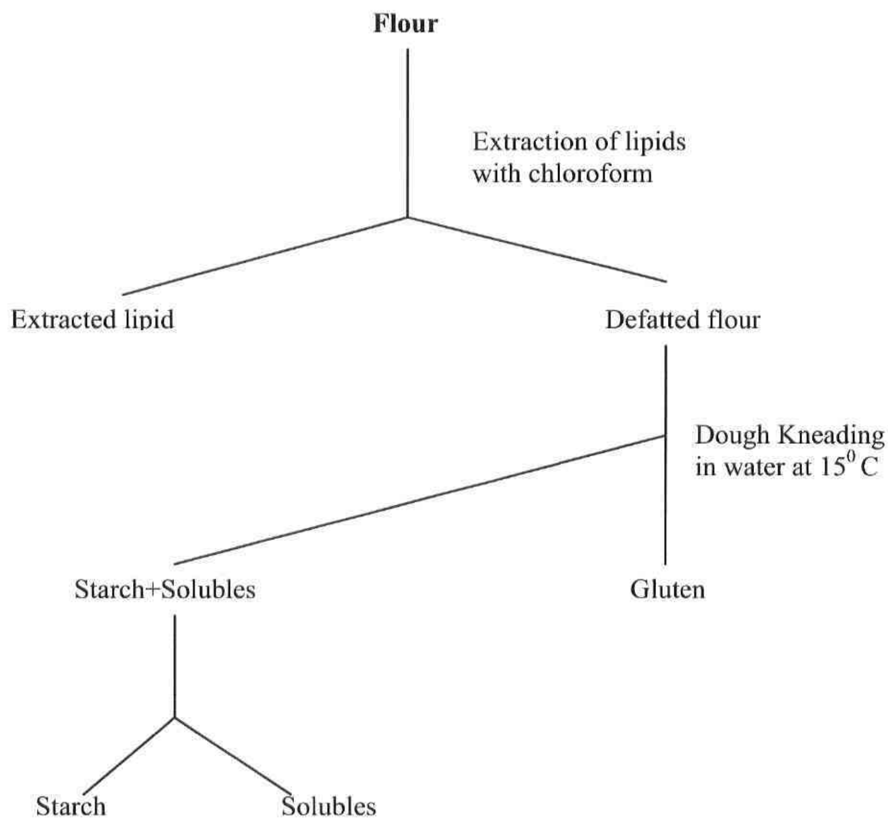
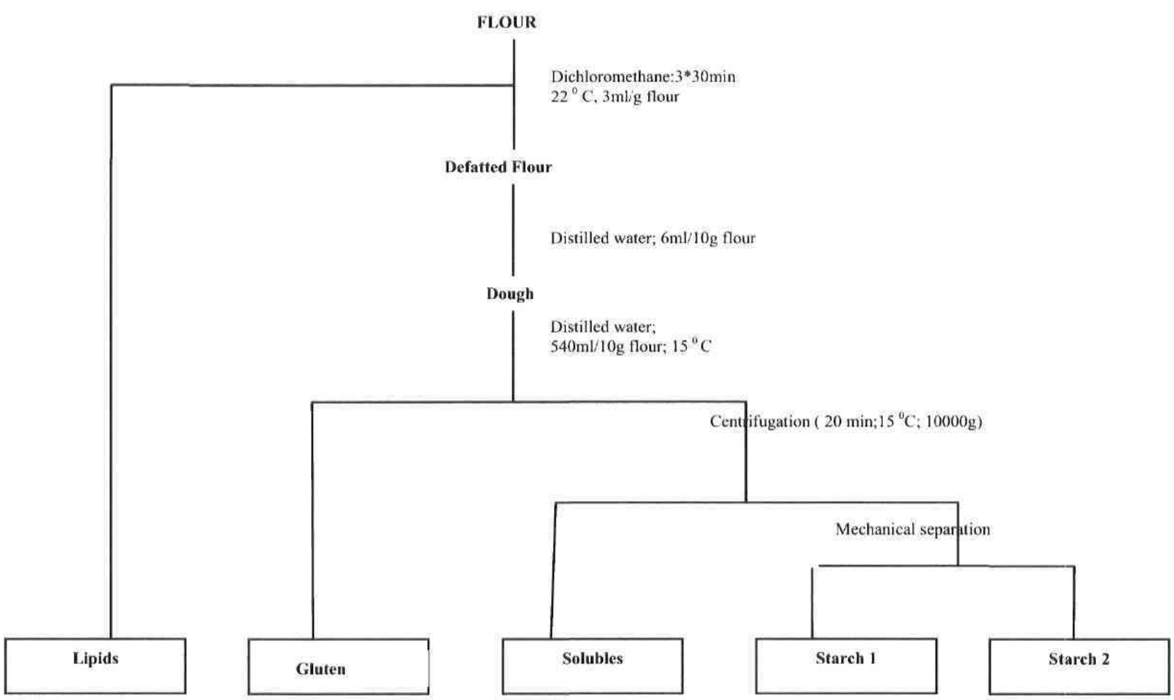


Figure 1.8: Component separation diagram for flour reconstitution studies (MacRitchie, 1985)

The fractionation technique used by MacRitchie involves the addition of 200 ml of distilled water at 15^0C per 100 g of flour, which is repeated six times until gluten is obtained with a ~70% to 80% protein content and 1,200 ml of wash waters which, after centrifuging (10,000 rpm, 20 minutes, 15^0C), produce starch and the solubles fraction. The starch contains roughly 0.5% lipids and a small amount of proteins adhering to the surface of the granules, which are extracted by several washings with a diluted base or acid. Starch is generally separated by centrifugation, which results in two layers: an upper, greyish-coloured gelatinous layer, the starch tailings, consisting mainly of damaged starch, insoluble pentosans and glycoproteins, and a lower layer, corresponding to the white prime starch. It should be noted that, during kneading, higher temperatures may induce proteolytic activity in gluten, while low temperatures may reduce dough cohesion.

Wu and Hoseney (1990) and Krishnarau and Hoseney (1994 a,b) used another fractionation technique involving mixing 300 g of flour with 60% distilled water for one minute in the blender (Waring Blender) and washing the dough thus formed with 800 ml of distilled water, four times. Once the gluten was extracted, the wash waters were centrifuged (20 minutes, 1,250 rpm) to recover the soluble fraction and starch. The fractions obtained were then freeze-dried and tempered at 90% relative humidity to bring the moisture content to roughly 14%. Petrofsky and Hoseney (1995) separated the prime starch and wheat gluten by using aqueous fractionation, without using a lipid extraction step in order to study the rheological properties of dough made from flour reconstituted from gluten and starch from various sources (oats, wheat, rice). The objective of the work was to study the starch-gluten-water interactions in dough and prevent interaction of other flour constituents such as pentosans. The technique used consisted of mixing 500 g of flour with 275 ml of distilled water in a Hobart mixer for ten minutes, washing the dough with 500 ml of distilled water, mixing it manually to separate the gluten from the wash waters, freezing the product at -18 ^{0}C and freeze-drying it, and centrifuging the wash waters for 30 minutes at a speed of 500*g. The supernatant obtained consists of the soluble fraction and the two-layers consisting of prime starch and starch tailings which could be isolated with a spatula. Starches isolated from different wheat cultivars and mixed with the same gluten generated doughs with very different characteristics. Miller and Hoseney (1999) continued their work on improving the efficiency of this process, in order to obtain gluten, the solubles fraction and starch to study the dynamic rheological properties (G', G'' and tan δ) of gluten-starch-water doughs. Their technique consisted, in short, of mixing 500 g of flour with 320 ml of distilled water using a mixer (National Manufacturing Division-TMCO, Lincoln, NE), followed by mixing of the dough with 500 ml of distilled water to obtain the gluten. Subsequently, the wash waters were centrifuged at 500 *g for 30 minutes to separate the solubles fraction from the starch.

Gra∃berger et al. (2003) tested various non-polar solvents to extract lipids from the flour (petroleum ether, diethyl ether, dichloromethane, chloroform and ethyl acetate). Dichloromethane (0.9 g/100 g of flour) and chloroform (0.94 g/100 g of flour) were found to be the best solvents for extracting nonstarchy lipids. Dichloromethane is preferable to chloromethane because of its low toxicity and its ability to preserve the rheological properties of the dough. The fractionation procedure (Figure 1.9) was as follows: mixing 200 g of flour with 300 ml of dichloromethane using a Polytron at ambient temperature for 30 minutes at 22 ^{0}C to extract the lipids. The mixture is filtered and the residues undergo two additional extraction processes. Lipid extracts are recombined and evaporated to obtain an approximate volume of 300 ml. The solution is subsequently stored under a nitrogen atmosphere (N_2) at -20 ^{0}C before use. Once the lipids are removed, the flour is allowed to air-dry overnight.



.Figure 1.9: Fractionation diagram for wheat flour (Gra∃berger et al., 2003)

3.4- Fractionation technical issues

All the research activities on fractionation illustrate the multitude of potential processes that can be used to recover the various flour fractions as well as the efforts put into improving these processes in order to restore fraction functionality. Despite this, a number of authors cite numerous problems encountered in separating flour components:

-the use of butanol to extract lipids leads to complex formation with wheat starch, an interaction that affects the functionality of the resulting extracted flour by decreasing bread volume (Finney et al., 1976). In addition, butanol causes changes to the structure of gluten proteins, likely modifying dough development and decreases the cookie diameter (Finney et al., 1975; MacRitchie, 1985).

-the loss of configuration and structural modifications of certain proteins with solvent used (Weegels, 1996a, b). The functionality of the vital gluten is thus modified (ex. hydration rate, water absorption, reduced gluten vitality)

-the aggregation of protein fractions: gliadins, for example, are irreversibly altered after precipitation with aqueous ethanol. In addition, differences are observed in the composition of protein fractions depending on the solvent selected. This explains why some authors draw different conclusions on the role of glutenins and gliadins in breadmaking (Weegels, 1998a,b).

-the kneading of the flour-water mixture may cause the aggregation of gluten proteins by mechanical denaturation. Noncovalent interactions develop between the different components of the flour upon hydration (Laszitity, 1984; Masi et al., 1998). When the dough is subject to external forces, physical cross-linking and the weak bonds keeping components together may break and reform, allowing the dough to partially or completely relax under pressure.

- the cleavage of disulfide bonds is responsible for changes in the rheological properties of doughs. This indicates that the protein chains have been modified and also implies changes in protein conformation and aggregation; and thus increased solubility and reduced elasticity (Wu and Hoseney, 1990).

-the difference between original and reconstituted flours; e.g. decreased volume and size of the end-products, can also be attributed to irreversible changes occurring during fractionation, such as the oxidation of redox systems under anaerobic conditions during fermentation and breadmaking, so that further softening process become impossible in the dough made with the reconstituted flour (Köhler et al., 1990). The addition of glutathione (GSH) can reduce these differences.

-the state of dispersion of the components in the native and the reconstituted flours is different (Tolstogusov, 1997, 2002).

-new distribution of water among the flours constituents (ex. protein, pentosans, starch).

-the possible inactivation of proteolytic enzymes during fractionation, so that the gluten breakdown during mixing could not take place (Köhler et al., 1999).

Although fractionation-reconstitution and supplementation methods are the most direct way of demonstrating cause and effect relationships involving quality, the results still remain too contradictory to draw relevant conclusions on the determining role of protein fractions such as the high and low molecular weight fractions in hard wheat on the quality of bread-making products. In the case of soft wheat flours, no studies have been reported on the role of these various protein sub-fractions in biscuit making. However, the role of prime starch, starch tailings, gluten, the soluble fraction and lipids appear to be better defined in cookie- making quality, but only on the spread and surface appearance.

Recently, an excellent overview of the main processes of fractionation and the factors affecting them describe several aspects that deserve attention when using the physical approach to isolate starch and gluten (Van Der Borght et al., 2005).

4- Physicochemical aspects of flour functionality

4.1- Physical properties

4.1.1- Water absorption capacity

Doesher and Hoseney (1985) reported that the flour moisture content affects biscuit symmetry and surface appearance. Any increase in flour moisture content is generally accompanied by an increase in the surface cracking of the cookies. According to these authors, the moisture content of flour is more important than the total water content in the recipe since cookie recipes prepared with the same quantity of water show different surface characteristics due to variations in flour moisture content.

It is well known that high-quality flour used in cookie and cracker making must not be excessively hydrophilic. This ensures that water will be more available to transform the sugar into syrup and the viscosity of the dough will decrease during baking, promoting the spreading of the cookies (Slade and Levine, 1994a, b). When used to make cookies and crackers, flours with high absorption capacities require longer baking times and also yield harder finished products.

According to Bartolucci and Launay (2000), the dimension and density attributes of semi-sweet biscuits are linked to water-absorbing components. The hydration potential of flour depends mainly on the quantity of certain components with water-absorbing properties, such as proteins (~2.8 g of H_2O/g of gluten), damaged starch (~1.5 to 2.0 g of H_2O /g of starch) and pentosans (~10 g of H_2O/g of pentosans) (Bushuk, 196; Bloksma and Bushuk, 1971; Hoseney et al., 1988). More recently, Berton et al (2002) provide higher values for the water-absorpting properties of damaged starch 200-430%, proteins 114-215% and pentosans 500-1500%. This gives an idea of competition for the available water among the different flour components, which can have a significant impact on the viscoelastic properties of the protein network (Biarnais, 1987). The greater the water retention capacity, the less the cookie spreads (Fuhr, 1962). The Alkaline Water Retention Capacity (AWRC) physicochemical test (AACC 56-10) was developed to predict cookie quality by quantifying total flour absorption using a sodium bicarbonate solution. Studies by Yamazaki (1953 and 1954) revealed the ability of the AWRC to predict cookie diameter; a negative correlation was found between diameter and the AWRC value, whether involving different varieties ($r = -0.95$, $n = 11$) or the same variety ($r = -0.63$ to -0.85). These coefficients were better than those obtained for protein content using the same samples. These results have been confirmed in more recent studies (Nemeth et al., 1994; Labuschagne et al., 1996 [r between -0.4 and 0.55] and Abboud et al., 1985 [$r = -0.78$, $n = 14$, $p < 0.01$]). Donelson (1990) showed that chlorination of flour with sodium chloride (1-2 g/kg of flour) using the method developed by Gaines and Donelson (1982) reduced the cookie diameter, which can probably be attributed to an increase in the AWRC value of the flour.

This method has gradually been replaced with the Solvent Retention Capacity (SRC) method (AACC 56-11) to more accurately quantify the impact of the main flour components that contribute to flour functionality. The test is based on the differential solvent absorption of damaged starch (5% sodium bicarbonate [Na_2C0_3] solution), pentosans and, to a certain extent, gliadins (50% sucrose solution) and gluten (5% lactic acid solution) and total absorption (in distilled water), which combines the absorption of all the components mentioned (Gaines 1990). This method is commonly used in the cookie making industry to better identify components that vary from one flour batch to another, from region to region, or from mill to mill. It is also used as a practical test to predict the performance of cookies and crackers (Slade and Levine, 1998, 2000; Souza, 2000) and as selection criteria for cultivars (Bettge et al., 2002; Guttieri et al., 2002). Moreover, the SRC test highlights aspects of the chemical make-up of the flour that assist in estimating the processing and baking parameters. For instance, knowledge of the specific source of viscosity of the dough is helpful in predicting the overall texture of some cookies. Thus, the SRC test provide some assistance in defining the origin of the viscosity, such as identifying whether viscosity originates from protein characteristics (Lactic acid SRC) or from pentosans content (SRC Sucrose). Decisions in obtaining flour with the appropriate processing and baking characteristics can be assisted by profiling this test (Guttieri et al., 2002)

Protein quality is generally linked to the genotype (cultivars), while protein quantity is associated with phenotypic factors (environment and fertilizer input). The amount of damaged starch is associated with the degree of milling, and pentosans content is controlled by genetic and environmental factors. The industrial experience of Nabisco and Pillsbury confirms that, in order to avoid lack of uniformity in cookie dimensions, it is better to have an overall SRC value for total absorption below 51% for each day of production rather than to have a value of 40% for some batches of flour and 55% for others. Although high absorption values of lactic acid solution are desirable, any value over 90% must be quickly dealt with by the formulator, since there is a risk of excessive elastic retraction in the dough. For absorption of the sucrose and sodium bicarbonate solutions, low absorption values and minimum variations between batches of flour are desirable (Souza, 1998; Slade and Levine, 2000).

Other diagnostic tests, based on the SRC principle, also allow the influence of other components to be confirmed, such as gliadins (70% ethanol), glutenin macropolymers (0.75% sodium dodecyl sulfate or SDS) and macropolymers of glutenins involving a disulfide mediated network (0.75% SDS with 0.03% sodium metabisulfite). These tests were recently developed by Nabisco to improve characterization of the functionality of soft wheat flours in biscuit making (Slade and Levine, 2000). To date, there have been no scientific studies on the results of these latter tests using soft wheat flours.

4.1.2- Glass transition and plasticization by water and fat

The quantity and quality of the water incorporated into dough are of prime importance in obtaining doughs with the appropriate rheological characteristics for biscuit making. There are three main types of water in dough: bound water, weakly bound water and free water. Bound water corresponds to the monomolecular layer of water attached to the polar groups, particularly COO^- and $NH3^+$ groups in proteins and OH^- groups in starches, which have high absorption energy (1–15 kcal/mole); so it is quite difficult to remove away the water from this layer. In addition, the water in this monomolecular layer cannot be frozen. In wheat flour dough, the quantity of water that cannot be frozen represents roughly 0.3 g/g of dry matter and this water is practically unavailable as solvent or reagent (Bloksma and Bushuk, 1971). In the state of weakly bound water, successive layers are attached to the first layer through hydrogen bonds. This water accounts for most of the hydration sphere of soluble components (proteins, salts, carbohydrates, etc.). Despite attempts to quantify weakly bound water using various instruments (NMR, DSC, dielectric properties), many authors have obtained rather inconsistent results. According to Cheftel and Cheftel (1992), there is no fundamental difference between weakly bound water and free water, in which the activity is very close to pure water (particularly its availability as solvent or reagent). In general, free water and weakly bound water can quickly reach equilibrium. In dough, water forms a continuous phase whose distribution is determined by the components present as well as their quantities (MacRitchie, 1976). Inside the dough structure, water exists as bound water (0.4–0.5 g of water/g of flour) and free water. The bound water contributes, just as other molecules do, to the supramolecular organization of the dough network. As previously mentioned, damaged starch, proteins and pentosans have different water-absorbing properties. Based on the hypothesis that water is distributed among the different components in flour in proportion to their water-absorbing capacity, the following results would be obtained: 46% of water is bound to starch (15% to damaged starch), 31% to gluten and 23% to pentosans. This observation still has to be confirmed, since these simple calculations do not take into account the interactions between these components. These results do seem to confirm, however, that the quantity of water to be incorporated must be adapted to the flour's characteristics, in other words, to the concentrations of these three main flour components (starch, gluten, pentosans).

From a rheological point of view, water acts as a plasticizer in dough, in the sense that due to its low molecular weight it promotes the mobility of macromolecular chains through an increase in free volume and a decrease in viscosity (Levine and Slade, 1990). This concept of water as a plasticizer complements the more traditional concept of free and bound water. The first fraction of absorbed water (bound water [unfreezable water], weakly bound water or free water) can either be slightly plasticizing or antiplasticizing depending on the polymer's degree of compatibility with water, which is associated mainly with the ability of water to lower the glass transition temperature of the polymeric constituents; i.e. flour hydration reduces the temperatures at which flour polymers, proteins and starches in particular, go from a glassy to a rubbery state according to the glass transition (Tg) theory. Based on this concept, at temperatures below the Tg temperature, molecules remain immobile and only noncooperative local motions of low amplitude can occur. Above the Tg, high-amplitude cooperative molecular motions occur involving large chain segments or entire molecules (Levine and Slade, 1990).

The transposition of this concept, which was initially developed to study synthetic polymers, to the study of flour components (starches and proteins) and cereal-based products (bread, biscuits, pasta and biofilms) dates back around 20 years. It has proven particularly useful in understanding the evolution of the physical properties of doughs: water, which can act as a plasticizer, induces a strong decrease in the glass transition temperature (Tg). However, with complex food systems such as starch based-products, the glass transition can occur over a wide range of temperatures. In addition, the mechanical properties of starch and proteins are controlled by the transition from a vitreous state to a rubbery state, as revealed by differential scanning calorimetry (evolution of specific heat) and dynamic thermomechanical analysis (the measurement of viscoelastic properties, based on the fact that the glass transition is accompanied by changes in the values of G', G'' and tangent * as a function of temperature). Given equal hydration, the temperature of the, in situ, glass transition of gluten in flour is roughly 30 ^{0}C higher than in isolated gluten, probably due to the interactions between gluten and other flour components and variation in water distribution among flour components of the composite network. Ingredients used in biscuits also modify the Tg value of gluten: the addition of glucose syrup or sodium carbonate as a leavening agent increases the Tg value very significantly, even above the initial Tg value for dry gluten (Doesher et al., 1987a,b).

It should be noted that, at the beginning of the dough mixing process, the dough tends to become sticky but this stickiness decreases gradually as the dough develops and then disappears altogether. The physicochemical mechanism behind this phenomenon is unknown although it has been attributed to the respective proportions of free and bound water in the dough and the hypothesis is that an excess of free water causes this characteristic. However, the quantity and nature of proteins, soluble and insoluble pentosans and the damaged starch content are other parameters that could contribute to dough stickiness. When biscuit dough with low water content is mixed, the flour proteins absorb water without changing state, since they remain below the Tg temperature. During the temperature rise that occurs during baking, the Tg value is reached and exceeded, and the protein matrix continues to form as the dough develops. The Tg value for biscuit doughs also depends a great deal on the nature and the concentrations of ingredients, such as sugar and fat, in the recipe as well as the hydration rate.

Researchers who are studying the spreading mechanisms of cookie during baking with both the soft and hard wheat flours draw different conclusions about how and why cookies spread. Heated cookie dough made with soft wheat flours have lower viscosity than dough made with hard wheat flour (Abboud et al., 1985b). The hard wheat doughs ceases spreading more rapidly during baking likely due to their higher viscosity at low temperature comparatively to doughs produced with soft wheat flours.

Doesher et al. (1987) observed that protein content is associated with the rate of dough spreading during baking. The rate of dough expansion and the time the dough set during the baking period determine the final cookie diameter. According to the same authors, the Tg value for hard wheat flours was lower than that for soft wheat flours. In addition, they found that, when the Tg value is reached, the gluten swells and forms a network in the form of a continuous phase, which decreases the Aw value for the dough and rapidly increases dough viscosity, which in turn influences the gravity of the dough and halts spreading. The gluten in the sugar-snap cookie dough is not developed into a web during mixing, thus the flour particles in the dough remain intact and discontinuous while the continuous phase corresponds to the sugar syrup. During baking, the gluten goes through an apparent Tg, thereby gaining mobility that allows to interact and form a network. The viscosity of the continuous gluten network is sufficient to stop the flow of the cookie dough and the apparent Tg of cookie made with hard wheat flour is lower than the cookie made with soft wheat flour (Miller et al., 1996), but the reason for these differences remains unclear. They also suggest that the set time is determined by the glass transition temperature for gluten proteins. Doughs with the lowest Tg temperatures produce cookies with smaller diameters.

Since cookies doughs are plasticized by water, Tg values of 155^{0}C, 55^{0}C and 12^{0}C have been reported for dough hydration levels of 3.1%, 6.2% and 12%, respectively (Nikolaidis and Labuzza, 1996). Levine and Slade (1990) confirmed that the Tg value for gluten proteins could govern changes in cookie size during baking. However, Slade and Levine (1994a,b) suggested that, in sugar-snap cookies, the glass transition of gluten is not crucial in the baking mechanism and cannot be used to explain the phenomenon of setting. According to these authors, cookie dough made with hard wheat flour forms a three-dimensional network which undergoes elastic expansion during baking and then collapses when the Tg value is exceeded. Doughs made from soft wheat flour are rubbery and do not form a functional network, but undergo viscous expansion and elastic recovery followed by a structural collapse when the Tg value is exceeded. They attributed these differences of the flour behaviour to the thermodynamic properties of the gluten: the latter's behaviour before the glass transition is similar to that of a material made rubbery by thermosetting or to that of a melted polymer, depending on whether the flour is of mediocre or of satisfactory quality, respectively. Gluten in hard wheat flours undergoes thermosetting in the dough at lower temperatures than in soft wheat flours, which remain thermoplastic longer during baking, thus promoting spreading of the cookies.

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4.1.3- Particle size distribution

The particle size distribution is crucial in the formation and baking of the dough, but the way in which it is obtained is also significant. During the separation of flour of different wheat varieties covering a wide range of wheat hardness into two fractions using a 325-mesh sieve, Yamazaki (1959a, b) also observed, along with a difference in particle size distribution, a physicochemical difference in flour composition. The biscuit-making potential of each flour was also different: the finest fraction, with a lower protein and ash content, produced cookies with a greater diameter than the coarse fraction, which contained starch with a greater quantity of hydrophilic components (ex. pentosans) and proteins, resulting in smaller biscuits. Maximum spreading could occur when both fractions were added in equal quantities. More recently, Badei et al. (1992) reported that the finest fractions result in cookies with superior organoleptic qualities (texture, colour and taste), while others researchers have reported that these fractions resulted in cookies with greater diameters (Yamamoto et al., 1996).

Particle size distribution determined by laser diffraction can distinguish flours made from hard, non-crunchy wheats (normal distribution with 25% of particles of a diameter less than 50 :m) from flours made from soft, crunchy wheats (bimodal distribution with 50% of particles with a diameter less than 50 :m) (Hareland,1994).

Considering that a gram of flour has a total peripheral surface area of roughly 1 m^2 accessible to water (Gutierriez et al., 1999), it is easy to understand that particle size distribution can play an important role in the kinetics of dough hydration, since a greater contact area promotes more rapid hydration.

On the other hand, particle size distribution may have an opposite effect depending on the type of biscuit being made: finer particle size distribution results in lower density in rotary molded cookies, but an increase in density in semi-sweet biscuits such as Rich Tea biscuits (Wainwright et al. 1985). Therefore, it is essential not to extrapolate the observations on the impact of particle size distribution from one type of biscuit to another. Cookie diameter decreases with the number of passes through the break and middlings rollers, which is undoubtedly linked to the greater quantity of damaged starch, which increases the flour's hydration capacity. The contribution of particle size distribution to hydration properties and cookie diameter still remains difficult to ascertain since changes in distribution are also associated with modifications in biochemical composition. Although it is recognized that water absorption capacity increases with a decrease in particle size, this increase in water absorption could also be associated with an increase in damaged starch or with the presence of pentosans and other components on the surface of the particles rather than with an increase in specific surface area (Gutierrez et al., 1999).

4.2- Biochemical components

The basic composition of flour shown in Table 1.2 eloquently demonstrates the complexity of the flour system and the potential interactions between these biochemical components. Flour components are a complex mixture of molecules: starch, proteins, lipids and pentosans. There is a paucity of studies on composition-functionality relationships in biscuit flours and those studies carried out basically involve sugar-snap cookies or the AACC 10-52 recipe. As previously discussed, the influence of these components in this cookie recipe is greatly reduced compared with other formulations due to its high sugar and fat content which masks and distorts the effects of flour (Slade and Levine, 2000). In view of the paucity of results on biscuit manufacturing products, numerous studies published in bread-making were used to predict the effects attributed to components such as proteins, lipids and pentosans. However, the results of such studies must be viewed with caution since the composition of bread doughs (quantities of flour and water) amplifies the effects these components have on dough and end-product quality attributes.

Table 1.2: Biochemical composition of flour and its main components (Atwell, 2001a)

Property	Percent
Moisture	14 (of flour)
Protein	7-15 (of flour)
Osborne classification	
Albumins	15 (of protein)
Globulins	3 (of protein)
Prolamin (gliadin)	33 (of protein)
Glutelin (glutenin)	16 (of protein)
Residue	33 (of protein)
Gluten	6-13 (of flour)
Gliadin	30-45 (of gluten)
Glutenin	55-70 (of gluten)
Starch	63-72 (of flour)
Amylopectin	75 (of starch)
Amylose	25 (of starch)
Nonstarchy polysaccharides	4.5-5.0 (of flour)
Pentosans/ hemicellulose	67 (of NSP)
Insoluble	67 (of pentosans/hemicellulose)
Soluble	33 (of pentosans/hemicellulose)
Beta glucans	33 (of NSP)
Lipids	1 (of flour)

4.2.1- Lipids

Although cookie recipes contain high levels of fats (10%–40% on flour basis), native lipids in flour (~1.0%–2.5% of flour) have a unique functionality (Le Roux, 1987). Native lipids can be divided into two main groups (Figure 1:10): (1) free polar lipids made up of complex lipids, glycolipids and phospholipids and free nonpolar lipids (tri-,di- and monoglycerides, fatty acids, hydrocarbures and sterols) and (2) bound polar lipids (glycolipids and phospholipids) and nonpolar lipids (Pomeranz 1977;Le Roux,1987). These native lipids are also sometimes classified in the literature as storage lipids, which are nonpolar or slightly polar (tri-, di- and monoglycerides), and structural lipids, which are polar (glycolipids and phopholipids) and have different functionalities depending on their polarity.

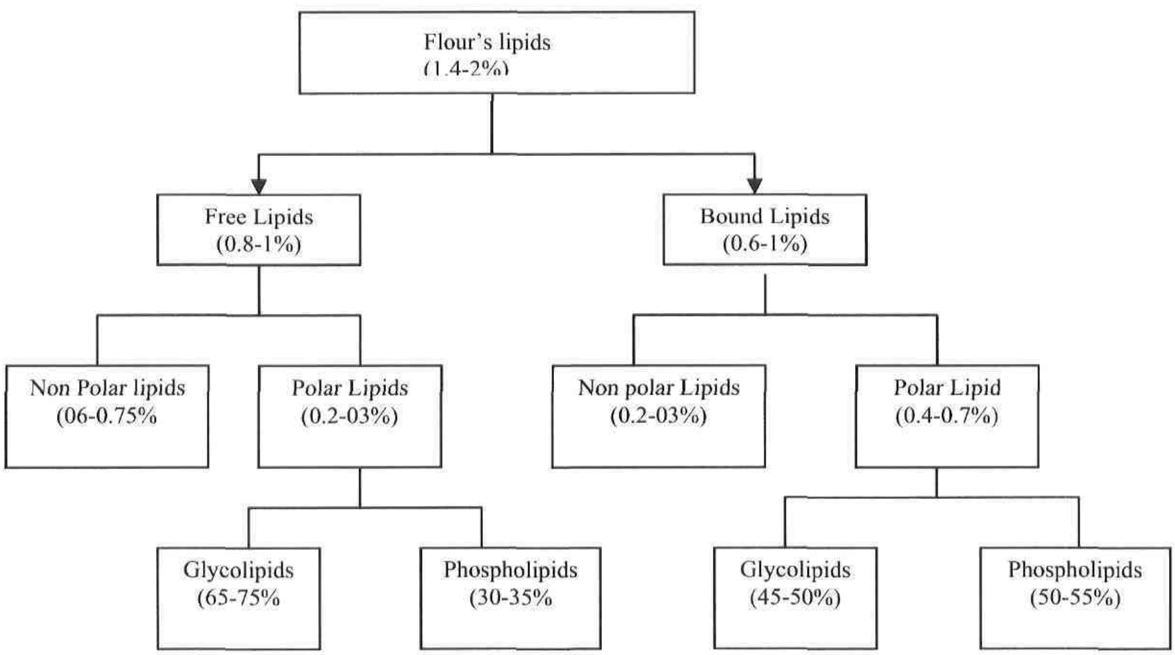

Figure 1.10: Classes of the main lipids in flour (percentages indicated in relation to weight of flour); Feillet (2000).

Interactions between glycolipids (monogalactosyl and digalactosyl diglycerides) and wheat flour molecules and complexes formed of glycolipids and starch, gliadins and glutenins have been described by Pomeranz (1977) and Hoseney et al. (1997). The latter studies demonstrated the presence of hydrophobic interactions between glycolipids and glutenins and hydrogen bonding between gliadins and glycolipids.

Free polar lipids are recognized to be essential in the complete restoration of flour functionality in sugar-snap cookie baking performance of wheat flour, owing to the presence of digalactosyl diglycerides and phosphatidylcholine (Clements and Donelson, 1981). Free polar lipids can be extracted using petroleum ether, while polar solvents such as alcohol are required to extract bound lipids. On the other hand the polar bound lipids are mainly associated with the storage proteins and contribute to the structure of the cell walls.

Native lipids are associated with starch, via the amylose complex (monoacyl lipids) and also inserted into the protein matrix. Flours with lipids eliminated by extraction with alcohol produce smaller cookies with a darker colour and brittle internal structure due to the formation of many large air bubbles in the product, compared with the cellular structure with small, regular bubbles produced using ordinary flours (Cole et al., 1960; Kissel et al., 1971, Yamazaki and Donelson, 1976; Clements., 1980). Such changes in the internal cellular structure of cookies baked with defatted flours could be attributed to the collapse and coalescence of air bubbles during the expansion of the dough. Cole et al. (1960) demonstrated that cookie diameter decreased as a function of the quantity of lipids extracted. Kissel et al. (1971) observed that any increase in free lipids added to defatted flour (up to three times the quantity of the initial lipid content) is accompanied by improved cookie quality. According to the same authors, the polar and non polar fractions in themselves are not sufficient to restore the quality of defatted flour. The presence of both fractions therefore appears essential in restoring flour quality. On the other hand, the work of Clements and Donelson (1981) suggests that flour functionality is associated exclusively with polar lipids; the addition of digalactosyl diglycerides or phosphatidylcholine alone appears to restore the functionality of defatted flours, while the nonpolar fraction probably plays a negligible role. These divergent findings can be explained by the various lipid extraction and reconstitution methods used by different authors and the differences in experimental protocols, particularly polarity differences in solvents, and methodology that may result in the denaturation of some flour components. It should also be noted that lipid surface tension is sometimes cited to explain the lipid's role in cookie structure. Native lipids exist in the form of vesicles that are physically trapped within the gluten matrix. The gluten can therefore be considered as a system that contains stabilized micro-emulsions. The role of native lipids, particularly polar lipids, is basically associated with the stability of gas cells in the dough, which, due to the stabilization of the interfacial liquid film, can be stretched without breaking. The molecules orient themselves to form lipid monolayers at the water-gas interface. The surface lipids of starch granules are important to dough rheology. The surface lipids of corn starch increase the interaction between gluten and corn starch, whereas surface lipids of wheat starch decrease the interactions (Sipes, 1993).

4.2.2- Proteins

According to Atwell (2001a), proteins make up 7%–15% of flour (14% moisture basis). Traditionally, flour proteins are divided into two categories, as illustrated in Figure 1.11, storage proteins or gluten (80%–85% of flour proteins) and cytoplasmic or functional proteins (15%–20% of flour proteins).

Cytoplasmic proteins comprise of albumins, globulins, peptides and amino acids. They do not alter the composition of gluten but, due to their enzymatic activity (lipoxidases, proteases, pentosanases and amylases), can influence its viscoelastic properties (Rouau 1996). Lipoxidases, for example, can catalyze the free radical-mediated reactions resulting in an unstable hydroperoxide radical that contributes to the formation of disulfide bonds and promotes oxidation of gluten, thus improving its tolerance to mixing (Potus et al., 1996). Amylases also play an important role in breadmaking but not in biscuit and cookie making due to the absence of fermentation and because of the high fat and sugar content of the products. Wheat protein classification was initially based on protein solubility characteristics, as shown in the diagram by Osborne (1907). In this classification, four groups can be distinguished: albumins, which are water-soluble; globulins, which are soluble in a saline solution (0.5 N NaCl); gliadins, which are soluble in diluted alcohol

(70% ethanol); and glutenins, which are insoluble in the previous solutions but partially soluble in a 0.01 N acetic acid solution. These four protein groups make up on average ~15.0%, 3.0%, 33% and 16% of total proteins, respectively (Table 1.2). Residual proteins, which make up 33% of the total proteins, cannot be extracted using the above-mentioned solvents.

Glutenins, which are not extractable by acetic acid, are often referred to as protein gel or gluten macropolymer since they form a gel on the surface of starch after centrifugation in acetic acid. The only way of extracting them is to use reducing agents. The development of more sophisticated methods of chemical analysis has demonstrated the limits of these extraction methods and their solubility may be modified depending on extraction conditions (Wieser et al., 1989).

The currently used definitions of glutenins and gliadins are based on their physicochemical properties and structural and genetic characteristics (MacRitchie, 1992). This classification takes account of the degree of protein polymerization while retaining Osborne's nomenclature. Under this system, proteins can be divided into various groups as shown in Figure 1.11:

-glutenins, or polymeric or aggregated proteins (MW > 100,000), which after reduction can be divided into subunits of two types according to their size as determined by SDS-PAGE: LMW-GS (low molecular weight glutenin subunits) and HMW-GS (high molecular weight glutenin subunits).The sub-units are released by disulphide reducing agents (Shewry, 1992). They contain high levels of proline and glutamine and low levels of charged amino acids. These multichains impart to dough its resistance to extension.

-gliadins, or monomeric proteins (MW = 25,000–75,000), which can be divided into four subgroups: T, (, ∀ and ß gliadins. The distinction between the subgroups is based on their increasing mobility during acid electrophoresis. The ∀, ß and (subgroups are very stable due to the presence of ∀ helices maintained by hydrogen bonds, while T gliadins have ß-bends instead. Similarly to the glutenins, they are rich in proline and glutamine. They may associate with one another or with the glutenins through hydrophobic interaction or hydrogen bonds. They act as plasticizers and promote viscous flow and extensibility. They are extremely sticky when hydrated. The disulphide bond in gliadins are exclusively intramolecular, while in glutenin, they are in both intra and intermolecular.

Shewry et al. (1992) established a different nomenclature for gluten that can be superimposed on the previous classification. Gliadins and glutenins are grouped together under the category of prolamins, due to their physicochemical properties and solubility in 70% ethanol after reduction. In turn, prolamins can be divided into three groups according to their sulfur amino acid content and/or molecular weight (Figure 1.11):

a) HMW (high molecular weight) prolamins;

b) sulfur-rich prolamins made up of ∀, ß and (gliadins and LMW-GS (these four groups have similar amino acid compositions); and

c) sulfur-poor prolamins, or T gliadins.

Overall, prolamins, which roughly combine gliadins and glutenins or storage proteins, contain 80%–85% proteins, 4%–9% lipids and traces of starch, fibres and sugars.

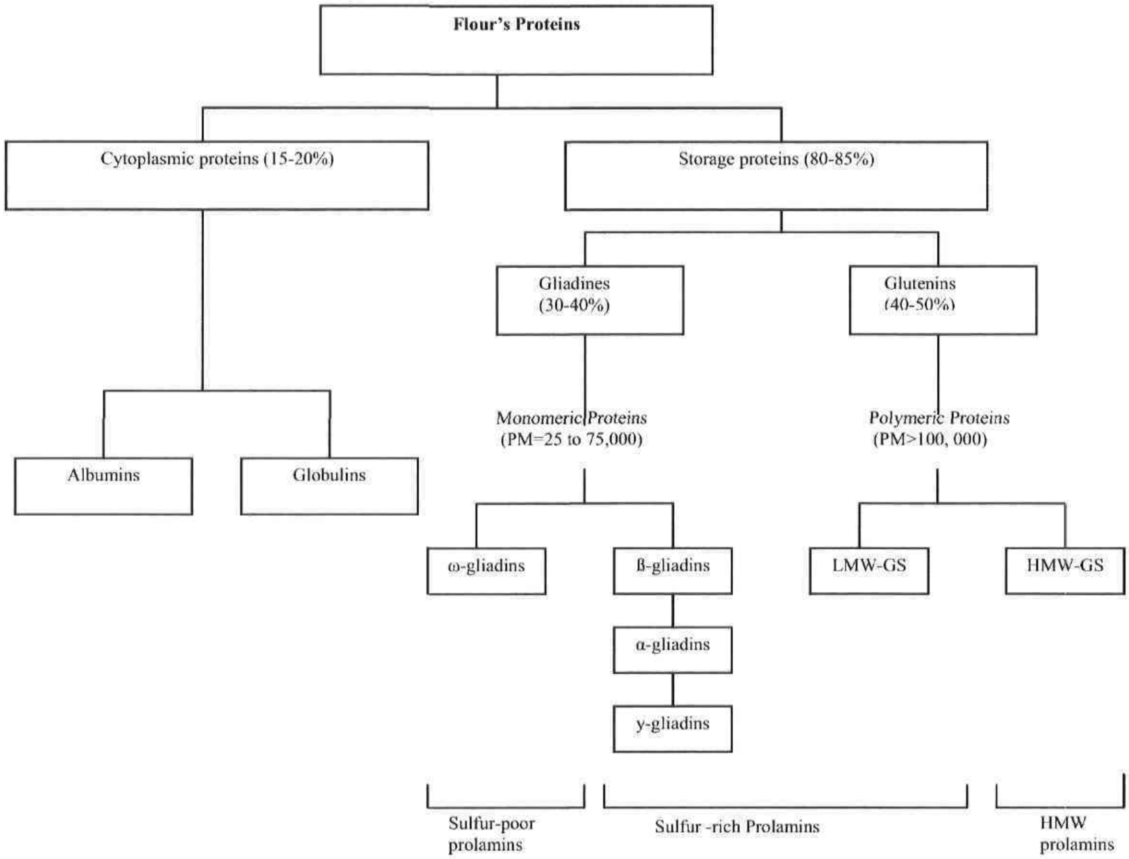

Figure 1:11: Composition of flour's proteins: correspondence between classification systems developed by Osborne (1907) and Shewry et al. (1992)

This system of nomenclature has been criticized since it does not take account of storage proteins' tendency to form inter- and/or intramolecular disulfide bonds, which determine their degree of polymerization, a key factor that distinguishes the functional properties of gliadins from those of glutenins. In fact, it is difficult to clearly distinguish among storage proteins using a single classification system (Ewart, 1972). According to Popineau and Denery-Papini (1996), certain characteristics of storage proteins—their molecular weight, amino acid composition, hydrophobicity and primary structure—allow these proteins to be better distinguished on SDS-PAGE (1.3). HMW proteins have the greatest molecular weights (100,000-160,000). The molecular weight of ∀, ß and (gliadins is similar to that of LMW-GS (30,000-50,000), while that of T gliadins is in the 60,000-80,000 range.

Table 1.3: Molecular weights of various wheat proteins (Popineau and Denery-Papini, 1996)

Characteristics	Gliadins			LMW-GS	HMW-GS
	ω-	α/ß-	γ-		
Apparent Mass (SDS-PAGE	60-80,000	30-40,000	30-40,000	30-50,000	100-160,000
Real Mass (Sequence)		28-35,000	31-38,000		67,000-880000

A general characteristic of prolamins is their high concentrations of proline and glutamine ; -gliadins have little or no sulfur amino acids or lysine. LMW-GS have almost the same amino acid profile as the ∀, ß and (gliadins. HMW-GS are poor in proline and rich in glycine compared with prolamins, which are rich in sulfur. In terms of hydrophobicity, HMW-GS have roughly the same hydrophobicity as Tgliadins but lower values than LMW-GS.

It should also be noted that gluten proteins contain large quantities of glutamic acid (~32%) and proline (~10%). Glutamic and aspartic acids occur most often in the form of amides (Popineau and Denery-Papini, 1996). Three groups of bonds are thought to be responsible for the viscoelasticity of dough: amides, sulfhydryl (SH) groups and disulfide bonds (SS) and hydrogen bonds.

An excellent review of Tatham et al. (1990) provides a valuable schematic representation of the primary structure of the various prolamins sub-group. The size of each of these domains varies depending on the type of storage protein. Moreover, these N-terminal and repetitive domain chain sequences are specific for each type of proteins. The N-terminal sequences and part of the internal sequences have been determined. The latter sequences suggest that the T gliadins also have N- and C- terminal regions and a central proline- and glutamine-rich domain (Popineau et al., 1996, Shewry et al., 1992, Tatham et al., 1990). These N-terminal and repetitive domain sequences are fairly specific for each type of protein. The schematic representation of the secondary structure of sulphur rich, of HMW glutenins confirms the presence of three structural domains: thee first (NH_2- terminal) is globular, consisting of ∀ helices and several cysteine groups; the centre consists of a repetitive ß-bend structure organized in a spiral, while the last (COOH- terminal) is similar to the first with only one cysteine group. The whole structure, roughly 500 Å in size, is rod shaped with a core that may be intrinsically elastic, which would explain the elastic behaviour associated with glutenins (Figure 1.12).

	Globular Zone	**Repetitive structure**	**Globular zone**
Residues No.	88-104	481-690	42
Cysteine No.	3 (type x) or 5 (type y)	0 or 1	1
Structure	NH_2		COOH
	α- helices	ß-bend structure	α- helices
Dimension	1.8 nm	~50 nm	1.8 nm

Figure 1.12: Secondary structure of high molecular weight glutenin subunits (Feillet, 2000)

Along with the albumins-globulins and prolamins, the presence of amphiphilic proteins should also be noted; they can be extracted with organic solvents or non-ionic detergents. Large quantities of polar lipids are found in the presence of these proteins. Within this protein group, puroindolines have exceptional foaming properties and can form a highly stable lipoprotein film at air-water interfaces (Compoint et al., 1994).

The aggregation of polypeptide units in glutenins occurs basically through S-S bonds, noncovalent hydrogen bonds and hydrophobic interactions. These aggregates have a molecular weight ranging from a few hundred thousand to several million; the largest aggregates form an insoluble gel after the extraction of soluble proteins in sodium dodecyl sulphate (SDS). They are referred to as polymeric glutenins or glutenin macropolymers (i.e., insoluble glutenins or glutenin aggregates). Several models of aggregation between HMW-GS and LMW-GS have been proposed and are discussed in the manual by Feillet to explain the possible structural gluten models of Kahn and Bushuk (1979); Graveland et al. (1985); Gao and Bushuk (1992). The diversity of these models testifies to the complexity of the hypotheses put forward to explain the formation of these aggregates. However, all the models recognize the fact that glutenins are composed of linear chains in which each subunit is able to establish two covalent interchain disulfide bonds.

Several models have been proposed to explain gluten structure. According to Popineau (1996) and Miflin et al. (1983), who have synthesized these hypothesis, the physicochemical properties of gluten fit well with a model in which the elastic behaviour is likely due to the presence of long linear polymers (glutenins) formed from high molecular weight subunits, joined by intermolecular disulfide bonds. The cross-linking of these polymers, their rearrangement and movements with respect to one another and the presence of smaller molecules associated through hydrophobic bonds (gliadins) allow viscous flow. Figure 4.5 shows the association capacity of gliadins with glutenins through hydrophobic interactions.

Macropolymers, also called protein gels, are used by some authors as a tool in predicting bread quality. It is not so much the quantity of the gel but rather the elastic modulus (the rate of destruction of the protein gel during kneading) that provides a useful way of determining bread quality (Pritchard and Bhandary, 1992) Bread quality has also been shown to be associated more with allelic variations in LMW proteins (including undoubtedly gliadins) than with those in HMW-GS. Some LMW proteins and various LMW-GS act like cysteine in promoting reassociation of the glutenin macropolymer (Pritchard et Brock, 1994).

Since the quantity of non-extractable or insoluble glutenins is fairly closely associated with bread quality (Weegels et al 1996a,b; Sapirstein and Johnson 2000), it can be assumed that a better understanding of the kinetics of their evolution could facilitate the use of the predictive ability associated with this fraction. Non-extractable glutenins tend to decrease during kneading (Weegels et al., 1996a), which could be explained by two potential mechanisms: the disassociation of the protein structure under mechanical forces (Tsen, 1967) and the depolymerization of glutenins through S S-S H exchange reactions under the action of intensive kneading (Tanaka and Bushuk, 1973). However, non-extractable glutenins increase slightly during the dough phase, which suggests a reassociation is taking place (Weegels et al., 1996 a, b).

It is possible to influence positively or negatively the rheological properties of the dough by adding reducing agents or by blocking the SH groups. Addition of oxidizing agents such as bromate (20-50ppm) can also influence the rheological behaviour of the dough; the quantity of oxidizing agents is in relation to the protein content, to the SH and SS groups. The interactions between the SH and SS bonds or the ratio SH-SS determine the quantity of oxidizing agents to be added (Weegels,1996a)

To explain the influence of protein content on the quality of different types of cookies, it is preferable to treat this subject separately owing to the great difference in sugar and fat content in cookie and biscuits recipes. For biscuit recipes, Wade (1972a) states that a minimum protein content of 7.5% is required to ensure quality (cookie's firmness and good surface appearance). Firmness increases with the protein content, particularly at levels above 10%. These observations concur with previous work by Kiger and Kiger (1967), if flour is too strong, the elasticity of the gluten causes the dough to retract, which has major consequences on the manufacturing operation (.i.e. biscuits that are too small in size and too thick). Branlard et al. (1985) showed that the most reliable chemical parameter related to the technological value of flour for biscuit making was protein content, the negative impact of which can explain 60% of variations in biscuit quality. However, other studies on dry biscuits do not seem to have raised the importance of the quantitative importance of proteins.

Several studies refer to the negative impact of protein content on spreading in sugar-snap cookies (Cottenet, 1986; Gaines, 1990 and1991). Kaldy and Rubenthaler (1987) and Kaldy et al.(1993) found a negative correlation ($r = -0.70$, $n = 20$) for soft wheats, in agreement with work by Bettge et al. (1989 and 2000) on a large sample number of samples of 51 soft wheat flours. The importance of proteins remains very controversial, however. According to Abboud et al. (1985), although the negative correlation observed in a group of 44 wheats of various origins (33 varieties of hard wheat and 11 varieties of soft wheat) was similar to that found in other references ($r=-0.69$), it was negligible for soft wheats as a whole. These findings were confirmed by Nemeth et al. (1994), who found no significant relation between cookie diameter and protein content in a group of 11 soft wheat flours from Canada, Australia and the United States. Within the same cultivar, however, cookie diameter depended strongly on protein content ($r = -0.87$) given a variable fertilization rate (Abboud et al., 1985). According to these researchers, another unidentified genetic factor is probably the main factor responsible for spreading in cookies. Gaines (1991) reported that, in a study of 53 soft wheat cultivars (red and white types) from different locations and harvest years, a correlation could be found within each cultivar between biscuit diameter and protein content. Correlation coefficients were negative for red wheats and slightly positive for white wheats. The author also observed that the two types of soft wheat could be clearly differentiated by their sensitivity to protein content: in red wheats, any increase in protein content was accompanied by greater grain hardness and cookies that spread less while, in white wheats, it had practically no effect. The results suggest that the controversial issue of the effect of protein content on cookie spreading can be explained by the sensitivity of the results to this factor, which is quite variable depending on the wheat cultivar, even within a group of soft wheats. There is a paucity of data on the contribution of the different protein fractions to biscuit quality. Hou et al. (1996) reports that the total glutenin subunits per unit of flour protein correlated negatively with cookie diameter and they also suggested that certain gliadin and glutenin subgroups may be functional during cookie production but their results varied depending on the class of wheat. Note that the freeze dried gluten recovered according to the method of MacRitchie (1985) can be extracted with aqueous ethanol to recover both the glutenin and gliadin in order to study the impact of these protein sub-fractions on bread baking performance (Graßberger et al., 2002). Similar work has never been reported on biscuit baking. If gluten is important to give cookies with appropriate textural characteristics, Donelson (1988) showed that normal appearing sugar-snap cookies could be even produced without the gluten fraction, suggesting that a simplified baking system model could be used to observe, how elevated levels of damaged starch, could influence the cookie spread through manipulation of the water-starch relationships.

4.2.2.1- Enzymes

A number of applications have been found for amylases, proteases and xylanases in cereal-based products such as crackers, biscuits and biscots (Moenen, 1998). Proteases of different origin have been used in biscuit production due to their endoprotease activity, which causes depolymerization of flour proteins; i.e. they hydrolyze the internal sections of the proteins; they hydrolyze the gluten molecules, allowing the biscuit dough to relax after sheeting and cutting with little or no significant shrinkage. Exoprotease activity, on the other hand, results in cleavage of the terminal residues on the polymers, which increases the availability of amino acids and small peptide chains, which promote browning via Maillard reactions and thus improve biscuit colour and flavour. Papaïn, a plant protease, has been reported to be effective in reducing the viscosity and elasticity of doughs and in controlling biscuit dimensions; amylase activity does not exist in papaïn, comparatively to the fungal and microbial proteases which usually have amylolytic activity (Ishida and Nagasaki,1989).

When the influence of several proteases, including papain and bromelain, on biscuit dough was studied, these enzymes were found to modify the rheological behaviour of doughs and the dimensions and texture of biscuits. Xylanases (pentosanases and hemicellulases) can also hydrolyze pentosans and modify the composite starch-gluten network to promote the extensibility of doughs and possibly increase bread volume (Rouau, 1993) and Rouau et al.(1992). Slade et al. (1994c) used an enzyme preparation containing pentosanases and/or beta-glucanase to treat biscuit, snacks and cracker flours in order to reduce the water regain phenomenon in these products during storage and thus increase their shelf life.

The appearance of the biscuit (the surface fractures pattern) and spreading can be improved by adding papaïn. This enzyme, when incorporated in hard wheat flours, produces biscuits of similar size to those made from soft wheat flour (Gaines and Finney, 1989). These studies appear to demonstrate the great potential of enzymes in altering flour functionality and the quality of finished products. In spite of these positive effects, the use of enzymes in commercial biscuit making remains rather limited, due to the fact that, in commercial production, the resting and baking stages are too short (30–35 minutes) to allow optimum enzyme activity. In addition, the low moisture content and high sugar and fat contents in commercial recipes significantly inhibit enzyme activity as suggested by (Belden SA Puratos group)

4.2.3- Starch

Starch represents approximately 63%–72% of thewheat flour, depending on the variety and growing conditions of the wheat (Atwell, 2001a; Pomeranz, 1988). Starch consists of two polymers, amylose and amylopectin, and two types of granules, the A granules and B granules, which differ in size and shape as discussed previously. The B granules are spherical with a diameter less than 10 μm and A granules are larger ($\geq$ 100 μm) and lenticular in shape. During milling, the endosperms of hard and soft wheats are broken up differently, producing flours with different particle size profiles and damaged starch concentrations. There is a relation between the particle size index (PSI) (proportion of particles smaller than 75 :m by weight) and the damaged starch content, which can be altered, or even inversed, under modified milling conditions (Wade, 1988; Nemeth et al., 1994). Any increase in pressure between the rollers results in finer powder containing more damaged starch. The starch granules are firmly bound within the protein matrix, which explains the physical damage to starch granules during milling. Some authors have shown the influence of damaged starch on biscuit diameter, including Abboud et al. (1985b), who found that damaged starch had a negative influence ($r = -0.63$, $n = 44$); this was confirmed by Nemeth et al. (1994) ($r = -0.68$, $n = 11$). In soft wheats, starch granules are not as strongly associated with the protein matrix as they are in hard wheats. This explains the low levels of damaged starch in soft wheat flours compared with hard wheat flours. Starch also plays a role in the structure of doughs and cookies. Wade (1988) describes starch as an inert component whose role is to fill the continuous matrix made up of gluten and pentosans. Its essential role in water absorption during mixing is also well known. Some authors suggest that damaged starch, compared with other flour components, modifies the consistency of doughs, even if other ingredients such as pentosans and gluten also absorb a large quantity of water. Damaged starch is more susceptible to amylase activity, but since no fermentation takes place in biscuit manufacturing, these enzymes play a negligible role. Greenwood (1976), using optical and scanning electron microscopes, demonstrated the organization of starch granules in biscuits. Depending on the type of biscuit, starch granules swell and disorganize to different degrees. In cookie, starch granules ranged from being in a swollen state to being undisrupted state. Lineback and Wongsrikasen (1980) found that only 9% of the starch granules present in a sugar-snap cookie recipe showed birefringence loss and only 4% of the starch was gelatinized. Due to the low moisture content of cookie recipes (less than 20% in the dough), the starch granules seem to conserve their structural integrity (Varriano-Marston et al., 1980; Burt and Fearn, 1983; Wade, 1988). Interactions between starch and gluten play a role in dough rheology (Petrofsky and Hoseney, 1995) and starch from different sources of cereals (soft and hard wheats, potato, rice, rye, oat and corn), when mixed with a constant gluten source, give doughs with different rheological properties. Sollars (1958) employed a fractionation and reconstitution technique to study the spread depressing effects of chlorination. Via a series of interchange studies between fractions from treated and untreated chlorinated flours, the baking data confirmed that both the starch and gluten fractions were affected most by the chlorination process. Chlorination of the starch fraction increased starch hydration and reduced the spread of sugar-snap cookie (Donelson, 1990). These features are interesting since the hydration test is conducted at ambient temperature and very little actual gelatinized starch has been noted in sugar-snap cookies (Abboud and Hoseney, 1985).

Globally, chlorination of flours has a deleterious effect on the sugar-snap cookie baking performance: cookies are smaller and the appearance impaired.

When the wash waters obtained during kneading, to recover gluten, are centrifuged, the starch is separated into two layers: the starch tailings and the prime starch and the supernatant recovered is referred to as the water soluble, fraction.

4.2.3.1-The starch tailings

Are a heterogeneous material consisting of 87%-94% starch, 1%-2% protein, 0.7% lipids, 0.3% ash, 4% pentosans and roughly 3% cellulosic material (MacMasters and Hilbert, 1944). This fraction, which is mucilaginous in texture and low in density, makes up roughly 15% of flour. The starch in this fraction is made up mainly of small granules and fragments swollen in water, large damaged granules, insoluble proteins and pentosans with a high absorption capacity (Miller and Hoseney, 1997a). Yamazaki (1955) observed that this fraction has a prominent effect on the spreading of cookies. The degree of spreading is in direct relation with the quantity of free water (Fuhr, 1962). In fact, the addition of others hydrophilic systems to flour such as cellulosic material (ex. bran) or endosperm cell wall had also a detrimental effect on cookie spread. The pentosan-rich fraction specifically has deleterious effects. Both the water soluble and water insoluble pentosans are hypothesized to influence the baking performance of wheat flour because of their high water binding capacity (Bushuk, 1966). The water soluble pentosans dissolve in water to give viscous solutions, whereas the insoluble pentosans become highly hydrated without truly dissolving (Jelaca and Hlynka, 1971). According to Yamazaki (1956) and Sollars and Bowie (1966), the absence of the low-grade fraction or starch tailing, promotes spreading while an excess of this fraction reduces it considerably. Additional fractionation to purify this material shows that the pentosan-rich subfraction substantially reduces the diameter of cookies, while the damaged starch subfraction has a rather moderate influence on diameter. In addition, the lipids in this fraction play a minor role in affecting cookie diameter, while the enzymes have no effect (Sollars and Bowie, 1968). To understand the effect of starch tailings on cookie spreading, Krishnarau and Hoseney (1994b) hydrolyzed this fraction in order to determine the role of the sugars in the fraction as sweetening agents that could partially replace sucrose incorporated into the cookies recipes. It was assumed that the two polysaccharides of the starch tailing fraction, the starch and insoluble pentosans, would be degraded during an acid hydrolysis, following the method developed by Kim (1990) with 0.4 N sulfuric acid during 0.5, 1, 2 and 5 hours of hydrolysis. The mixture was then neutralized with a 0.2 N barium hydroxide solution, with the barium sulfate precipitate filtered through a frozen and dried Whatman paper. Cookies containing sucrose had pronounced spreading, while those containing xylose had a limited amount of spreading. When sucrose in cookies was completely replaced by hydrolyzed starch tailing fraction, the spread of cookie depended on the molecular size of the polysaccharides in the fraction added, and this spread was controlled by the extent of hydrolysis, so a two hours hydrolysis gave cookies that spread the most. Hoseney and Krishnarau (1994) showed that cookie dough viscosity determines spread of the cookies prepared using the sugar-snap recipe. According to Shanot (1981), Doescher et al., (1987) and Krishnarau and Hoseney (1994b), crystallization of sugar is responsible for the well-defined cracks on the surface of cookies, while recipes containing glucose, fructose and maltose produce cookies with a smooth surface. The replacement of sucrose with hydrolyzed starch tailings results in a significant decrease in cookie spreading. The resulting dough was hard and rubbery and the cookies whitish in colour. In cookies prepared with hydrolyzed starch tailings (duration of hydrolysis of 0.5, 1 or 2 hours), the longer the duration of hydrolysis, the more pronounced the spreading; such cookies also had a darker colour due to the Maillard reactions (greater formation of reducing sugars) and a smoother surface. Longer hydrolysis (five hours of treatment), however, was found to result in diminished spreading since most of the starch and insoluble pentosans are converted into their respective monosaccharide (glucose and xylose and in smaller amounts of arabinose) and small fragments of the polymeric polysaccharides chains.

4.2.3.2- Prime starch

Consists mainly of large, undamaged type A starch granules, forms a dense white layer (Miller and Hoseney, 1997a). According to Sollars and Bowie (1966), cookie diameter depends, above all, on the damaged starch content. The higher the damaged starch content, the smaller the diameter. When prime starch containing 9.4% damaged starch was used in a recipe, a slight increase in cookie diameter was noted; when a mixture containing 92% damaged starch was used, the inverse effect was observed. Kuhn and Grosch (1985) showed that any addition of pentosans from starch (tailings and/or water soluble fraction) resulted in lighter doughs. In fractionation studies, sugar-snap cookie diameter was increased by substituting the isolated prime starch fraction from either gluten or the tailing fraction (Donelson 1988). The substitutions lowered the hydration values of the dough system, suggesting that all components appear critical in explaining the baking performance.

4.2.3.3-**The water-solubles fraction**

Is obtained after centrifugation of the crude starch is made up of soluble proteins (albumin and globulin), amino acids, peptides, starch, pentosans and several materials of low molecular weight. Sollars (1959) demonstrated that incorporating this fraction into a gluten-starch mixture increased bread volume; this was attributed mainly to the presence of albumin. He fractionated flour with 95% ethanol (1 part flour: 3 parts alcohol). Following this step and centrifugation, he obtained a white precipitate corresponding to the alcohol-soluble and alcohol-insoluble fractions. The components of the alcohol-insoluble fraction (21-31% of the original soluble fraction) were basically high-molecular-weight polysaccharides like pentosans and proteins. They had a marked effect on the reduction of cookie diameter. However, the alcohol-soluble fraction (68-78% of the original material) contained basically low-molecular-weight components such as simple sugars, amino acids, peptides and oligopolysaccharides. In the latter case, no reduction in cookie diameter was noted when this fraction was incorporated in the flour. According to Yamazaki (1955) and Sollars (1959), purified starch tailings have a pronounced negative effect on spreading, mainly because of their absorption power. The soluble fraction, however, had less effect on cookie diameter although this fraction could exercise a large effect per gram of material. Several schemes have been proposed to separate or even concentrate the components in this fraction to be able to better understand the role of pentosans (Pomeranz, 1980). The first step consists in carrying out dialysis on the small molecules and ions to reduce the ionic strength and precipitate the albumin fraction. The second step consists in separating the albumin fraction by centrifugation. The supernatant obtained was very rich in water-soluble components, pentosans and glycoproteins. Baldo and Wrigley (1978) used another protocol which first involved extracting the albumin and globulin fraction using 10% sodium chloride and secondly carrying out dialysis on the water soluble and precipitating and centrifuging the globulins; the supernatant containing the albumin was then freeze-dried, while the residue was dissolved in 10% sodium chloride, centrifuged and dialyzed to produce the globulin fraction. This method avoids the denaturation step used to prepare the albumin fraction. A study by Chen and Hoseney (1995) showed that dough stickiness is mainly associated with the soluble fraction obtained by dialysis of the dough. An analysis of this fraction by HPLC suggests that it contains mainly feruloyl residues attached to a carbohydrate component.

The water solubles fraction is known to affect the rheological properties of the dough. Removal of the water soluble fraction from flour increases the mixing time (Mattern and Sandsted, 1957). Miller and Hoseney (1999) reported that flour with shorter mixing time had more water soluble material than the flours with longer mixing time. Hoseney et al. (1994) also found that omission of the water soluble fraction from reconstituted flour resulted in bread with reduced volume.

4.2.4- Nonstarchy polysaccharides

The main non-starchy polysaccharides in soft wheat flour are found basically in the water soluble fraction and in the starch tailings collected in the aqueous phase after the dough have been washed to extract the gluten. Pentosans originate from the aleurone cell walls and endosperm, making up ~1.5% to 2.5% of the flour (Abboud et al., 1985); the pentosan content is basically determined by genetic and environmental factors (Hong et al., 1989; Kaldy et al., 1991). The pentosans are subdivided into solubles or insolubles pentosans or arabinoxylans (stricto-sensu pentosans) and arabinogalactans depending if the basic structure is constituted of D-xylose or D-galactose. The two main polymeric components of pentosans are arabinoxylans and arabinogalactan-peptides. The arabinoxylans, are made up of linear chains of xyloses (D-xylopyranosyl) connected with ß-bonds (1, 4); the arabinose (arabinofuranosyl) molecules are linked onto the 2 and 3 carbons of the xylose residues (Figure 1.13). The arabinose-xylose ratio is between 0.5 and 1 (Cole, 1967).

Arabinoxylans can also contain small amounts of ferulic acid (roughly 2 mg/g), depending on the wheat variety; the feruloyl groups are covalently linked to the arabinose units. Through dimerization of the ferulic acid, two chains of arabinoxylans can cross-link to form a polymeric conjugate with a higher molecular weight. About 25% to 50% of arabinoxylans are water-soluble and can be differentiated from the insoluble arabinoxylans by the degree of substitution (0.6-0.7 instead of 0.7-1.1) and lower MW (20,000-60,000 instead of 100,000-150,000). The wheat arabinogalactan-peptides consist of large polysacharrides moieties (approximately 93%) covalently linked with 15 amino acids peptide and are known as an excellent stabilizer, similar to gum arabic

The insoluble pentosans cannot be extracted by water, since they are polymerized via cross- linking with ferulic acid, whereas, the soluble pentosans contain free ferulic acid. Moreover, the soluble pentosans, once they are cross-linked, they become insoluble, and as a result, water absorption capacity increase tremendously. Meuser and Suckow (1986) as well as Rouau and Surget (1994) showed that insoluble pentosans have high water sorption capacity, while the soluble pentosans can form a gel and the gel viscosity depend on the chain length. Pentosans play an important role in the rheological properties of doughs, particularly affecting the dough development time during mixing, the consistency and the extensibility. This may result from immobilizing some of the water necessary for complete hydration of gluten proteins and/or due to interference of the pentosans interaction between the gluten proteins. (Roels et al., 1993). By increasing dough viscosity, pentosans may help to stabilize gas bubbles during baking (Izydorcyk and Biliaderis, 1995).

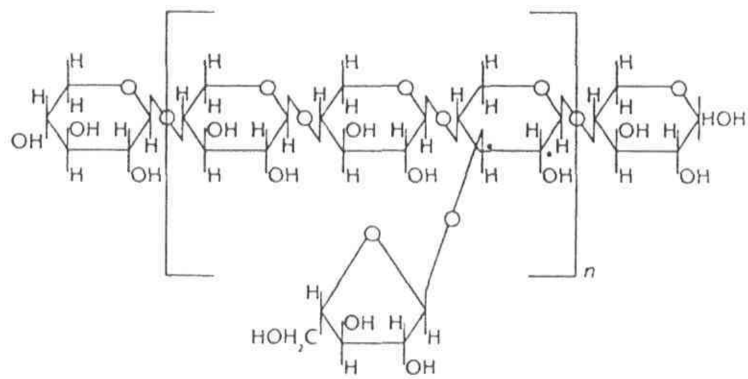

Figure 1.13: Arabinoxylan structure (D'Appolonia, 1985)

However, their role in breadmaking remains controversial; the soluble pentosan content is thought to have a slightly negative impact on bread volume in British breadmaking; however, instead, in French bread-making, a significant positive correlation was reported by Rouau and Saulnier (1994) between bread volume and soluble pentosan content (r = 0.59) with 12 commercial flours. This factor is perhaps the one that best explains product volume variations, one that is even better than protein content; this may be due to the fact that the protein content shows little variation among wheat cultivars. In biscuit manufacturing, the role of pentosans remains fairly difficult to define. It is recognized, however, that adding tail-end fractions, which are rich in insoluble pentosans, reduces cookie diameter (Upton and Hester, 1966; Abboud et al., 1985). Soluble pentosans probably increase the diameter of cookies made of soft wheat flours and decrease them in the case of hard wheat flours (Yamazaki et al., 1977). More recent studies on the cookie-making potential of 20 varieties of wheat showed a negative correlation between soluble pentosan content and cookie diameter (Kaldy et al., 1991). It should also be mentioned that the complete absence of pentosans in flour is undesirable, resulting in very large cookies with an unacceptable surface appearance; when pentosans were isolated, concentrated and then added to the dough, the diameter decreased (Yamazaki, 1955). Pentosans, therefore appear to act as a factor affecting cookie size (Sollars and Bowie, 1966). According to Cottenet (1986), pentosans are of great importance in the expression of biscuit quality and two mechanisms can be advanced to explain their role:

-the first is physical in nature: in general, insoluble pentosans, due to their high water absorption capacity (more than 10–11 times their weight in water), have a negative effect on biscuit spreading (Sollars, 1959). Yamazaki et al. (1977) obtained AWRC (alkaline water retention capacity) values of 165.9% for starch tailings from Shawnee starch wheat, a hard wheat variety, and 193.4% and 266.1% respectively for tailings from Thorne and Blackhawk soft wheats. The hydrophilic characteristics of pentosans, which immobilize free water in the dough system, are a detrimental factor in cookie quality. Bushuk (1966) showed that pentosans, which only represent ~2% of flour, are responsible for 23% of the absorbed water; with the starch (~70% of flour) and gluten (10% of flour) being responsible for 46% and 31%, respectively, of the water absorbed; this corresponds to ratios of 11.5, 0.7, and 3.1 for pentosans, starch and gluten, respectively. The technological value of flour thus seems to be determined largely by the pentosan's water absorption capacity (D'Appolina and Shelton, 1984; Cottenet, 1986 and Jeltema et al., 1983). A study by Abboud et al. (1985) found that, even though the movement of water was an important element in the cookies baking mechanism, there was no significant correlation between pentosan content and flours' water absorption capacity or between total pentosans and cookie diameter. From a practical point of view, variations in pentosan content from one flour to another do not seem to explain cookie quality. It is mainly when pentosan-rich subfractions are added that a significant effect is observed. However, more recently, a study by Bettge and Morris (2000) showed that total pentosans, quantified in 13 samples of soft wheat flour, were responsible for 87% of the variations in cookie diameter.

-The second mechanism is qualitative in nature and is based on the potential of pentosans to become associated with the gluten matrix; starch granules will then insert themselves into the gluten-starch matrix through the pentosans, which could explain the hardness of the grains (Bettge and Morris, 2000). According to this view, pentosans could contribute to the differences in behaviour between soft and hard wheats in cookie making. Some pentosans associated with gluten are water dispersible, forming viscous solutions in the presence of oxidizing agents. Soluble pentosans form solid (elastic) gels that are insoluble in water. Pentosans in their isolated state may also form covalently linked networks gels in the presence of oxidizing agents; in this case, the ferulic acid that is bound to the arabinoxylans is responsible for this gelation (Izydorczyk and Biliaderis, 1995). High concentrations of ferulic acid, a xylan chain with a low degree of substitution and a high molecular weight are all conditions favouring cross-linking and the formation of a three-dimensional network structure (Izydorczyk and Biliaderis, 1992). Therefore, the pentosan content is not the only factor differentiating flours and it is possible that other compensation effects may mask the functionality of these polysaccharides. This may also mean that further to the concentration of pentosans, their structure (i.e., molecular size and degree of branching) is also important determinant of their functionality in the dough of the final product quality.

It should also be mentioned that others non polysaccharides such as β-glucans, cellulose are also located in the walls of cells of the endosperm and bran tissues.

5- Rheological properties of biscuit doughs

5.1- Microscopic structure of flour dough

Pre-existing gluten proteins are found in the endosperm cells in the form of small fragments that acquire viscoelastic properties upon hydration (Amend and Belitz, 1991). Figure 1.14a shows the hydration of a flour particle and the appearance of protein fibrils surrounding the starch granules (Bernardin and Kasarda, 1973; Amend and Belitz, 1991). These granules adhere to the surface of the fibrils, which are oriented in all directions. When the hydrated particle is flattened between a slide and coverslip, several fibres oriented in the direction of applied force can be observed through the optical microscope, and the fibrils display viscoelastic behaviour).

At the beginning of the mixing process, the protein chains in the hydrated flour particles adhere to each other when the individual particles come into contact. At this stage, the proteins are not yet stretched out and the gluten structure in the dough resembles that in the flour particles (Figure 1.14b). Subsequently, the flour particles begin to agglomerate and through the mechanical action, force is applied to the protein molecules, stretching, unfolding and orienting them in a particular direction. At this stage of mixing, a protein film forms (Figure 1.14c), and as the mixing continues, the dough becomes increasingly homogenous, with many stretched-out protein fibrils transformed into films; at this point, a three-dimensional network becomes visible (Figure 1.14d) and the dough attains its optimum consistency. The final network consists of two phases: starch as the solid dispersed phase and an aqueous phase in which the water dilutes the soluble components and ensures the dispersion of the lipid droplets. When the dough is over-mixed, however, the network is torn, causing local breaks in the protein films. According to Bernardin and Kasarda (1973), the starch likely acts as filler in the dough and the interaction between fibrils and starch granules indicates that granule surface characteristics affect the dispersion of the granules in the matrix formed by the fibrils.

When this interaction is complete, almost the entire granule is wrapped in protein fibrils; when it is only partial, the fibrils only cover part of the granule. Under the optical microscope, bread doughs have a gluten network that forms a continuous structure. On the other hand, in biscuit doughs, although hydrated proteins are dispersed homogeneously, a network is not formed, leading to a discontinuous structure. It is suggested that this structure can be explained by the fact that, due to the low water content and high sugar and fat contents of biscuit doughs, the protein macromolecules cannot aggregate together and form a continuous network.

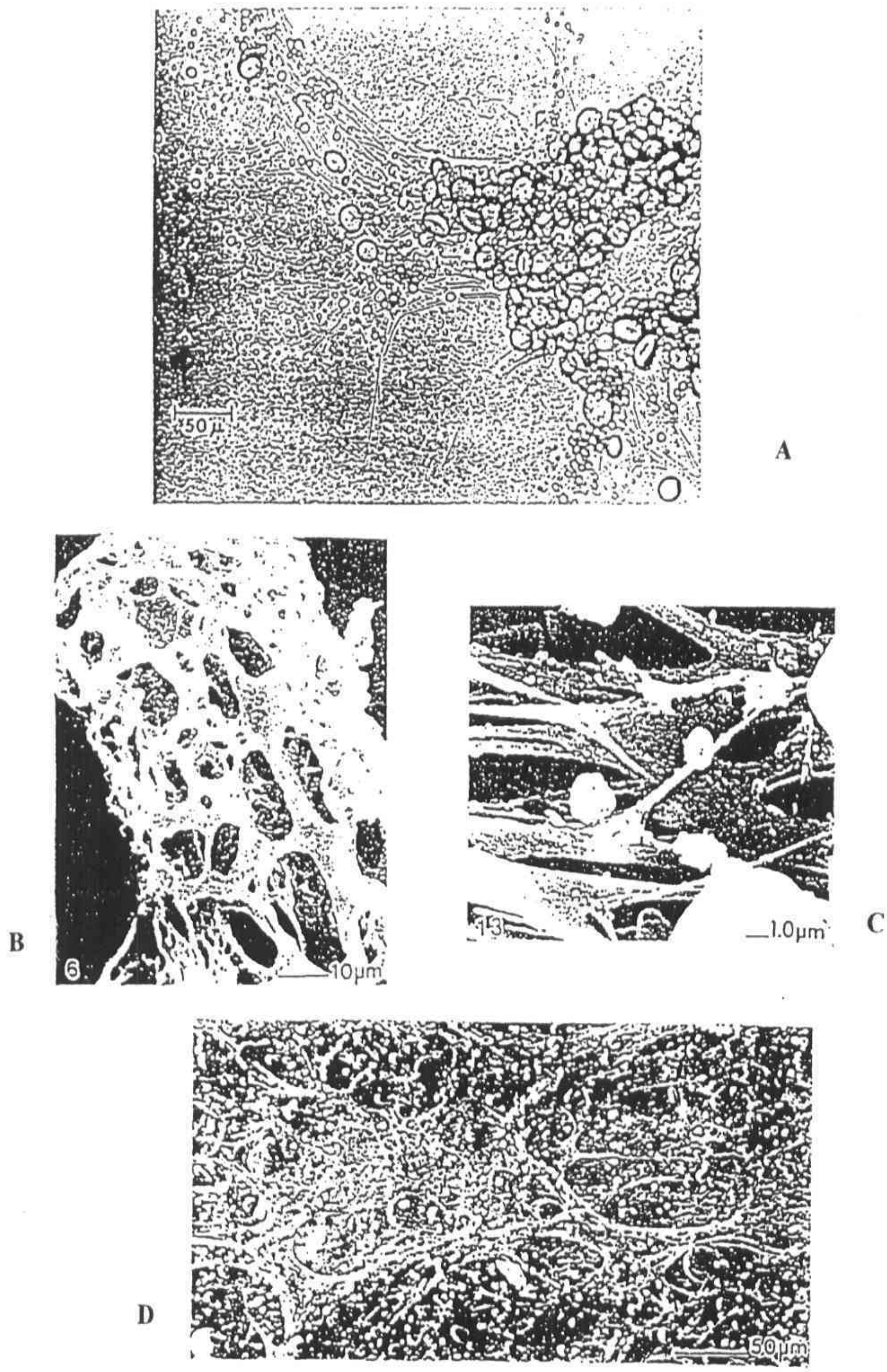

Figure 1.14: Microscopic view of moistened flour particles (Amend and Belitz, 1991)

5.2- Protein network development

Flour can be considered as a reactive biological material. Its reactivity is due to the capacity of gluten proteins, but also pentosans, lipids and starch, to form new bonds and break other ones in the presence of water during mixing. As discussed previously, the protein fibrils form when water is added to flour. During subsequent mixing, several phenomena occur causing a physicochemical transformation of the system (Mecham, 1968; Dubois, 1995): the rearrangement of the spatial configuration of the proteins, formation of noncovalent bonds between the proteins and other components such as pentosans, the breakage and reformation of disulfide bonds and the reappearance of the protein network. During mixing, the gluten proteins move in the same direction, line up and partially unfold, thus promoting hydrophobic interactions and the formation of disulfide bonds through disulfide-sulfhydryl exchange. The three-dimensional network becomes established as the gluten particles are transformed into thin membranes (films) surrounding the starch granules and other components in the flour.

Mixing of the dough results in an increase in protein solubility. The protein fibrils, with a diameter of 50–100 Å, join to form 10,000 Å protein fibres, a true network that gives the dough its elasticity; the glutenin aggregates break up into fractions of lower steric hindrance (Bache and Donald, 1998). The quantity of glutenin macropolymers also decreases during mixing and increases when the dough is resting. In addition, dough development causes a reduction (through oxidation) in the quantity of SH groups from 0.58 to 0.32 micromoles per gram of flour, some of which may play a part in lipid-protein interactions. In contrast, the amount of S-S bonds does not seem to vary (11.1 versus 11.2 micromoles per gram of flour) (Hoseney et al., 1997).

The mixing of dough allows ingredients to be thoroughly combined, forming a homogenous mixture on the macroscopic scale, which ensures dough development through the swelling and organization of proteins, giving the dough unique gas retention properties, and lastly to ensuring the incorporation of air, which is integrated into the dough in the form of gas cell nuclei. The mechanical action and accompanying mechanical energy input allow numerous interactions among the dough components to occur.

The distribution of water at the different adsorption sites (polar groups of glutens, starch and pentosans) depends on the mechanical work provided during mixing. This results in changes in the consistency and viscoelastic behaviour of the dough. Modifications to the rheological properties of doughs during mixing have been found to be caused by the cleavage of S-S bonds, under the mechanical influence of the dough mixer, rather than by oxidation-reduction reactions (Mecham, 1968). In effect, this author observed the same changes when mixing was carried out in a nitrogen atmosphere, with an increase in the sulfhydryl (-SH) radical content.

During mixing, the protein film appears, uniformly distributes around the starch granules. If over-mixing occurs, covalent and other non-covalent interactions may be modified, causing major changes to the network structure: the dough may stick, due to the breakdown of the continuous membranous structure of the protein network, which results in the appearance of individual fibrils and pieces of the network in the dough. Dough development is a reversible process. Dough can be mixed, left to relax, and mixed again since weak bonds (hydrogen and/or hydrophobic) rather than covalent bonds are involved. While the dough is resting, many different molecular modifications occur, attributed essentially to the hydrogen bonds which alter its rheological behaviour (Belton, 1999).

5.3- Rheological properties of doughs

Variations of around 10-12% have been observed in the dough length,along the sheeting direction with biscuits prepared from a single batch in plant production (Jurgens et al., 2000). A number of factors seem to alter the rheology of doughs during mixing, resting and sheeting. They include the relaxation of the stresses induced during mixing, the continued hydration of flour components and the redistribution of water (Hibberd, 1970). Another possibility is that the thiol-disulfide interchanges continue to occur during the dough resting phase, resulting in a change in the molecular weight of proteins and the elastic modulus (G') (Dong, 1992).

These factors depend basically on the structure of gluten and affect the dough rheological characteristics and cookie quality (Faridi and Faubion, 1986; Menjivar and Haridi, 1994). In "short dough" cookie doughs, minimum gluten development occurs, serving only to ensure a certain degree of cohesion in the dough so that it can be handled and set. Inversely, in semi-sweet "hard doughs," such as the French petit beurre (butter biscuit) type (35% sugar, 17% fat and 20% water, on flour basis), the gluten network is partially developed, so that the dough is extensible enough to be sheeted, but not so developed that the dough is too elastic and shrinks during sheeting (Levine and Drew, 1994). During the manufacturing process, the dough is sheeted in several reduction steps, which causes the gluten network to be aligned in the direction of machining. The sheeting applies significant stresses to the dough, and the elastic components cause a gradual contraction of the dough sheet. This variability in the elastic recovery and thereby in the weight and dimensions of the biscuits causes considerable issues in fully automated packaging operations. The rheological characteristics of doughs depend on the quantity of ingredients in the recipe, particularly flour, and also process conditions such as the mixing method, resting time and molding and sheeting conditions (Bloksma and Bushuk, 1988). As discussed in Section 2, the different types of biscuit recipes produce doughs with different rheological characteristics. Compared with breadmaking, little work has been done on the rheology of biscuit dough (Manohar et Rao, 1999a, b and c). Some doughs are so dry that there is not enough water for the gluten in the flour to become hydrated and the starch cannot become gelatinized during baking.

As discussed previously, the gluten complex is believed to be a protein network held together by extensive covalent and non-covalent bonding. The elastic and cohesive character of gluten is due to a great extent to the presence of disulphide bonds which are exclusively intramolecular in gliadin, while in glutenin they are both intra and intermolecular. Wheat flour doughs are considered to be viscoelastic liquids, with their elastic properties basically associated with the network of gluten without covalent bonds, but also other components such as starch granules and cell wall polysaccharides which compete for the free water (MacRitchie, 1976). The rheological properties of doughs depend on this equilibrium between elastic and viscous properties which manifest themselves throughout production (mixing, resting time, sheeting and start of baking). Additionally, the glutenins provide the elastic modulus, whereas, the gliadins are responsible for the viscous modulus. During baking, numerous chemical and physical interactions occur at the molecular level and cause changes to the dough's viscoelastic properties, eventually influencing the structure of the product. These temperature-induced interactions can be attributed to the partial gelatinization of starch, cross-linking of proteins and the redistribution of water between starch and the protein fractions (Eliasson, and Larsson, 1993). During baking, cookie diameter increases in a linear fashion and then suddenly becomes fixed (Abboud et al., 1985a). The final diameter of the cookies therefore depends on the speed at which the dough spreads during baking. Spreading is caused by the expansion of the dough under the action of leavening agents and gravity flow. In sugar-snap recipes, flours that produce cookies with a good spread (i.e., ~ 50% increase in size) are considered to be of high quality (Miller and Hoseney, 1997a). The spread rate appears to be linked to dough viscosity, which in turn depends on the proportion of ingredients in the recipe, particularly water, which acts as a solvent-plasticizer, and the melting of shortening, which decreases viscosity. It should also be noted that water-gluten dough has a lower elastic modulus than flour-water dough, which suggests that starch is definitely not an inert ingredient (filler) that does not interact with the other ingredients in flour-water dough.

As discussed previously, the dough made with hard wheat flour demonstrated a controlled elastic expansion which continues until a maximum and the dough shrinks in diameter through a controlled elastic shrinkage (Slade and Levine, 1994). On the other hand, dough made with soft wheat flour, spread slowly to a maximum diameter and then collapse. Behaviour of poor quality flour resembled to the elastic recovery in a rubbery thermoset polymer system, whereas the behaviour of good quality flour is characteristic of structural collapse in a rubbery predominantly thermoplastic polymer system.

5.4- Rheological methods for biscuit doughs

The rheological properties of biscuit doughs are important as they influence the machinability of the dough as well as the quality of the finished products. An extensive literature is available on factors influencing the rheology of bread dough, and their effect on bread quality but there is limited information available on biscuit doughs.

Determination of the rheological properties of a dough consists at measuring both the strain or speed of deformation as a function of the applied stress. The deformation is translated by either a variation in length or angle (distorsion). During the various stages of process of a cereal based product, i.e. mixing, resting, laminating, sheeting, baking and cooling, different deformation speed and stresses are applied to the biscuit doughs (Dobraszczyk, 1997). It is therefore crucial to select the most appropriate rheological test to mimic the dough behaviour in a plant operation.

Both biscuit and bread doughs exhibit viscoelastic behaviour combining the properties of both purely viscous fluid and purely elastic solids. For example, due to its viscous component, a freshly mixed dough will flow under the force of gravity. The same dough when rapidly stretched and then released will spring back (elastic recover); that elastic component helps to determine the dough's resistance to deformation.

A number of factors influence dough rheology after ingredient mixing and dough development. These include relaxation of the stresses induced during mixing, continuing hydration of flour components, and the redistribution of water (Hibberd, 1970).

While most of the differences in dough are usually related to gluten proteins, the starch can also contribute to dough rheology in bread making (Medcalf and Gilles,1968) especially due to the starch gelatinization. Dong (1992) demonstrated that dough tested immediately after mixing had a higher G' and smaller loss tangent than the same dough that was allowed to rest in a bowl for 15 min before testing. Thus, dough does not relax rapidly after being placed between the parallel plates of the rheometer (Dresse et al.,1998).

The rheological characterization of the dough provides a better understanding of the physical phenomena that occur during industrial production and also give information complementing other physicochemical analyses, particularly involving flour components functionality. A number of authors have characterized the rheological behaviour of biscuit doughs, using either empirical or imitative methods or basic rheological methods. All these methods must be adapted to the high degree of consistency found in cookie doughs.

Miller (1985) developed a penetrometry test on low-moisture-content doughs (10%–14% water on flour basis). In this test, dough with a constant weight is placed in a cylindrical container and the surface is flattened with compression. Several needles penetrate the dough simultaneously at constant speed until they reach a predetermined depth. The force measured by the texture analyzer indicates the consistency index. According to Miller, the purpose of the test is to replace a plant operator's subjective evaluation in adjusting dough hydration. This consistency or resistance to compression appears to be strongly correlated with biscuit thickness and weight. A relation between the logarithm of consistency (targeted) and hydration has been established, giving a unique scatter diagram for flours. The method has proven to be difficult to replicate because of differences between operators. Gaines and Finney (1989) measured the dough consistency and correlated it with the cookie diameter. Gaines (1990) reported also the influence of mixing and additives on dough consistency and quality of sugar snap cookies.

Another approach, which is well suited for firm doughs, is an extrusion test, which was developed as part of a biscuit test by the Centre Technique d' Utilisation des Céréales (CTUC) (Tharrault, 1994). In this test, a quantity of dough is extruded using a piston which descends at a constant speed: the time taken for the extruded dough piece (log) to reach a certain length is measured to calculate the linear extrusion speed (V). This speed is inversely proportional to the square of the radial expansion, which is the ratio of the log and die diameters; the lower the value of V, the greater the radial expansion, an expression of the elastic effect. In addition, the energy required for extrusion (E), which corresponds to the energy (area under the curve) required to extrude a given volume of dough as a function of time, can be measured and expresses the viscous effect. The V/E ratio is a good measure of dough machinability. The test is particularly useful in adjusting production parameters in the factory, but did not appear useful for characterizing the potential of flour in the laboratory for flour functionality studies

The importance of mixing in determining dimensional characteristics in the manufacturing of dry biscuits has been reported by Contamine et al. (1993 and 1995). These authors found dimensional instability above the energy threshold of 60 kJ /kg for their mixer.

Jurgens et al. (2000) studied the effect of resting time, temperature and mechanical history on the elastic and viscous moduli of hard doughs used in the manufacture of dry biscuits and on gluten microstructure under microscopic examination. Dough temperature was found to be a critical parameter in biscuit making. In addition, these authors observed that there was a critical period during which the elastic properties of doughs changed rapidly; therefore effective control of all these parameters is crucial in obtaining a high-quality product.

The rheological properties of dough are strongly linked to the composition and concentration of the ingredients such as fat and sugar incorporated into a recipe, the method of mixing, and the temperature of the ingredients (Faridi and Faubion, 1986). These ingredients influence the gelatinization temperature and post-baking biscuit quality. This explains the very different rheological behaviours found in numerous commercial recipes since gluten development occurs only partially, along with a low rate of starch gelatinization. Due to low water content, biscuit doughs have low extensibility properties, but are more elastic than bread dough. The biscuit doughs are sheeted and laminated in several reduction steps, which causes the gluten network to be aligned in the same direction with machining (Levine and Drew, 1994). The sheeting process applies considerable stresses to the dough, and the elastic component in the dough cause a gradual contraction of the biscuit dough. This variability in the elastic recovery alters significantly the dimensions of the biscuits and thus is considered as a major technical issue in a fully automated plant production line (Maache Rezzoug et al., 1998).

For the production of short doughs, the creaming method (mixing of sugar with shortening) is often used to minimize gluten development. However, a weak development of gluten is necessary to allow sufficient cohesion for handling and shaping of the dough (Olewnik and Kulp, 1984). The cohesion in the dough that is observed on pressing it into the mould is reported to be related to the presence of plastic shortening.

Most studies on the impact of flour on the rheological properties of doughs have been carried out using empirical or imitative tests on flour-water-salt doughs, basically using the Chopin Alveograph and Farinograph, as well as the extensograph (Bloksma and Bushuk, 1988). These methods are mostly used to characterize the rheological properties of bread doughs which have a strong gluten structure and a high resistance to deformation. With biscuit dough, due to the low protein content of flour, low water absorption properties and also low resistance to deformation, the dough characteristic requirements are quite different. Nevertheless, some of the empirical tests were used to determine quantitative relations with biscuit dimensions.

In the manufacture of dry biscuits, Branlard et al. (1985) observed that the W Alveograph value, associated with the strength of the flour, did not provide satisfactory results. On the other hand, parameters related to mixing appear to be very useful; in particular, there is a negative correlation ($r = -0.74$) between biscuit-making quality and the difference in consistency between four and six minutes as measured by the Farinograph test. In the case of cookies, several studies have shown that the strength of the flour, expressed by the Alveograph W value (also known as the deformation energy [10^{-4} $J.g^{-1}$ of dough]), is negatively correlated with cookie diameter ($r = -0.51$, $n = 58$, $p < 0.0001$, Bettge et al,. 1989; $r = -0.45$, Labuschagne et al., 1996). The tenacity, as expressed by the P value (maximum pressure), also appears to be a useful indicator; the higher the value, the less the cookie spreads (Nemeth et al., 1994; Labuschagne et al., 1996). In contrast, Rasper et al. (1986) did not find the Alveograph results to have any predictive value, using either the standard protocol or constant consistency. Bettge et al. (1989) observed that, although the P value does not have a predictive value in itself ($r = -0.25$, non significant), it is an effective complement to the protein content, increasing the coefficient of determination for biscuit diameter (in 58 soft wheat flours) from 30% to 63%. To validate the results of this multiple linear regression, these authors tested this combination of parameters on a second set of flours ($n = 6$) and obtained a high coefficient of determination (87%) and a slope not significantly different from that found for the calibration samples. The lower the water hydration property as estimated by the Brabender Farinograph ($r = -0.52$, $p < 0.01$, Labuschagne et al., 1996) or the shorter the development time ($r = -0.53$, $p < 0.01$, Labuschagne et al., 1996; $r = -0.72$, $p < 0.05$, Nemeth et al., 1994), the better the spread of the cookies.

Since rheological phenomena play an important role in the processing of flour dough, the study of dough behaviour in terms of elongational (and particularly biaxial) deformation seems to be gaining in popularity in the area of starch based products these last ten years. During mechanical sheeting and molding, dough pieces undergo elongational deformation and the biscuit dimensions, particularly length and width as well as thickness, are strongly linked to the viscoelastic behaviour of doughs as they leave the sheeting unit. Dimension stability increases with the limitation of elastic recovery after setting. One predictive criterion is provided by the kinetics of the stress relaxation after lubricated compression of the dough disks using a compression-relaxation test on a dough piece of a given size (Renard and Théry, 1998; Launay and Bartolucci, 1997). Relaxation following lubricated squeezing flow is often referred as an indicator of biscuit dimensions, which are in turn related to the viscoelastic properties of the dough (Bartolucci and Launay, 2000). The analysis of the relaxation curves using the Maxwell model (presented in the appendix section) allows the determination of several parameters: Tla, k and n. Tla referred to the time necessary to reduce the final compression force by half, once the compression is removed due to the elastic recovery of the dough. The k, the relaxation rate constant, on the other hand, reflects the internal constraints accumulated in the dough and n, is the flow behavior index which vary from 0 (ideal elastic solid with no relaxation) and 1 (viscoelastic liquid with total relaxation). Following sheeting, biscuit dimensions will decrease only in the direction of the sheeting direction and its width will increase as a result of strain recovery. Thus, the more the internal constraints accumulated in the dough are relaxing fast (high k), the less the recovery is important, and the biscuits lengths will be higher with larger width. This can be explained in terms of elastic recovery; dough compressed during sheeting stores mechanical energy, inducing partial strain recovery. A high value of k is equivalent to a fast relaxation process, and thus a low level of stored energy and then to a weak strain recovery phenomenon. The relaxation rate constant after compression explains over 70% of variations in biscuits length .

Miller and Hoseney (1997b) also reported that there is a significant correlation between apparent biaxial extensional viscosity using the lubricated squeezing flow method and the diameter of cookies manufactured with various soft wheat cultivars (r = -0.796).

Compliance and elastic recovery of the biscuit doughs were also measured using a penetrometer. In this case, the penetrometer had a circular metallic plate of 4.5 cm in diameter and 0.15cm of thickness. The two bytes texture profile analysis of Bourne (1978) is also often used to measure the dough hardness, cohesiveness, adhesiveness (Manohar and Rao, 1999 a,b,c).

Harmonic regime studies on biscuit doughs have been summarized in excellent literature reviews by Menjivar and Faridi (1994). Comparing cookies and crackers doughs, approximately the same G' was found with the two types of doughs, which confirm that there is no influence of the gluten network formed in the cracker dough. However, it is often mentioned that the type and amount of fat incorporated into the dough has a strong effect on the viscoelastic properties, and the reduction in the fat content changes the system from bicontinuous to a dispersed system (Baltsavias et al., 1999). Oliver et al. (1995) found that dynamic rheological measurements gave a better prediction of the tendency of the dough to contract than the protein content. The harmonic regime approach will not be discussed in detail in this review, since the biaxial extension using large deformations rather than small shear deformations (the former being the conditions closest to those found in the biscuit manufacturing industry) were used in the present work. In addition, our preliminary tests using a harmonic regime showed that the large number of variables to be studied with repetitions do not allow the use of the oscillatory test, unlike the double compression and uniaxial compression tests which could be carried out quickly on numerous dough pieces of predetermined size.

Hypothèse, but et objectifs de travail

Hypothèse :

L'étude approfondie des connaissances actuelles sur les courants de mouture (fractions de mouture référées comme patent, de coupure et basse) et de leurs constituants nous permet de conclure que i) l'influence de ces courants de mouture incorporés en différentes concentrations dans une recette biscuit secs, de type «petits beurre» et cookies de type à la rotative et coupe fil sur les caractéristiques rhéologiques des pâtes et des produits finis n'est pas bien définie et ii) l'effet qualitatif et quantitatif des constituants (amidon, protéines, pentosanes et lipides) isolés de ces trois courants de mouture par une procédure de fractionnement/reconstitution comme moyen de manipuler les caractéristiques des pâtes et caractéristiques des produits finis n'est pas connu et que iii) les critères physico-chimiques et rhéologiques permettant de prédire la valeur technologique d'une farine pour la biscuiterie, sont essentiellement réalisés sur le diamètre des cookies sugar-snap et non sur des biscuits secs, semi-sucrés

Suite à la revue littéraire, l'hypothèse suivante a été soulevée :

L'étude des relations structure et fonction permettra de déterminer par quel mécanisme les variations de la quantité de macro- polymères endogènes à la farine (protéines, amidon et pentosanes) endogènes à des «farines modèles» expliqueront celles des propriétés rhéologiques des pâtes et caractéristiques dimensionnelles et mécaniques des biscuits secs. Les critères biochimiques et rhéologiques permettant de prédire la valeur technologique d'une farine de blé tendre pour la biscuiterie pourront ainsi être identifiés.

But

Le but de ce projet de recherche était de comprendre l'impact des courants de mouture incorporés individuellement ou en combinaisons dans des « farines modèles» sur les propriétés rhéologiques des pâtes biscuitières et caractéristiques dimensionnelles et texturales des produits finis. Plus spécifiquement, cette étude vise à isoler les fractions gluten, amidon et hydrosolubles de chaque courant de mouture, quantifier leur teneur en protéines, lipides, pentosanes et induire des variations biochimiques majeures dans les farines modèles afin de mieux définir le rôle de ces constituants sur les caractéristiques des pâtes et produits finis. Les nouvelles connaissances acquises permettront d'identifier des critères analytiques et rhéologiques qui permettront de prédire la valeur technologique d'une farine de blé tendre et ainsi améliorer sa fonctionnalité biscuitière par fortification avec des courants de moutures ou avec des fractions isolées à partir de ces courants de mouture pour de pallier aux problèmes rencontrés durant une production usine.

Pour atteindre cet objectif principal et vérifier l'hypothèse de recherche, les objectifs spécifiques suivants ont été fixés :

Objectifs spécifiques

1. Déterminer l'influence de trois courants de mouture (farine patente, de coupure et basse) sur les caractéristiques rhéologiques des pâtes et des cookies produits à partir de trois recettes typiques des procédés coupe-fil, à la rotative et laminé dont les proportions farine : sucre : gras : eau varient largement. Ces résultats, présentés dans le chapitre 2, permettront d'expliquer l'impact de la variation des proportions de ces trois courants, ajoutés individuellement ou en combinaison sur les paramètres physico-chimiques des farines (protéines, cendres, granulométrie, données alvégraphiques, AWRC), la consistance des pâtes, les dimensions (volume, surface, étalement)et propriétés texturales des produits finis (densité et dureté). Ces résultats serviront ainsi à mieux appréhender «l'effet farine» comme matière première sur les modifications des critères physico-chimiques et les attributs rhéologiques des pâtes et paramètres de qualité des cookies. Cette étude permettra aussi de déterminer laquelle des trois recettes aura le plus grand effet sur les caractéristiques des pâtes,

puisque ces propriétés sont plus sensibles aux conséquences de la formation d'une matrice gluténique viscoélastique.

2. Déterminer l'impact d'une procédure de fractionnement/reconstitution pour isoler le gluten, l'amidon et la faction hydrosoluble des trois courants de mouture trois farines sur les caractéristiques des pâtes et des biscuits qui en résultent. (Chapitre 3) Des études similaires ont déjà été réalisées sur l'étalement des cookies sugar-snap Cette étude, réalisée sur des biscuits secs, nous permettra de déterminer si le comportement rhéologique et caractéristiques des biscuits formulés à partir des farines reconstituées seront similaires aux farines originales correspondantes.

3. Induire des variations dans la concentration des fractions amidon, gluten et fractions solubles isolés des trois courants de moutures afin de déterminer leur impact sur les attributs rhéologiques des pâtes et paramètres des biscuits secs. L'utilisation de « farines modèles» permettra de faire varier significativement les teneurs des farines en protéines, pentosanes et lipides afin d'explorer l'impact des constituants de la farine sur ces attributs (chapitre 4). Cette étude vise à identifier les paramètres biochimiques qui vont altérer la fonctionnalité des farines reconstituées pour mieux appréhender le rôle encore très contradictoire des protéines et pentosanes solubles et insolubles et lipides sur les pâtes et biscuits.

4. Déterminer l'impact de la substitution partielle d'une farine commerciale problématique par des courants de mouture ainsi que la fortification de cette farine de référence par des fractions gluten, amidon principal et de refus et fraction soluble sur le comportement rhéologique des pâtes et caractéristiques des biscuits. Cette étude permettra ainsi de confirmer les effets quantitatifs et qualitatifs des protéines, pentosanes et lipides pour améliorer la valeur technologique des farines commerciales pour la biscuiterie (Chapitre 5)

« Un savant dans son laboratoire n'est pas seulement un technicien, c'est aussi un enfant placé en face de phénomènes naturels qui l'impressionnent comme un conte de fées»

Marie Curie (1867-1934)

CHAPTER II

Influence of a soft wheat flour patent, middle-cut and clear flour streams on dough rheology and cookie characteristics using three recipes.

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1-Résumé

Trois courants de mouture, soit les farines patente (P1), intermédiaire (P2) et basses (CLR) ont été incorporés individuellement ou en combinaisons dans trois recettes biscuitières dont les proportions farine, sucre, gras et eau sont très variables. Les biscuits préparés avec la farine basse étaient sensiblement plus denses et plus durs, avec une pâte à consistance élevée et une surface inférieure, par comparaison aux biscuits préparés avec les farines patente et intermédiaire. Tout changement apporté dans les proportions relatives de ces trois courants de mouture résulte en des pâtes de consistances variables et aussi des produits finis avec des caractéristiques dimensionnelles et de texture très différentes. L'analyse de régression par les moindres carrés partiels a révélé une corrélation positive de la consistance de la pâte ainsi que de la densité et de la dureté des biscuits avec la fraction CLR et CLR^2, et, dans une moindre mesure, avec les interactions P1*P2 pour les trois recettes; par ailleurs, on a constaté un lien négatif entre ces dernières fractions et P1, et un lien positif entre la fraction P1 et la surface des produits des recettes de pâte laminée et à la rotative. D'après les coefficients de régression (C.R.), une fraction donnée ne se comporte pas de la même manière selon qu'elle est utilisée seule ou en combinaison avec une ou plusieurs autres fractions et ce, dans les trois recettes et pour toutes les variables étudiées; ainsi, la consistance de la pâte s'est révélée particulièrement sensible à l'influence de P1 (-C.R.) et de P2*ClR (+C.R.), pour la recette de pâte laminée, à CLR et à CLR^{*2} (+C.R.) ainsi qu'à P1*CLR et P2*CLR (-C.R.) pour la recette de pâte à la rotative, et à CLR(+C.R.) et à P2*CLR(-C.R.) pour la recette, pâte coupe-fil.

Les paramètres P et P/L de l'alvéographe, les propriétés viscoélastiques des pâtes farine /eau exprimées par les indices Tla et k du test compression/relaxation biaxiale, et la granulométrie ont permis de prédire la consistance des trois pâtes($R \geq 0,75$). La consistance de la pâte laminée était corrélée avec la teneur en protéines des farines ($R \geq 0,79$). La densité des cookies à la rotative était corrélée avec la granulométrie de la farine, sa teneur en protéines et les valeurs de Tla, k des pâtes; quant à la surface des cookies, elle était prédite selon la valeur de la capacité de rétention d'eau alcaline ou « AWRC» ($R \geq 0,85$).

2-Abstract

Commercial patent (P1), middle-cut (P2) and clear (CLR) mill stream fractions which exhibited a wide range of physico-chemical characteristics were incorporated alone or in combination into three cookie recipes at various flour: sugar: fat: water ratios. Cookies prepared with the clear fraction had higher dough consistency and were notably denser and harder with lower surface than those made with the patent and middle-cut fractions. Varying the ratios of the P1, P2 and CLR flour fractions produced cookies that exhibited superior or inferior dough consistencies, and end-product quality attributes than the commercial soft wheat flour. The influence of the flour fractions and their interaction (main, interaction and square effect) on the dough rheology and cookie characteristics was studied by the Partial Least Square (PLS) regression analysis. Dough consistency, cookie's density and hardness were all positively correlated to the CLR and CLR^2 variables, and to a lower extent to P1*P2 and P2*CLR for the three recipes. Regression coefficients indicated a stream fraction will not behave similarly when alone or in combination in the three cookie recipes for the variables studied, as dough consistency, for instance, is particularly influenced by P1 (-R.C) and P2*CLR (-R.C) for the laminated recipe and CLR (+RC) for the rotary and the wire-cut recipes.

The P alveograph's parameter, lower granulometry and protein physico-chemical parameters allow the prediction of the cookies dough consistencies ($R \geq 0.75$) and also the rotary's cookie density. Moreover, the viscoelastic properties of a simple flour/water dough could complement the physico-chemical parameters to predict the consistency of the laminated biscuit recipe ($R \geq 0.82$). Both the cookies volume and surface could not be predicted from neither the rheological nor the physico-chemical parameters.

3-Introduction

Cookie manufacturers observe disparity in the functionality of soft wheat flour over various cookie recipes and processes. Uncontrolled cookie dimensions and density, variation in the cookie dimensions, texture and surface appearance and too consistent or sticky dough are some examples of technological difficulties encountered during processing of these products. The sugar-snap cookie recipe (AACC 10-52) is commonly used to evaluate soft wheat flour quality. Soft wheat flour that can produce large cookie spread with fine grain texture is considered as good quality flour (Gaines, 1993). However, it should be recognized that an ideal flour for a specific cookie product may not be ideal for another (Finney, 1989; Rogers et al., 1993). The wide variation in the sugar, fat and flour contents in various cookie recipes may account for such trends (Abboud et al., 1985a; Gaines et al., 1996; Maache Rezzoug et al., 1998 b; Manohar and Rao, 1999a,b; Zoulias et al., 2002). Flour functionality is influenced by many factors, namely the type of wheat cultivar, the kernel hardness variability within one cultivar due to crop season and growing conditions, the differences in the commercial milling processes and the variation in flour protein content (Nemeth et al., 1994; Gaines et al., 1996). The flour, as a final product of wheat milling is made up by a combination of partial products coming from a variety of size reduction and sieving procedure sequences, referred as streams, which the wheat grains, undergo in a milling diagram. Flour streams of a specific cultivar usually have different levels of grain components (MacMasters et al., 1971; Posner and Hibbs, 1997; Berton et al., 2001); a progressive increase in protein content is noted from the first to the last break and such a protein gradient results in different qualities and quantities of the derived streams (Kent and Evers 1969; Ranhottra et al., 1990). Tail-end streams have higher starch damage and pentosans content, weaker dough characteristics, whereas the patent flour, the purest flour from the mills, contains less starch, less protein, as well as protein with different functionality. Such compositional variations in the flour streams affect dough rheology and baking performance (Holas and Tipples, 1978; Gaines et al., 1996). The miller's task is to ensure that the flour composition and its performance characteristics are kept within a narrow range by appropriate selection of the wheat cultivars and blending several streams of flour varying in protein and ash content (Nelson and MacDonald, 1977; Dubé et al.,1987). A large cookie spread is generally associated with soft wheat flour of low protein and smaller granulometry and this can be achieved by appropriate blending of flour streams (Finney 1989). Flour with most of the bran and germ removed, representing about 72% of the kernel, is termed straight grade flour and the streams are usually treated separately as patent, middle-cut and clear flour (Hoseney, 1994). There has been already a lot of work done on trying to improve the understanding of the role of fat, sugar and water on the dimensional and textural properties of cookies (Hoseney and Rogers 1994; Manohar and Rao, 1999a,b; Zoulias et al., 2002). However, little attention has been devoted to the influence of the patent, middle-cut and the clear flour streams used alone or in combination on the quality attributes of cookies and dry biscuits. Hence, the objective of this study was to define the physico-chemical characteristics of patent, middle-cut and clear fractions when added alone or in combination into cookie recipes and to determine their impact on the dough rheology and the dimensional and textural characteristics of products made up with three different cookie recipes, typical of wire-cut, laminated and rotary processes.

4-Materials and methods

4.1-Flour streams fractions

Milling stream fractions used for this study were obtained from commercial cultivars consisting of 70% soft white winter (CEWW) wheat and 30% soft red winter (CESR) wheat grown in the Eastern region of Canada, obtained from the 1998 harvest. The unbleached and untreated flour fractions were collected from a pilot plant using a typical commercial flow at the Robin Hood milling facility, Port Colborne, Ont, Can. The stream fractions were referred by the miller as the patent or P1 (first 45% w/w of the material obtained by a 72% extraction rate), middle-cut or P2 (middle 20% w/w) and clear fraction or CLR (tail-end 7% w/w of the material). Blending of the flour streams constituted the straight grade flour. To assess the effect of the ratio of fractions on cookie making performance, the fractions were mixed together in various proportions for 30 min using a Reliance Double Action mixer VI-07 (Leland South West, Sagineau, Texas, 76179) as shown in Table 2.1.

For the 1998 crop, the commercial soft wheat flour blend consisted of 40, 48 and 12% w/w of the patent, middle-cut and clear stream fractions, respectively (i.e. sample 4). In the present study, a maximum level of 30% clear was judged acceptable to mimic all possible flour combinations produced under commercial plant production. All the fractions and blends were stored in the dark at –20°C until used.

4.2-Flour streams physico-chemical analysis

Moisture content of the flour fractions and combinations were determined according to the AACC method 44-15 (AACC, 2000). The protein content was determined by the Leco nitrogen/protein determination FP-428 (Leco Instruments Ltd, Mississauga, Ont., Canada); the nitrogen content was multiplied by 5.7. Alkaline water retention capacity (AWRC) was determined according to the AACC method 56-10 (2000). Damaged starch was determined spectrophotometrically according to the AACC method 76-31 and ash content was determined by the method of AACC 08-01. Granularity was carried out according to the Alpine Air-Jet sieve instruction manual using the 20, 38 and 75 Φm screen air jet sieve A200LS with a 200 mm diameter screen. The amount of large polymeric glutenin or insoluble glutenin was measured by the method of Sapirstein and Johnson (2000).

4.3-Dough Rheological tests

A portion of 250 g of flour (moist basis) was mixed for 8 min at 24°C in the Chopin alveograph kneader with a 2.5 % NaCl solution at constant total water content of 43.1% dough basis. For each flour sample, 2 doughs were prepared; one for the alveograph test and one for the relaxation test using the Lubricated Squeezing Flow (LSF) method. The dough samples were rested for 20 min at 25°C prior to alveograph testing and for 35 min in the LSF test.

The following Alveogram values were determined using the Chopin alveograph equipped with hydrostatically controlled air flow unit: W, work (10^{-4}J) or deformation energy;G, swelling index; P (mm), maximum pressure for tenacity; L (mm), extensibility; P/L configuration ratio stability, according to the AACC method 54-30 (2000).

Relaxation after lubricated squeezing flow (LSF) was performed according to the method of Launay and Bartolucci (1997) and Bartolucci and Launay (2000). The discs of the rested dough (35 min at 25°C) had 32 mm diameter and 12 mm height and were coated with a thin paraffin oil (viscosity of 25 mPa.s) just before the test to avoid sample dehydration. A texture analyzer (TAXT2, Stable Microsystems, Surrey) with a 10 cm diameter plate was used at a constant crosshead speed (0.2 $mm.s^{-1}$) to reduce sample thickness by 50% of its initial value. After stopping the crosshead, the decay of the compression force was followed during 150 sec (50 data points/sec). The data were fitted to the equation of the model corresponding to a Maxwell liquid in which the viscosity follows the Power law with the deformation speed, introducing a non-linear behaviour. The equation can be written as:

$$\sigma(t) = \sigma_i \left[1 + k\left(\frac{1}{n} - 1\right)t\right]^{\frac{n}{n-1}}$$

where σ is the stress (Pa) and $\sigma = \sigma_i$ at t=o; *k* (s^{-1}) corresponds to the rate of relaxation speed and *n*, is the flow behaviour index (dimensionless). This equation was used to fit (software Table curve 2D, Jandel) stress relaxation data obtained with the lubricated squeezing flow method. The *k* and *n* symbols were used for the relaxation after lubricated squeezing flow and $F=F_i$ at start of relaxation in Newtons, the dough's hardness; *k* (s^{-1}), is the rate of relaxation, *n*, the flow behaviour index, and Tla (s) calculated as the half relaxation time according to Bartolucci and Launay (2000).

Cookies dough consistency index was evaluated by performing a texture profile analysis (TPA) with a texture analyzer (Stable Microsystems, Surrey, UK, and 25 kg load cell). The cookie doughs were rested in plastic bags immersed into a water bath at 25°C for 30 min. The consistency of the deaerated doughs compacted into the plastic pot was carried out according to the method of Miller (1985) using a 6.3mm diameter cylindrical head (TA–23 probe) and penetration at 2.5mm/sec to a depth of 15mm. Plots of force versus time were thus generated; the data are the average values of 9 measurements for each cookie dough.

For the extrusion test, the extrusion test cell consisted of a 100mm long stainless steel cylinder having an internal diameter of 50mm and a spherical hole of 10mm at the base. Dough of the laminated recipe (100g) was rolled into a cylindrical shape and pressed firmly into the extrusion cell. The force-distance curve during the compression of 50% of the total dough length was recorded with an Instron Universal Food Tester (Model 4201) equipped with a 100kg load cell, using a crosshead speed of 10mm/min. The characteristics determined from the curve included the force-distance curve, the peak extrusion force and the area under the curve representing the extrusion energy (Joule). The speed of exit of the dough was recorded and expressed in cm/min (Tharrault, 1994c). Six measurements were performed for the determination of the mean value for each dough formulation.

4.4- Cookie making procedures

All ingredient levels are expressed on a flour weight basis (f.w.b) as tabulated in Table 2.2

a) wire-cut recipe: Sugar, cereal flakes and salt were weighted together separately from the blend of flour and sodium bicarbonate and also shortening. All the ingredients were tempered for 24 hours at 20°C in a temperature controlled cabinet. Dry raisins (20% d.b) were crushed in a kitchen aid mill (4-6 mm opening) and finally blended with part of the formulation water to ensure uniform distribution in the dough. A 1.2 kg recipe preparation was carried out by mixing all the ingredients, except flour and sodium bicarbonate in a 10 kg bowl (Model A-120, Hobart, low and medium speed at 106 and 196 rpm) for 5 min at low speed, then another 2 min at low speed. The screened flour and Na bicarbonate were subsequently added to the mix and remixed for another 2 min at low speed and 1 min at medium speed. After resting for 60 min at 27°C, the dough was sheeted once to a thickness of 7 mm, with a pilot scale sheeter (Model SM-250M, Picard Bakers LP, Victoriaville, Quebec, Canada) and punched out manually with a circular cutter of internal diameter of 46.0 mm. After stamping, the dough samples were weighted and deposited on an aluminium tray. Baking was made at 450°C for 6 min in an oven (Model MT-4-8 Picard Bakery, Equipment LPINC, Victoriaville, Quebec).

b) Rotary recipe: Sugar, salt and sodium bicarbonate were weighted together. The remaining ingredients, shortening, water and flour, were weighted separately; all these ingredients were tempered for 12 hours at 20°C. All the ingredients, except the flour, were mixed in a Hobart mixer Model N-120 for 30 sec at low speed for 30 sec at medium speed and twice at high speed, with intermittent bowl wall cleaning between each speed change. Then, the screened flour was added to the bowl and mixed for 12 min at low speed, the total blend being 3.0 kg per batch. Following a rest time of 30 min at 27°C, the dough was moulded in a rotary unit (Jansen and Sons, Rotative, Model Form-Fix 2500, Krefeld) using the maple leaf geometry. The dough pieces were deposited on a baking tray and baked for 3.30 min at 550°C in the rotary oven as mentioned previously.

c) A semi-sweet biscuit, referred as laminated recipe developed by the CTUC (Centre Technique d' Utilisation des Céréales) to decide if a wheat variety could be registered as suitable cultivar for biscuit making was used in this study. In this case, a higher level of flour is used, so the quality of end-products is primarily dependant on flour quality (Tharrault 1994a,b, 1995. A slight modification in the dough preparation was required since the Morton mixer and the laminating unit used by the CTUC laboratory were not available. The dough was prepared with 1.2 kg of flour (100%, moist basis). Sodium bicarbonate, ammonium bicarbonate and sodium pyrophosphate were weighted together and screened with half of the required flour. Then sugar, salt, and half of then screened flour were added into a 10 kg bowl (Model A-120, Hobart) and mixed for 1 minute at low speed. The shortening was then incorporated into the mix and finally the water was added to get a constant water content (24% dough basis). All the ingredients were mixed for 2 min at low speed and 5 min at medium speed. After resting for 30 min at 27°C, the dough was sheeted between 2 cylinders of the pilot scale sheeter Model SM-250M (Picard Bakers LP, Victoriaville, Quebec, Canada). The gap settings were as follows: gap 15 mm-1 passage, 7.5 mm-1passage, 4.5 mm-1passage, 2.5 mm-2 passages with folding between each passage through the sheeter and 1.75 mm-2 passages with 1 folding and a 30s. resting time between each passage, at the last reduction step. Then, the dough was punched out manually with a square cutter 60 x 60 mm. The biscuits were then baked immediately at 250°C for 5.5 min in the rotary oven.

4.5-Cookie dimensional and textural properties

Dimensions of the biscuits (length, width, diameter, thickness) were evaluated using a Vernier Caliper on a set of 12 biscuits for each recipe, after 2 weeks storage in aluminium pouches and placed in a tight container. The cookie surface (SU), expressed in cm^2, was calculated as L(length)*w (width) for the cutting cookie, πr^2 for the wire cut and $L^{1/2}$* $w^{1/2}$(g) for the rotary, whereas the volume (VO), expressed in ml, was measured with a volumeter filled with rapeseed. Density (DE) was calculated directly from the weight and volume data and recorded as the average value of 12 measurements. A three-point bend test was carried out on 12 biscuits using a Texture Analyser ($TATX_2$) Stable Microsystem, (25 kg Load Cell), Surrey, Mono Research to evaluate cookie hardness. Biscuits were placed on base beams that were 3.5 cm apart for the laminated recipe and 2.5 cm apart for the wire-cut and rotary recipes, respectively, using the 3-point bending rig (HDP/3PB, fixture TA-92). The analyzer was set at a return to start cycle, with a speed of 0.5 mm/sec and a return distance of 3cm. The force required to break the biscuits was recorded in this snap-test with 12 biscuits stored for 2 weeks in aluminium pouches at room temperature.

4.6-Statistics

An experimental design (Myers and Montgomery, 1995) was used to study the effect of the 3 milling streams fractions (P1, P2 and CLR for the patent, the middle-cut and clear fractions, respectively). The combination ratios were selected on the basis of possible ratios encountered in a commercial straight grade flour. The factor level of CRL was constrained to a maximum of 30% to mimic the maximum level that can be found in commercial straight grade flours. Two replicates were used for all the measurements.

The PLS (Partial least Square) regression was used to model the relationship between the variation in response variables (Y variables) to the variation of predictors (X variables) and also the interrelationships between the X data and those of the Y variables, employing the Unscrambler v 8.0 software (Camo Smart Inc,Woodbridge, NJ, 07095, USA). The PLS regression is a projection method that decomposes variations within the X-space (predictors, e.g. design variables) and Y-space (responses to be predicted) along a set of PLS components (referred as PCs). For each dimension of the model (i.e. PC1, PC2, etc), the summary of the X is ``biased`` so that it is as correlated as possible to the summary of Y, so the projection process manages to capture the variations in X that can explain variations in Y. When building the PLS model, the X-data are centered; i.e. further results will be interpreted as deviations from an average situation, which is the overall centroid of the design. The Y-data are also centered, i.e. further results will be interpreted as an increase or decrease compared to the average response values and finally the mixture constraint is implicitly taken into account in the model; i.e. the regression coefficients will show the impact of variations in each mixture component when the other ingredients compensate with equal proportions (Maartens and Martens, 1986). During the model development, all the Y variables may simultaneously influence the compression of the X block. In a mixture design, the interaction and square effects are linked so P1, P2 and CLR respectively vary from 0 to 1 and P1+P2+CLR = 1 for all mixtures. Therefore, CLR can be re-written as 1 - (P1+P2) and as a consequence, the square effect CLR^2 can also be re-written as $(1-(P1+P2))^2 = 1 + P1*P1 + P2*P2^2 -2P1 - 2P2 + 2P1*P2$. The square effect allow to see if there is curvature (non linear) relationship between response variation and the factor. Similarly, P1*CLR can be re-expressed as P1*(1-P1-P2) = P1 – P1*P1 – P1*P2, which shows that interactions cannot be interpreted without also taking into account main effects and square effects.

The regression coefficient from a PLS model summarizes the relationship between all predictors and a given response for a model with a particular number of components. The raw coefficients are those that may be used to write the model equation between a Y and n X-variables in original units:

$Y = B_0 + B_1 * X\text{-variable}_1 + B_2 * X\text{-variable}_2 + \ldots\ldots.. + B_n*X_n$

The scaled regression coefficients take into account the weighting options (1/Sdev) selected for the analysis. Since all predictors are brought back to the same scale, the coefficients show the relative importance of the X variables in the model and the importance of the effects can be assessed by the size of the regression coefficients.

Simple linear regression analysis using Proc reg (SAS program) was also used to determine the significant relations between recipe parameters and flour physico-chemical attributes. The results include the intercept, the slope, the p-value and the r^2. The relation can be defined by an equation determined by the slope and the intercept: Response variable= Intercept+ slope*variable.

5-Results and Discussion

5.1-Physico-chemical characteristics of flour streams

Table 2.3 shows some of the quality parameters related to flour physico-chemical properties for the three flour streams and the 6 combinations investigated in this study. The protein content increased with the patent, middle-cut and clear flour from 7.68, 9.31 to 11, 9%, respectively, and it varied from 8.62 to 10.1% for the remaining combinations (samples 4-9); these levels are typical of values reported for soft wheat flour in cookie manufacturing, as reflected in the 1998 commercial flour fraction combination (0.40/0.48/0.12 of P1/P2/CLR), respectively, which corresponds to the control or sample 4. In parallel, the insoluble glutenin content increased proportionally since the protein content rises from the central to the peripheral portion of the kernel endosperm; this fraction, also referred as insoluble or aggregated protein (Sapirstein and Johnson, 2000), has been reported to promote elasticity and accelerate retraction of a semi-sweet biscuit dough, thus exerting a negative effect on product dimensions (Tjomb, 1995).

According to Posner and Hibbs (1997) smaller granularity is also noted with the patent, as less flour is retained in the 38 and 75μm screens than for the middle-cut and clear flour stream fractions. Smallest particles of less than 17μm consist of interstitial protein and small starch fragments; this is usually the high protein fraction. Ash content increased from 0.403 to 0.984% and starch damage increased from 3.2 to 5.1% in the patent to the clear fraction (Table 2.3). The cytoplasmic origin of these fractions explains such trends; the central endosperm walls being easier to mill than the peripheral walls resulting in a greater concentration of starch and lower protein and ash contents in the head fractions. The AWRC values or the amount of alkaline water held by the flour after slurring it in excess water and then centrifuging the slurry rose from 62.6 to 71.1% from the patent to the clear fraction, implying that the peripheral layers of the endosperm are more hydrophilic in nature. Low AWRC values are considered as a predictor for soft wheat quality due to its inverse relationship with the cookie spread (Yamazaki, 1953; Finney, 1989; Nemeth et al., 1994; Labuschagne et al., 1996). The variation in water absorption is due to the intrinsic differences in protein content and quality, in starch damage and pentosan content. An increase of protein content and starch damage levels coupled with a decrease of flour particle size; generally tend to increase, flour water absorption or AWRC (Holas and Tipples, 1978). Moreover, pentosans are also reported to have a relatively large effect on flour's water sorption (Meuser and Suckow, 1986; Cottenet, 1986).

The alveograph and lubricated squeezing flow (LSF) data of the various flour stream fractions and their combinations are presented in Table 2.4. The ranges for the alveograph flour properties were 90 to 96 * 10^{-4} $J.g^{-1}$ for strength (W), 30.73 to 50.18 for tenacity (P), 18.55 to 24.96 for (G) (swelling index), and 0.270 to 0.730 for configuration ratio P/L. The energy of deformation W appears to be related to the protein content and any increase of the W value is reported to reduce the biscuit length (Bartolucci and Launay 2000). All flour combinations, under study, demonstrated alveograph properties typical of soft wheat flour; the clear fraction had higher W, P and also P/L ratio due to the higher protein content. The (LSF) indices show that the initial force of relaxation (Fi) of simple water/flour dough, which corresponds to the dough hardness, is substantially higher in the clear fraction, 2.78 N, versus 1.82 N for the middle cut and 1.54 N for the patent. The time for half relaxation (Tla) increased substantially for the clear fraction, but was not modified substantially in some of the flour combinations. In contrast, the k values decreased with increasing protein content of the flour (samples 7 and 3). Both the Tla and k responses can be explained in terms of the elastic recovery of the dough due to their viscoelastic properties: the dough when submitted to stress during the sheeting process can accumulate mechanical energy, inducing partial strain recovery. A high k value indicates that the energy stored in dough dissipates more rapidly and as a result, the dough retraction will be minimized and the final biscuit length will be higher. The higher k values of the patent 1.11 versus $0.118s^{-1}$ for the clear confirm that the internal constraints accumulated in the dough are relaxing more rapidly in the patent, so that the recovery or retraction is lower resulting in higher final cookie length especially with the laminated recipe (Launay and Bartolucci, 1997; Renard and Théry, 1998).

5.2-Cookie dough rheology and dimensional and textural characteristics of cookies

Three cookie recipes were compared to study the influence of different flour stream fractions added alone or in combination on selected rheological parameters based on their recognized relation with the cookie making quality of soft wheat flour, namely dough consistency index of the dough and dimensions, as well as density, volume, surface and hardness of the cookies (Miller, 1985). The ratios of flour: sugar: fat: water were 100:52:49:21 for the wire cut, 100:24:24:10 for the rotary and 100:30:8:26 for the laminated recipes, on 14% moisture basis, respectively. The proportions of added sugar/fat in some cookie recipes are large enough to interfere with the gluten network development; i.e. proteins are insufficiently hydrated and the starch, also competing for the added water in the recipe, can largely be ungelatinized in the biscuit structure (Flint et al., 1970). Since fat and sugar content tend to mask and even alter the flour functionality (Slade and Levine, 1994a, b), it was important to consider the impact of these ingredients in different cookie recipes. The flour/sugar/fat and water ratios of the wire cut recipe could be easily compared to the wire cut AACC 10-53 cookie formulation (100/42/40/22) which has been reported as being more sensitive to variation in flour protein content than the AACC 10-52 sugar-snap cookie (100/60/15/30). The rotary and laminated recipes have much lower sugar and fat content than the wire-cut recipe, and the hydration level of the laminated recipe is ~ 1.7 times higher than the wire-cut recipe as shown in Table 2.2. Previous studies have demonstrated that the variability in dough consistency can explain the variation in weight and cookie dimensions. A higher consistency results in a reduced cookie spread as cookie spread seems to be controlled by the dough viscosity; a lower viscosity allows the cookie to spread faster (Hoseney and Rogers, 1994; Miller et al., 1997). Due to the lower water content, the rotary dough's consistency is ~6-7 times higher than the laminated and wire-cut recipes (Table 2.5). The clear fraction by itself and the combination middle-cut and clear (0.7/0.3) increased tremendously the consistency of the dough in all three recipes; this is due to the higher protein contribution of the peripheral fraction of the wheat endosperm. On the other hand, cookies produced with the patent flour, the central portion of the endosperm, had the lowest consistency and were followed by the middle-cut, irrespective of the recipes. The variation noted in dough consistency of the different flour mixture combinations (samples 4-9) was principally related to the level of the clear; i.e., any increase or decrease of this fraction in the recipe was accompanied by an increase or decrease of the consistency values of the respective cookie dough. Also, the cookies made with the control flour (sample 4) or with the commercial flour produced by the plant with a ratio of 40/48/12% (P1/P2/ClR), respectively, had very similar consistency to the P2 fraction alone, suggesting that this ratio may produce an end-product of similar quality to the middle-cut flour, likely due to the higher contribution of the P2 fraction.

Due to the various shapes of cookies, i.e., square for the laminated, circular for the rotary and maple leaf shape for the wire cut, the cookie's volumes were measured with the rapeseed displacement method. Volume was selected as a dimensional index to compare the flour functionality in the three recipes as this criterion has been reported to be a good descriptor of soft wheat flour quality (Slade et al., 1989). Cookies produced with the clear fraction demonstrated the lowest volumes with the wire-cut and rotary recipes. In contrast, in the laminated recipe, the clear fraction gave cookies with increased volume, along with a concomitant decrease of the surface, expressed as length*width of the square semi-sweet biscuit. These observations can be related to the retraction of the dough which was visible with the clear fraction during the laminating process, resulting in a diminution of the cookie length and an increase of the thickness and volume. The protein content of the clear fraction may explain this observation with the laminated recipe that might be more sensitive to the variation in protein content due to lower sugar and fat levels and a more hydrated recipe than the wire-cut and rotary recipes. The results of the flour fraction blends also indicated that the combinations of the fractions studied resulted in cookie volumes that are almost similar or slightly superior (sample 8) to the standard flour (sample 4) with all three recipes. These observations may partly explain some of the contradictory data reported in the literature, on the influence of protein on cookie spread. For example, Kaldy and Rubenthaler (1987), Gaines (1991) and Souza et al. (1994) showed a negative impact of the protein on spread, while Nemeth et al (1994) found no correlation between cookie diameter and protein content for Canadian soft wheats, but within the same cultivar, the cookie diameter was mainly dependent on protein content ($r= -0.87$) when fertilization levels vary. Cookies with the largest surface were also produced with the patent and the control flour (sample 4), while the clear by itself showed significantly smaller surface of the cookies made with the wire-cut and cutting recipes. Surface and volume parameters of cookies made by the rotary method were the least influenced by flour composition (Table 2.5). The low hydration of this recipe may be responsible for the insufficient hydration of proteins and their conversion into a properly developed gluten network. As a result, the starch granules embedded in the protein matrix may not be changed during baking as confirmed by Flint et al. (1970). Density is an important quality parameter for biscuit, particularly in predicting crispness. (Bartolucci and Launay, 2000) It is expressed as the ratio of weight/volume and follows the reverse of the volume trend. For the three recipes, the clear fraction by itself increased substantially this parameter, especially in the cases of wire-cut and rotary cookies. In contrast, the incorporation of the patent flour produced cookies with the lowest density values. Most of the flour combinations yielded higher density than the standard flour (sample 4) for the wire-cut and rotary recipes. Instead, for the laminated recipe, the density value of cookies made with most of the flour blends were lower than for the standard flour. This may be explained by the complexity of the interaction between the fractions with recipes containing higher flour content and lower sugar and fat levels.

Cookie texture is reported to be related to the relationship between geometry and water content and also to the dependency of the mechanical properties on water content and sugar concentration (Slade and Levine, 1994a, b). The hardest cookies were produced by the wire-cut recipe, likely due to the high ratio of sugar and fat to water content. Substantial increase in the hardness was noted with the clear flour irrespective of the cookies recipes, whilst a softer texture is obtained with the use of the patent flour in these recipes (Table 2.5). These results are in agreement with the work of Kiger and Kiger (1967) and Wade (1995) as cookie's hardness increased with protein level, especially above 10%. The lowest hardness values obtained with the patent confirmed the view that earlier streams produce softer cookies, whereas later milling reduction streams produce the hardest cookies (Gaines et al 1996). The influence of the fraction ratios indicated that the responses of this attribute differ with each recipe, as highest hardness values were obtained with a ratio of 0.425/0.425/0.15 for the wire-cut, 0.3/0.5/0.2 for the rotary and 0/0.7/0.3 for the laminated (cutting) recipe, respectively. On the other hand, softer cookies were produced by the ratios of 0.30/0.50/0.2 for the wire cut, 0.333/0.660/0 for the rotary and 0.3/0.5/0.2 for the laminated recipe, respectively. The higher dough consistency of the clear fraction also yielded higher cookie firmness. However, this was not the case for the various fraction blends due to the complex interactions among their constituents; an increase in dough consistency was not necessarily accompanied by an increase in cookie hardness. This further indicates that the other dough ingredients (sugar, fat and water) used in each recipe exert an important influence in altering the final cookie texture during baking and cooling.

5.3-Influence of the flour stream fractions and their interactions on the dough properties and cookie making quality

The PLS regression was used to explore the influence of the three flour fractions and their interaction (main, interaction and square effects) on the dough and cookie parameters. In this multivariate analysis, the X-block is designed with the independent variables P1, P2, CLR and P1*P1, P2*P2, CLR*CLR, P1*P2, P1*CLR and P2*CLR. The Y-block includes all the responses or dependent variables such as volume, density, surface, hardness, speed and energy of extrusion and consistency of the dough. The response variables are known to be influenced by the physico-chemical properties of the flour stream fractions which, in turn, affect end-product quality. The PLS method was preferred over the Principal Component Analysis (PCA), as a regression method, since it handles the correlation of X and Y variables based on the nature of two types of blocks and it allows the display of a sample and variable maps, making easier the interpretation of the models. Additionally, it quantitatively describes the variation in X that is relevant to Y-variations.
The 2D scatter plot of X loading weights and Y-loadings of the model for the laminated recipe is presented in Figure 2.1, indicating that 72% of the variation in X (flour streams fractions and interactions) and 59% of the variation in Y (dough and cookie parameters) can be explained by the first two components. The explained variance of the X-matrix is 42% for PC1 and 30% for PC2 and for the Y-matrix, the variance is 44% for PC1 and 15% for PC2. This plot allows the detection of the important predictors and furthermore the understanding of the relationships between the X- and Y-variables. In the X-loading weight and Y-loading for the first two components from PLS, for the laminated recipe presented in Figure 2.1, predictors P2 (middle-cut) and CLR (clear) had positive link with the volume,

consistency, hardness, thickness and density responses since they are closely located to these fractions. The figure also indicates that the clear stream fraction variables, CLR and CLR^{*2} (square effect), mainly influence these attributes in this recipe. Predictors projected in the opposite direction had a negative link, e.g. predictor P1 versus the above variables. Furthermore, the CLR^{*2} and P1*P2 interactions, located close to the CLR, are positively linked to CLR indicating that these variables also exert an influence on the above attributes. Conversely, these fractions are negatively linked to P1 and interactions P2*CLR, suggesting that higher dough consistency will have an adverse effect on cookie's surface development with this particular recipe. The speed predictor projected of PC1 is not well represented in the plot. Both the speed and energy of extrusion are positively linked to P2 and P1*CLR and negatively linked to P1 and interactions $P1^{*2}$ and $P2^{*2}$ on the PC2 axis. The flow behaviour of the laminated biscuit dough by an extrusion test is reported to provide valuable information on the dough's machinability as the extrusion energy is controlled by the viscous properties of the dough, and the speed by its elastic properties. The ratio of speed and energy of extrusion is commonly used to monitor dough machinability of the cutting dough; a high ratio suggests that the laminating process may be difficult (i.e., a low speed of extrusion is translated to a more pronounced reduction of the biscuit length) (Tharrault, 1994c). This plot confirms the extreme complexity of the flour system with regard to its functionality in cookie making, especially when different flour streams are mixed in varying ratios and that the model precision is greatly improved by taking into account the interactions among the flour streams fractions. This also indicates that one fraction will not behave similarly when used alone or in combination.

The respective plots of the rotary and wire-cut recipes, presented in Figures 2.2 and 2.3, showed that the percentage of the explained variation in X variables (predictors) is slightly lower (3-8%) by the the first 2 PCs, but explained the variation in Y (responses) slightly higher (5-7%) than the latter recipe. The multivariate analysis of these two recipes confirmed that the dough consistency, cookie density and firmness, located on the right side of the plots were, similarly to the latter recipe, all positively related to the CLR and CLR^{*2}, to a lower extent to P1*P2 and negatively to P1 and the P2*CLR interaction, suggesting that these flour combinations had similar influence on the above parameters regardless of the recipe composition. In the laminated recipe, the PC2 component differentiated between the P1*[2] and P1 with P2; however, these variables are not similarly linked with end-product quality. For instance, the surface of the laminated recipe is positively linked to P2*CLR but negatively linked to CLR. On the other hand, for the rotary recipe, the surface is linked positively to the P1*CLR and negatively to CLR, while for the wire-cut, the surface is negatively linked to P1*CLR. The volume parameter also responded differently to the fractions and interactions as it is positively linked to P1*P2 with the laminated recipe and to P1*CLR and P2*CLR with the two others recipes. The unexpected responses of both volume and surface could be attributed to the different cookie geometries (square, circular and maple leaf shape), the complex role of the fractions, their interactions and the effects of different flour: sugar: fat: water ratios on end-product quality.

Regression coefficient analysis (R.C) was also conducted to better determine the relative importance of the fractions, their interaction and square effects on the cookie quality attributes; i.e. dough consistency, volume, density and hardness. To assess the variation of impact in each mixture, the following rule of thumb was recommended by Camo Smart Inc: if the R.C for a variable is larger than 0.2 in absolute value, then the effect of that variable is most probably important; if it is smaller than 0.1, then the effect is negligible and for values between 0.1 and 0.2, no certain conclusion can be drawn. The relationships between the predictors and the property responses (dough consistency, cookie volume, density and hardness) generated from the PLS models of the three recipes with the P1, P2, CLR flour stream fractions, their interactions and the square effects are illustrated in Figures 2.4 to 2.6. The P2*CLR and P1 of the laminated yielded a negative link (-R.C) with the dough consistency. In parallel, the CLR for the wire-cut and rotary recipes demonstrated a positive link (+R.C) with the same parameter, suggesting that these variables are important on dough consistency (R.C $\geq$ 0.2). These data confirmed the important role of the clear fraction and the interactions between specific flour fractions; such predictors may vary with the recipe composition. For the volume, most of the R.C's absolute values were lower than 0.20 confirming the negligible impact of the predictors for this parameter. The cookie's density yielded superior positive correlation with the CLR and CLR^2 of the rotary recipe, but negative RC with P2*CLR of the rotary and laminated. Hardness (firmness) of the cookies formulated with the laminated recipe demonstrated negative R.C with P2*CLR and P1, and positive R.C with the CLR^{*2} and CLR was observed for the wire-cut cookies only. These data confirmed that there are only limited numbers of predictors that influence significantly the response variables of this model; as reflected by the values of the regression coefficients.

5.4-Relationship between flour physico-chemical properties and dough properties and end-products quality

Previous data have demonstrated the effect of each fraction and their interaction for each cookie recipe quality attribute and also the correlation or redundancy among the end-product parameters with each predictor. The linear regression to predict the relationship between physico-chemical parameters of the flour fractions and dough and end-products quality attributes for the three recipes are presented in Tables 2.6 to 2.8. The response variables can be mathematically predicted from the regression equation with the variable intercept and slope values where the response variable= Intercept+ slope*variable. For the three recipes, the dough consistency response variables appeared to be the most easily predicted by the physico-chemical attributes of the flour, the R^2 values (proportion of variance explained by the regression equation) being superior to the end-products parameters such as volume, density and firmness of the cookies. The dough consistency of the laminated recipe showed a strong relationship with the viscoelastic properties of the flour dough as reflected by the Tla, Ka and Na indices of the LSF test (R2 > 0.83). Additionally, the alveograph data used for assessing the rheological properties of the flour dough by measurement of the alveograph P and P/L indices also gave significant relationships with the dough consistency of the rotary and wire-cut recipes ($R^2 > 0.75$). The consistency was also related to smaller granulometry, e.g. 20μm for the three recipes ($R^2 > 0.73$) and with the protein and insoluble glutenin of the laminated recipe only ($R^2 > 0.79$). With regard to the end-product quality, only the surface of the wire-cut demonstrated a good relationship with the AWRC ($R^2 > 0.85$) and the alveograph P and P/l values. Cookie density of the rotary recipe was strongly related to the low granulometry, protein content and all the LSF parameters (Fi, Tla, Ka and Na). Protein and insoluble glutenin content did not appear to influence the surface, volume and firmness attributes of the cookies made with all three recipes. In contrast, it showed a strong relationship with the density of the rotary ($R^2 > 0.88$) and to a lesser extent to the wire-cut, as well as also with the dough consistency of the wire- cut and laminated recipes. Moreover, more variables could be used with the laminated recipe followed by the wire-cut recipe for the prediction of dough rheological and end-product parameters, than for the rotary recipe, suggesting that these recipes with higher ratio of water may be more sensitive to detect property changes with flours exhibiting variation in their biochemical composition. From a practical standpoint, these regression data can be used to select an adequate parameter to measure and predict rapidly its influence on a particular quality attribute. For instance, since the cookie dough density of the rotary recipe is being correlated with the protein content, the latter might be adopted because of ease of implementation in a cookie manufacturing facility.

6-Conclusions

As the overriding aspect of flour quality is consistency of performance, an endeavour not simple or straightforward, an appropriate mixture of milling streams appears to be a viable alternative to improve the end-use of the soft wheat flour to meet quality requirements for a given cookie recipe. More importantly, from a practical point of view, any combination of milling streams of different refinement in terms of ash and protein content cause greater changes than an individual flour fraction. However, because of the complex interactions among constituents of different flour stream fractions on the quality attributes, coupled with the low variation in protein content of standard commercial soft wheat flours, one cannot extrapolate the data from one recipe to another due to the differences in recipe composition; i.e. the contributions of water, sugar and fat are far from being negligible. In addition to the empirical methods (e.g. alveograph) used regularly to assess the rheological behaviour of wheat flour doughs, their viscoelastic behaviour by stress relaxation following biaxial extension provided interesting information to predict the dough consistency. Consistency appeared to be linked to end-product quality attributes such as biscuit's density. Moreover, protein content, granularity and viscoelastic properties of the laminated recipe seem to exert a major influence on the dough consistency, which in turn, appeared to be related to more physico-chemical and rheological parameters than the two others recipes. This work also suggested that the role of soluble and insoluble proteins and pentosans, protein quality versus quantity, as well as lipids endogenous to these flour materials need to be better defined to better understand the flour constituent's contribution to the rheological properties of the dough and biscuit quality attributes.

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Table 2.1: Ratio of flour milling streams

Sample	Patent	Middle-cut	Clear
1	1.0	0	0
2	0	1.0	0
3	0	0	1.0
4*	**0.4**	**0.48**	**0.12**
5	0.3	0.5	0.2
6	0.7	0	0.3
7	0	0.7	0.3
8	0.333	0.667	0
9	0.425	0.425	0.15

Note: *4 equivalent to straight grade or the commercial soft wheat flour

Table 2.2: Cookie recipes composition

	On flour basis		
Ingredient	Rotary recipe	Wire-cut recipe	Laminated recipe*
Flour (moist basis)	100	100	100
Sugar	23.10	54.04	30
Salt	1.00	1.91	0.63
Sodium bicarbonate	0.42	1.91	0.50
Shortening	23.97	46.38	8.0
Water	9.47	20.42	26*
Ammonium carbonate	—	—	1.0
Sodium pyrophosphate	—	—	0.50
Cereal flakes	—	10.64	—
Dry fruit (20°mb.)	—	10.64	—

Note: on 24% moisture dough basis

Table 2.3: Physico-chemical analysis of the fractions and combinations

Sample	Moisture (%)	Granulometry (% retained)			Protein * (%)	Insoluble glutenins (%)	Ash (%)	AWRC
		20 μm	38 μm	75 μm				
1	12.6	63.59	22.06	4.63	7.68	1.21	0.403	62.6
2	12.2	71.91	52.09	25.04	9.31	1.39	0.569	64.2
3	11.3	85.04	49.95	25.36	11.96	1.60	0.984	71.1
4	12.2	70.16	39.82	16.91	8.82	1.36	0.552	64.2
5	12.1	72.04	42.65	18.98	9.20	1.41	0.602	64.8
6	12.2	70.03	30.43	10.85	8.92	1.35	0.577	66.2
7	11.9	75.85	51.45	25.14	10.06	1.48	0.693	65.2
8	12.2	68.45	41.66	18.05	8.62	1.34	0.508	62.5
9	12.2	70.34	39.01	16.41	8.91	1.37	0.561	64.5

Note: Protein and insoluble glutenin contents on 14% moisture basis (m.b)
Damaged starch determined with the AOAC method 08-01 were 3.2, 3.5 and 5.1% for the patent and clear, respectively

Table 2.4: Rheogical properties of the water/flour dough made with various fractions and combinations

Run	Alveograph				Lubricated squeezing flow		
	W 10^{-4} J	G mm	P mm	P/L	Fi (N)	Tla (s)	k (s^{-1})
1	90	21.71	30.73	0.325	1.54	2.10	1.111
2	92	24.96	33.62	0.270	1.82	2.29	0.507
3	96	18.55	50.18	0.730	2.78	2.79	0.118
4	92	22.89	34.45	0.347	1.82	2.36	0.701
5	93	22.70	36.06	0.379	1.92	2.31	0.608
6	90	20.76	36.56	0.447	1.90	2.38	0.807
7	94	23.03	38.59	0.408	2.09	2.20	0.387
8	92	23.63	32.33	0.285	1.71	2.35	0.703
9	92	22.62	34.88	0.362	1.84	2.35	0.704

G: Swelling index
P; resistance to deformation (tenacity)
L: Abcisse to rupture (extensibility)

Table 2.5: Cookie dough consistency and end-product quality parameters of cookie made with various fractions and combinations

	Recipes	1	2	3	4	5	6	7	8	9
Consistency(N)	**Wire-cut**	**1.76**	**1.85**	**2.50**	**1.80**	**1.98**	**2.07**	**2.29**	**2.09**	**1.98**
	Rotary	**14.1**	**16.2**	**24.0**	**14.3**	**16.4**	**16.2**	**16.6**	**14.0**	**14.7**
	Laminated	**2.17**	**2.71**	**3.04**	**2.47**	**2.43**	**2.20**	**2.91**	**2.49**	**2.33**
Volume (ml)	**Wire-cut**	**14.28**	**14.37**	**12.75**	**13.50**	**13.33**	**13.96**	**14.04**	**14.75**	**13.54**
	Rotary	**20.44**	**19.25**	**19.08**	**20.42**	**20.39**	**20.22**	**20.28**	**20.39**	**20.34**
	Laminated	**31.54**	**32.31**	**33.34**	**32.31**	**32.66**	**32.44**	**34.16**	**34.13**	**32.50**
Surface (mm^2)	**Wire-cut**	**1559.6**	**1552.9**	**1328.8**	**1563.9**	**1492.1**	**1519.9**	**1556.2**	**1575.6**	**1517.0**
	Rotary	**3035.3**	**2996.4**	**2981.5**	**2959.4**	**2945.7**	**2889.4**	**2925.9**	**2890.8**	**2885.8**
	Laminated	**4759.3**	**4383.8**	**4314.2**	**4615.6**	**4718.5**	**4640.8**	**4574.9**	**4548.7**	**4651.9**
Density (g/cc)	**Wire-cut**	**0.468**	**0.488**	**0.524**	**0.476**	**0.503**	**0.499**	**0.500**	**0.486**	**0.501**
	Rotary	**0.458**	**0.486**	**0.513**	**0.465**	**0.480**	**0.468**	**0.480**	**0.465**	**0.467**
	Laminated	**0.313**	**0.334**	**0.348**	**0.318**	**0.322**	**0.306**	**0.307**	**0.304**	**0.311**
Firmness (N)	**Wire-cut**	**38.3**	**41.6**	**48.5**	**38.3**	**36.9**	**40.2**	**40.6**	**39.1**	**41.1**
	Rotary	**17.7**	**19.3**	**19.5**	**17.0**	**18.9**	**17.4**	**17.8**	**15.3**	**17.6**
	Laminated	**13.6**	**16.2**	**17.9**	**14.9**	**12.7**	**12.2**	**16.1**	**12.5**	**13.1**

Table 2.6: Relationship between physico-chemical data of the flour and the cookie attributes for the laminated recipe.

Response Variables	Variables	Intercept	Slope	P- value	R^2
Dough consistency	Granul 20	-67.06	4.52	0.00258	0.7491
	Granul 38	146.40	2.72	0.00183	0.7716
	Granul 75	186.63	3.98	0.00092	0.8116
	Protein	-8.63	28.63	0.00130	0.8024
	Ins glutenin	-93.35	247.18	0.00188	0.7897
	Ash	163.77	155.77	0.00743	0.6644
	Alveog P	114.72	3.94	0.02699	0.5261
	Fi	122.58	0.69	0.01315	0.6085
	k	321.86	-102.28	0.00022	0.8736
	n	-90.51	1459.89	0.00049	0.8413
Cookie Density	Granul 20	0.20	0.01	0.04534	0.4578
	Protein	0.23	0.01	0.04527	0.4580
	n	0.20	0.48	0.04784	0.4503
Cookie Surface	Granul 20	2194.86	-9.46	0.02024	0.5608
	Granul 38	5025.58	-10.90	0.02265	0.5475
	Granul 75	4865.43	-16.00	0.01705	0.5803
	Protein	5675.18	-117.77	0.01529	0.5924
	Ins glutenin	5927.41	-949.10	0.03283	0.5013
	Ash	4969.37	-645.18	0.03177	0.5055
	Fi	5143.45	-2.87	0.04206	0.4682
	k	4316.34	419.52	0.00875	0.6492
	n	6007.88	-5988.47	0.01117	0.6253
Cookie Firmness	Granul 75	1098.93	20.27	0.03951	0.4767
	Protein	-42.68	161.64	0.01855	0.5709
	Ins glutenin	-408.69	1316.6	0.03484	0.4935
	Ash	908.57	914.39	0.0848	0.5194
	Fi	652.19	4.12	0.03503	0.4927
	k	1810.34	-556.59	0.01642	0.5846
	n	-487.65	8170.37	0.01486	0.5955
Cookie Thickness	Granul 38	6.21	0.02	0.03285	0.5012
	Granul 75 *	6.50	0.03	0.02956	0.5147

Table 2.7: Relationship between physico-chemical data of the flour and the cookie attributes for the rotary recipe.

Response Variables	Variables	Intercept	Slope	P- value	R^2
Cookie Density	Granul 20	0.28380	0.00267	0.00019	0.8784
	Granul 38	0.42666	0.00119	0.03320	0.4997
	Granul 75	0.44323	0.00180	0.01999	0.5622
	Protein	0.32253	0.01643	0.00018	0.8796
	Ins Glutenin	0.28231	0.13598	0.00148	0.7846
	Ash	0.41745	0.09594	0.00038	0.8524
	AWRC	0.16536	0.00477	0.02606	0.5305
	Alveog P	0.38042	0.00262	0.00154	0.7822
	Alveog P/L	0.43884	0.09300	0.01683	0.5817
	Fi	0.38942	0.00044	0.00063	0.8302
	k	0.50951	-0.05431	0.00064	0.8295
	n	0.28310	0.80637	0.00028	0.8646
Dough consistency	Granul 20	-1475.92	43.58	0.00691	0.6709
	Protein	-641.49	247.09	0.01899	0.5681
	Ins glutenin	-1215.86	2023.14	0.03412	0.4962
	Ash	628.04	1702.70	0.00196	0.7671
	AWRC	-5954.76	117.10	0.0006	0.9129
	Alveog G	4934.31	-146.75	0.00327	0.7319
	Alveog P	-211.45	51.42	0.00028	0.8641
	Alveog P/L	793.25	2193.61	0.00003	0.9248
	Fi	66.77	8.09	0.00093	0.8108
	n	-1073.72	11450.9	0.03363	0.4981

Table 2.8: Relationship between physico-chemical data of the flour and the cookie attributes for the wire-cut recipe

Response Variables	Variables	Intercept	Slope	P- value	R^2
Dough Consistency	Granul 20	-43.802	3.49	0.00335	0.7500
	Protein	13.180	20.86	0.00580	0.6862
	Ins.gluten	-56.088	185.41	0.00455	0.7064
	Ash	128.186	130.79	0.00196	0.7671
	AWRC	-261.326	7.21	0.01615	0.5863
	Alveog. P	73.043	3.69	0.00234	0.7555
	Alveog. P/L	151.324	1425.0	0.00804	0.6571
	Fi	88.619	0.60	0.00204	0.7645
	k	249.743	-67.73	0.01121	0.6249
	n	-32.661	1005.8	0.00855	0.6514
Cookie Density	Granul 20	0.324	0.00	0.00832	0.6539
	Protein	0.359	0.01	0.00836	0.6535
	Ins.glutenin	0.313	0.13	0.00802	0.6574
	Ash	0.442	0.09	0.00733	0.6655
	AWRC	0.182	.00	0.03110	0.5082
	Alveog P	0.406	0.00	0.00879	0.6488
	Alveog P/l	0.458	0.09	0.02332	0.5440
	Fi	0.415	0.00	0.00746	0.6640
	k	0.524	-0.05	0.02077	0.5577
	n	0.329	0.70	0.01342	0.6063
Cookie Volume	AWRC	25.75	-0.18	0.0121	0.6172
	Alveog P	16.54	-0.08	0.0311	0.5081
	Alveog P/L	15.11	-3.30	0.0178	0.5756
	Alveog G	8.73	0.23	0.0378	04826
	Fi	16.10	-0.01	0.0435	0.4635
Cookie surface	Protein	2012.89	-53.57	0.0405	0.4734
	Granul 20	2194.86	-9.46	0.0202	0.5608
	Ash	1740.32	-373.5	0.0083	0.6544
	AWRC	3255.24	-26.78	0.0004	0.8463
	Alveog G	746.90	34.38	0.0042	0.7121
	Alveog P	1930.37	-11.44	0.0022	0.7586
	Alveog P/L	1710.73	-498.1	0.0005	0.8452
	Fi	1865.93	-1.79	0.0048	0.7016

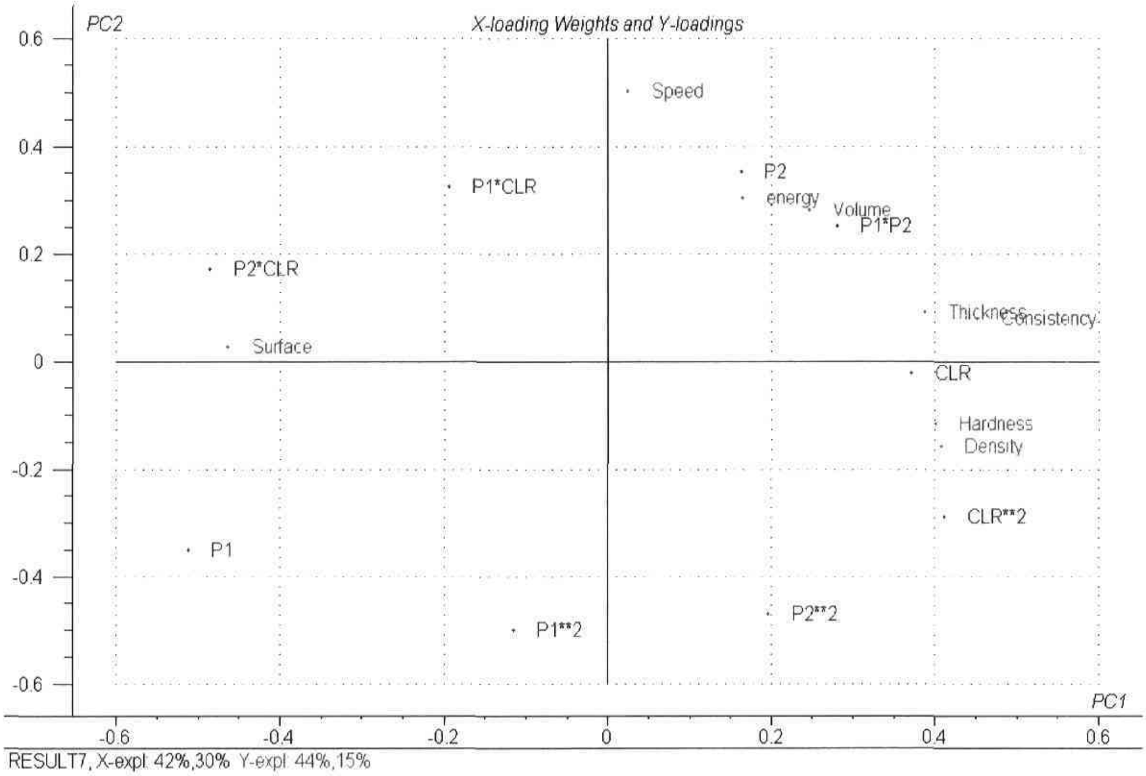

Figure 2.1: Loading weights of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) and loadings of Response variables (Surface, Thickness, Volume, Density, Firmness, and Consistency) for the first 2 components in the calculated PLS quadratic model for the laminated recipe

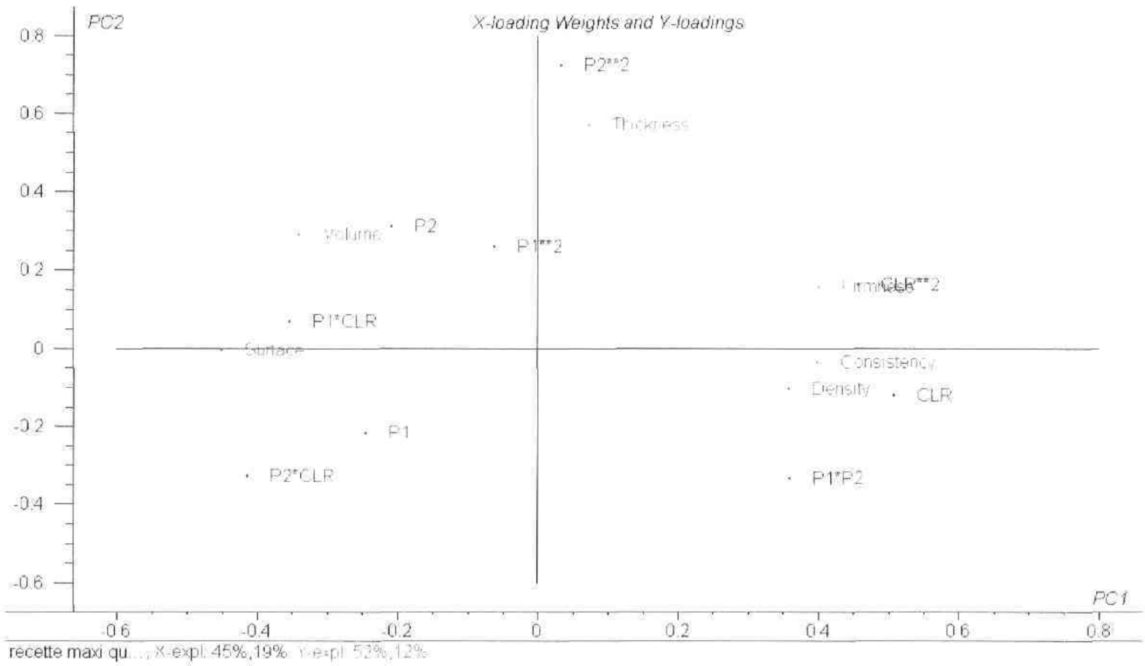

Figure 2.2: Loading weights of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) and loadings of Response variables (Surface, Thickness, Volume, Density, Firmness, and Consistency) for the first 2 components in the calculated PLS quadratic model for the rotary recipe data

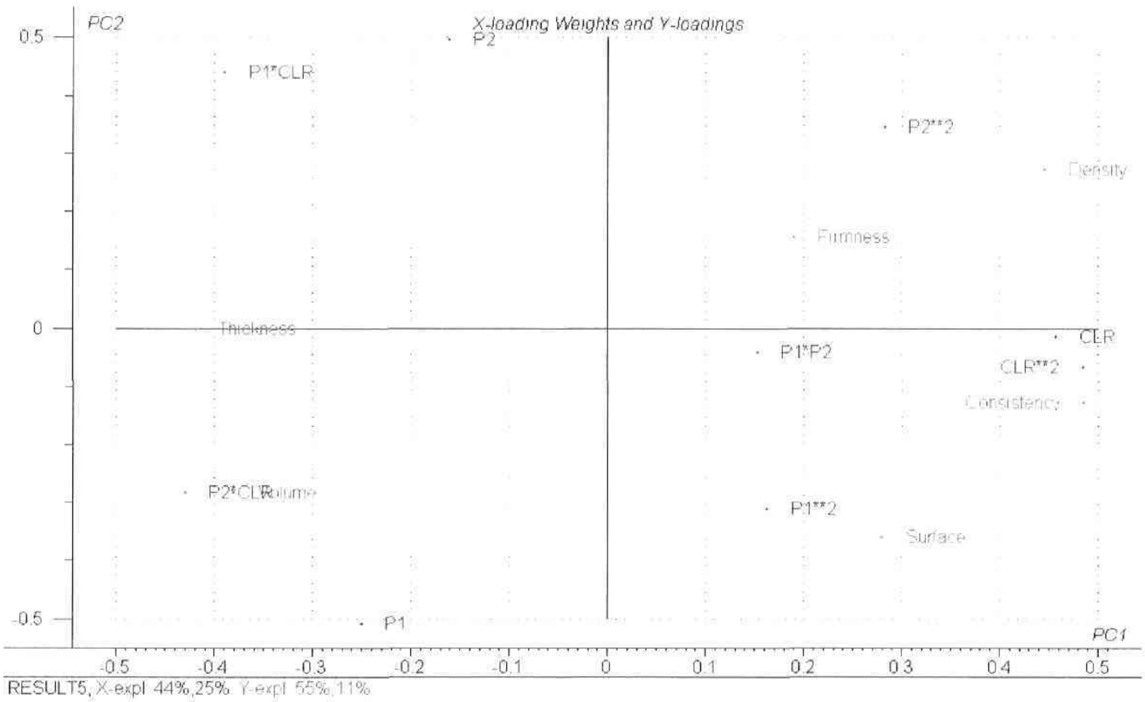

Figure 2.3: Loading weights of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) and loadings of Response variables (Surface, Thickness, Volume, Density, Firmness, and Consistency) for the wire-cut recipe.

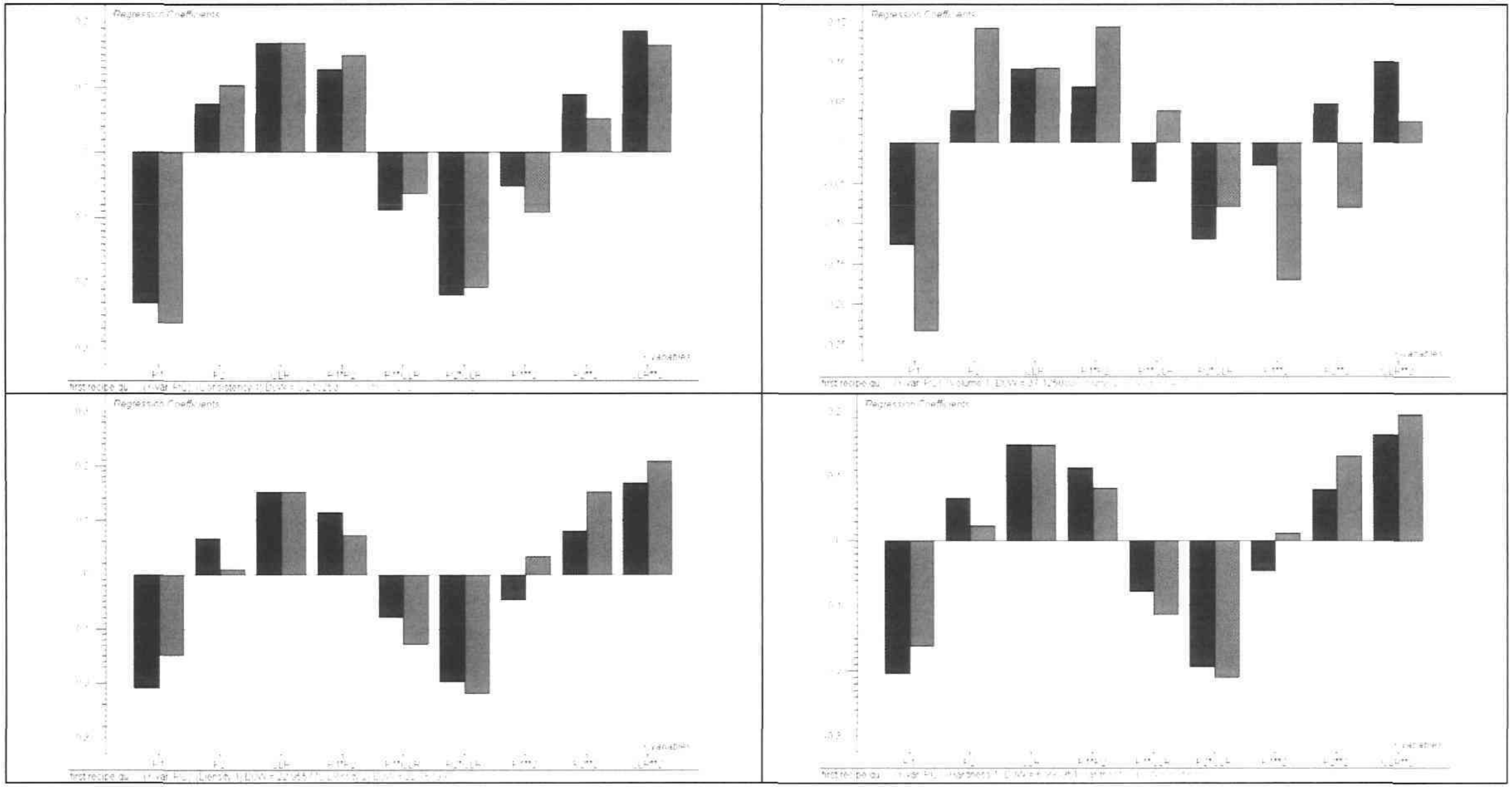

Figure 2.4: Regression coefficients of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) for Response variables for the first 2 components in the calculated PLS quadratic model for the laminated recipe

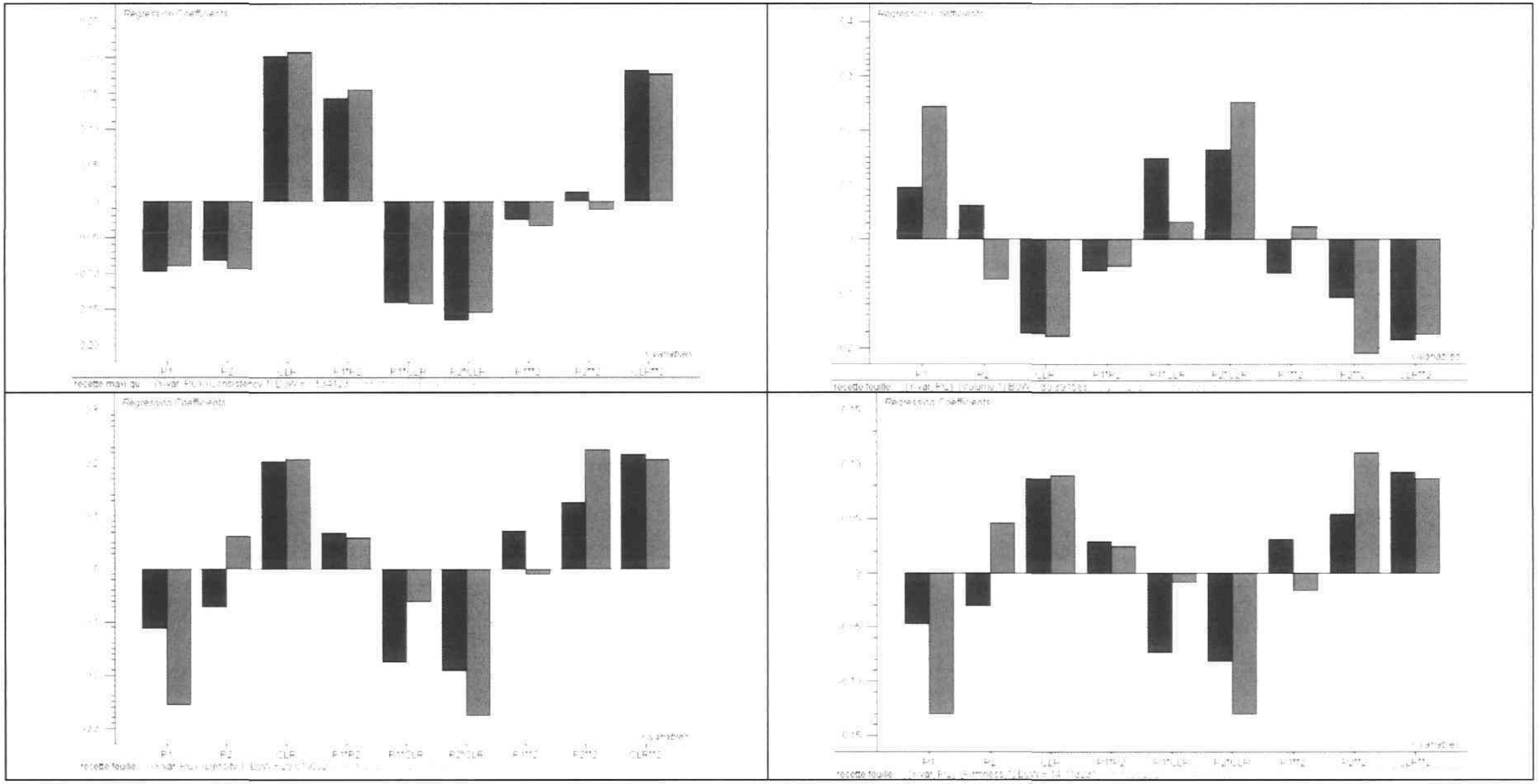

Figure 2.5: Regression coefficients of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) for Response variables for the first 2 components in the calculated PLS quadratic model for the rotary recipe

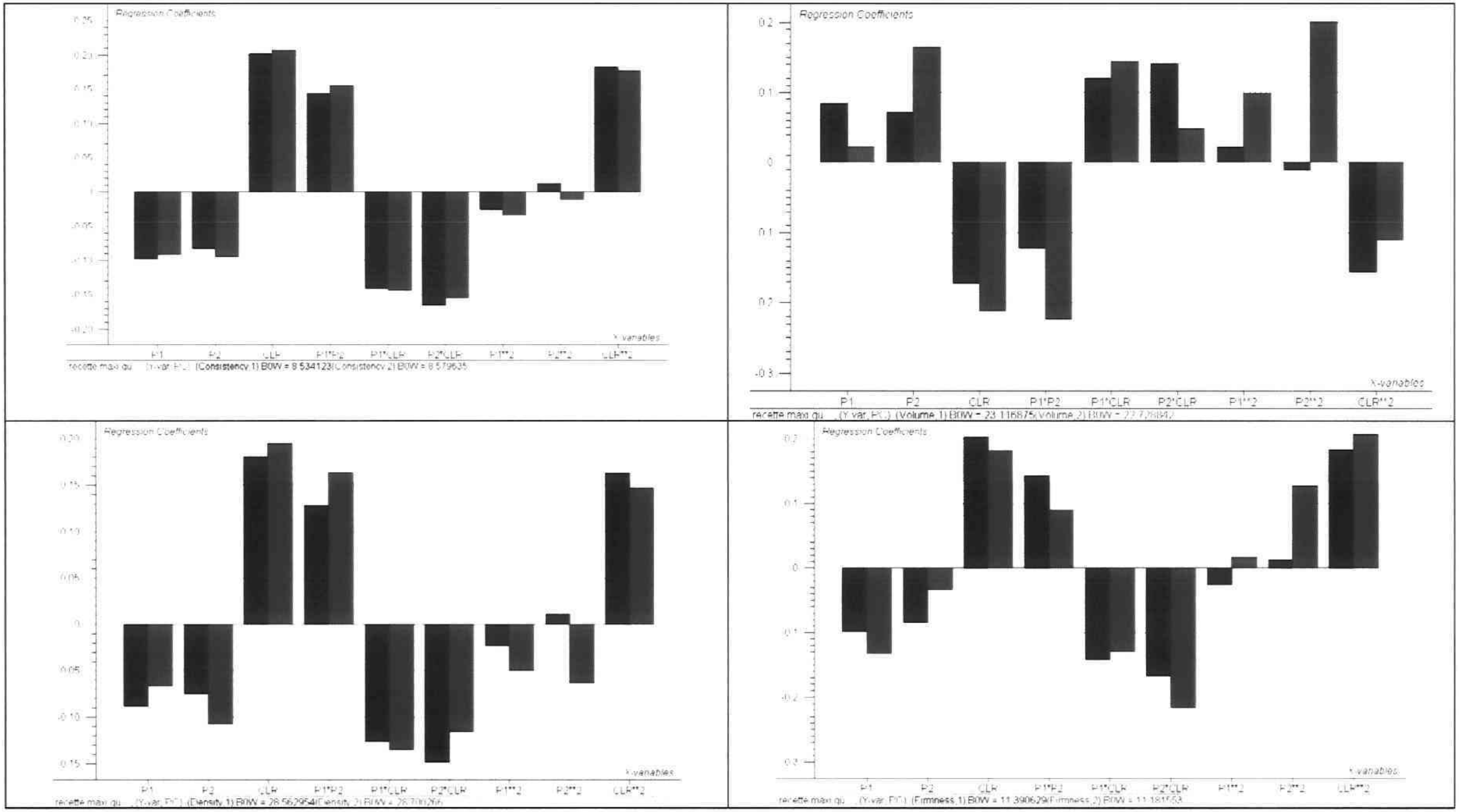

Figure 2.6: Regression coefficients of Predictors (P1, P2 and CLR, interactions P1*P2, P1*CLR, P2*CLR and square effects P1**2, P2**2, CLR**2) for Response variables for the first 2 components in the calculated PLS quadratic model for the wire-cut recipe

« *La plus belle chose que nous pouvons éprouver, c'est le mystère des choses*»

Albert Einstein (1879-1955)

CHAPTER III

Semi-sweet biscuit making potential of soft wheat flour patent, middle-cut and clear mill streams made with native and reconstituted flours.

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1-Résumé

L'influence de trois grades de farines soit les farines patente, intermédiaire et basse, à l'état d'origine ou reconstitué, sur les caractéristiques rhéologiques de la pâte à biscuits semi-sucrés et sur la qualité des produits finis (rapport farine/sucre/gras/eau de 100/30/8/36) a été étudiée. L'utilisation de la portion périphérique (farine basse) par rapport à la portion centrale (patente) de l'endosperme du grain de blé a entraîné une augmentation des attributs dureté (de 3,77 à 4,84 N), consistance (de 19,3 à 25,5 N.s), viscosité élongationnelle (de $4,13*10^{-5}$ à $5,54*10^{-5}$ Pa), temps de demi-relaxation (de 0,45 à 0,59 s) de la pâte à biscuits; par contre, la vitesse de relaxation a diminué de 4,51 à 3,09 s^{-1} pour la pâte produite avec les farines à l'état d'origine, ce qui s'explique par la grande variation des propriétés physico-chimiques de ces fractions. Sur le plan quantitatif, le processus de fractionnement/reconstitution a modérément abaissé ces paramètres rhéologiques, la fonctionnalité de la farine ne pouvant être totalement restaurée. La farine patente a donné des biscuits avec des longueurs supérieures et épaisseurs moindres, tandis que la farine basse a donné des biscuits plus denses à structure interne d'une plus grande cohésion (force de déchirement), avec plus de grains ou de groupes de grains par unité de pénétration (nombre de ruptures spatiales). Les biscuits produits avec les fractions de farine reconstituées présentaient des caractéristiques dimensionnelles pratiquement équivalentes et une surface d'excellente apparence, mais ils étaient plus foncés que ceux produits avec la farine native correspondante; la teneur en eau est demeurée relativement inchangée.

Le temps de demi-relaxation (Tla) et la vitesse de relaxation (k), déterminés d'après la courbe de relaxation des pâtes à biscuits, sont d'excellents prédicteurs de la qualité des biscuits (longueur, densité, cohésion structurale et résistance à la sollicitation). Nous avons constaté que la dureté de la pâte a une corrélation positive avec la viscosité élongationnelle, le temps de demi-relaxation et la consistance, et une corrélation négative avec la vitesse de relaxation; par ailleurs, certaines des caractéristiques des biscuits, comme la densité, étaient en corrélation positive avec la cohésion structurale et la résistance à la sollicitation.

Abstract

The influence of patent, middle-cut and clear flour grades as native or reconstituted flour blends on both the rheological properties of the dough and the quality of semi-sweet biscuit (flour/sugar/fat/water ratio of 100/30/8/36) was studied. Moving from the central portion (patent) to the peripheral portion (clear) of the grain endosperm increased the dough hardness from 3.77 to 4.84 N, consistency from 19.3 to 25.5 N.s, elongational viscosity from $4.13*10^{-5}$ to $5.54*10^{-5}$ Pa.s, half relaxation time from 0.45 to 0.59 s, but decreased the rate of relaxation from 4.51 to 3.09 s^{-1} of the biscuit's dough produced with the native flours due to the wide variation in the physico-chemical properties of these fractions. Quantitatively, the fractionation/reconstitution procedure reduced moderately these rheological parameters, and the flour functionality could not be restored completely. Biscuits produced with the patent flour showed the largest length and lowest thickness, whilst the clear fraction led to production of denser biscuits with greater cohesion (mean tearing force) of the biscuit inner structure and also contain more grains or group of grains per unit of penetration (number of spatial ruptures). The biscuits made with the reconstituted flour fractions had almost equivalent dimensional characteristics, and excellent surface appearance, but were also darker in colour than their native flour counterparts.

The half relaxation time (Tla) and the rate of relaxation (*k*) obtained from the biscuit's dough relaxation curves were excellent predictors of the biscuits quality (length, density, structural cohesion and resistance to solicitation). Dough hardness was correlated positively with elongational viscosity, half-relaxation time and consistency and negatively with the rate of relaxation, whilst some of the biscuits characteristics such as density correlated positively with structural cohesion and resistance to solicitation.

3. Introduction

Quality of commercial soft wheat flour depends mainly on the quality of the wheat cultivars selected and the flow sheet followed during the milling operation. Non-uniform distribution of components within the wheat kernel gives rise to variation in composition and functional properties of different flour streams (break, sizing and reduction) obtained by roller flour milling (Morrisson and Hargin, 1981; Morrisson and Barnes, 1983; Gaines et al., 1996; Prablansankar et al., 2000; Berton et al., 2001, 2002). The stream-splitting method, where a group of similar and complementary streams are blended for particular end-use, is gaining popularity to produce flours with specific properties where a particular quality of wheat is not available (Dubé et al., 1987). These streams are recombined during the milling operation to yield the patent, the middle-cut and the clear flour grades. Mixed together in an appropriate ratio, these mill fractions constitute the straight grade flour (Hoseney, 1994). Although the biochemical and physical characteristics of the various breaks, sizing and reduction streams are well known, information of their influence on the rheological behaviour and baking performance for semi-sweet biscuit making are rather scarce and fragmentary.

A good quality soft wheat flour for cookies making should yield cookies of large spread with uniform surface cracking pattern. These properties can be controlled by the adjustment of the level of sugar, water, emulsifier, fat (Abboud et al., 1985; Doesher et al., 1987; Doesher and Hoseney, 1987; Manohar and Rao, 1997, 1999 a, b, c; Maache Rezzoug et al., 1998) or by appropriate selection of quality soft wheat flour, the key functional ingredient in biscuit manufacture.

Due to the multiphasic nature of dough, some flour constituents can be separated either by physical means via centrifugation (Czuchajowka and Pomeranz 1995; Larsson and Eliasson, 1996; Tolstoguzov, 1997, 2002; Van Der Borght et al.,2005) or by the use of solvents to extract specific sub-fractions (Finney et al.,1976; MacRitchie, 1985; Graβberger et al., 2003). Flour fractionation has been used extensively to determine the contribution of individual flour fractions such as lipids, gluten, starch tailings, prime starch and water-solubles essentially on sugar-snap cookie diameter (Yamazaki 1955; Sollars, 1956; Cole et al., 1960; Sollars and Bowie, 1966; Pomeranz, 1977; Clement and Donelson, 1981;

MacRitchie, 1985; Donelson, 1988 and 1990; Miller and Hoseney, 1997a). The reconstituted flour is often intended to serve as standard flour for the investigation of the functional role of multiple additives (improvers, enzymes) or constituents (lipids, protein, pentosans). Most of these flour constituents are reported to physically or chemically influence the flour quality. Such effects have been examined mainly with the standard AACC 10-52 sugar-snap cookie formula (flour: sugar: water: fat ratio of 100: 60: 15: 30 on 14% moisture basis). However, such studies have not established a strong relationship between flour properties and cookie diameter, probably due to the high content of sugar and fat coupled with a low hydration that could mask or even alter the flour functionality (Slade and Levine, 1994). Numerous authors reported the fractionation and reconstitution of hard wheat flours in bread making (Köhler et al., 1999; Graβberger et al., 2003; Uthayakumaran and Lukow, 2003), but much less is known about the baking performance of a semi-sweet biscuit made with soft wheat flour reconstituted with isolated fractions of gluten, starch, water-solubles and other constituents extracted and re-incorporated in the same or different proportions in which they appear in the native flour. Understanding the role of these fractions and their endogenous constituents implies that the reconstituted flour should retain its original properties when the fractions are added in the same proportions as in the parent flours. A semi sweet biscuit recipe is usually richer in flour, lower in fat and sugar and higher in water (flour: sugar: water: fat ratio of 100: 30: 26: 8 on 14% moisture basis) than the sugar snap cookie. Thus, the dough properties might be more sensitive to the gluten development. Additionally, the end-product quality attributes sought for semi-sweet biscuits differ significantly from the sugar-snap cookies, e.g., lower retraction of the dough after sheeting, lower density and appropriate friability, while for cookie the spread and hardness (the snap) are the key quality attributes.

Therefore the present study was undertaken to investigate the biscuit making potential of soft wheat patent, middle-cut and clear mill fractions with dough systems made with both native and reconstituted flours by focusing on dough rheological behavior and baking performance with a semi-sweet biscuit recipe.

4- Materials and methods

4.1- Mill stream fractions

The wheat flour fractions studied were obtained from a blend of cultivars consisting of 60% soft red winter (CFSR) and 40% soft white winter (CEWW) taken from the 2000 crop harvest, grown in Ontario. The unbleached and untreated flour streams were collected from a commercial flour mill (Robin Hood, Port Colborne, Ont, Can). The stream fractions were referred to as the patent (first 45% w/w of the material obtained by a 72% extraction rate), middle-cut (middle 20% w/w) and clear flour (tail end 7% w/w of the material). The combination of these three flour streams obtained from various break and reduction rolls constitutes the straight grade flour intended for cookie making. All samples were stored in the dark at -18°C and defrosted for 12 h at 20° C before use.

4.2- Physico-chemical analyses

Moisture contents of flour grades and flour fractions (gluten, starch and water soluble) were determined according to the AACC method 44-15A (2000). Protein content was measured by the LECO nitrogen/protein determinator PF-428 (LECO Instruments Ltd., Mississauga, Ontario, and Can) and nitrogen content was multiplied by 5.7. Flour starch damage was determined enzymatically according to the method AACC 76-30A and ash content by the method AACC 08-01(2000).

The AACC 54-30 method for the Chopin alveograph was used to monitor the W, deformation energy ($W*10^{-4}J$), P, maximum pressure (mm) and L, average abscissa at rupture (mm). Four solvents were independently used to produce the water Solvent Retention Capacity (SRC), 50% sucrose SRC, 5% sodium bicarbonate SRC and 5% lactic acid SRC. The weight of solvents held by the control and reconstituted flours after centrifugation were expressed as percent of flour weight (14% m.b) according to the AACC approved method 56-11 (2000). Total pentosans were determined according to the method of Rouau and Surget (1994). Lipids determination of the native flours and reconstituted flours made from isolated fractions of starch, gluten and water-solubles, added in the same proportions as in the original flours, was carried out by an acid hydrolysis method AOAC 922.06 (2000).

4.3- Fractionation of flour

The study of MacRitchie (1985) was used as a basis for the procedure. However, slight modifications of the extraction procedure were adopted for our study to avoid solvent defattening of the lipid from the flour grade samples with dichloromethane. Flour (210g) and distilled water (100ml) were mixed into dough for 60s at low speed in a dough mixer of 800g capacity (National mfg, Co., Lincoln, N.E. USA). Distilled water (500ml-aliquots) at 15^{o}C was added to the dough to minimize the endogenous proteolytic activity and the dough was massaged manually to wash the starch from the gluten (approximately 5min/wash). Six (6) successive and independent washes were necessary so that the final rinse water was clear. The rinse waters were bulked, sieved through a 355 μm screen and centrifuged at 5000 x g for 10 min (rotor JA-10; model 12-21, Beckman, Spinco Division, Palo Alto, California) to separate the water soluble fraction (supernatant) also referred as the water-solubles from the starch (sediment). After centrifugation, the upper brown layer (starch tailings) was also carefully separated physically with a spatula from the white layer of prime starch for the recovery determination. In a separate experiment, the non-starch lipids were extracted with dichloromethane (210g of flour and 420g of solvent) for 30 min and the flour was recovered by filtration on a Whatman no.1. This step was repeated three times and finally the oil solvent-free was immediately incorporated into the defatted flour fractions previously recovered to avoid hydrolytic and oxidative changes to assess the effect of free lipids on biscuit dough rheology and end-product quality. The dry gluten fraction was cut into cubes of about 1 cm^3. Gluten, starch and water soluble fractions were then rapidly placed into large aluminium trays (5mm depth maximum), frozen at -40^{o}C in a freezer . Then the samples were freeze dried in a Lyo-Tech Freeze drier (Lyo-San Inc, Lachute, Qc, Canada) with heating temperature adjusted electronically at 18 ^{0}C for 48 hr and at 200 mtorr of vacuum. After drying, the starch and gluten fractions were ground in a centrifugal grinding mill (Brinkmann Netsch L-49) and finally passed through a 250 μm

(70 mesh screen). The fractions were then hermetically stored in double aluminium foil placed into plastic jars and stored at 4°C prior to use (within 2 weeks). Gluten, starch and water soluble fractions were weighed, and protein and moisture determinations were made on all samples for recovery determination.

For preparation of the reconstituted flours, the ground starch and gluten were tempered in a humidifying cabinet to about 12% moisture (85% RH at 30°C for ~1 hour). Then the respective fractions (starch, gluten and water-solubles) were mixed in the appropriate concentration to match the native flours on dry weight basis (d.b). The final water content of the dough was adjusted at 24%, according to the fractions moisture contents.

4.4- Biscuit baking

The baking test was developed by CTUC (Centre Technique des Utilisateurs des Céréales) to decide if a wheat variety could be registered as suitable for biscuit making (Tharrault, 1994). This recipe contains higher level of flour, so the quality of end-product is more dependent on flour quality compared with the sugar snap recipe. Dough was prepared with 200g of flour (100, moist basis), sugar (30% of flour mass), shortening (8 %), ammonium bicarbonate (1.0 %), salt (0.63 %), sodium pyrophosphate (0.5 %) and distilled water, to get a constant water content (24 % dough basis). Ammonium bicarbonate is dissolved in distilled water and the sugar was weighed with the remaining sieved flour. The dry ingredients were all introduced into the Farinograph resistograph dough mixer (CW. Brabender Instruments Inc, Hackensack, USA) coupled with a thermostated bath (Fisher Scientific Isotemp 3016) to maintain the temperature at 27°C and mixed for 30 s. at 47.5 rpm. The shortening was then deposited at the surface of the dry ingredients and mixed for 30 s, prior to the addition of the distilled water containing ammonium bicarbonate. The dough was mixed for 13 min. Biscuit dough was then laid out in an hermetically closed bag placed for 30 min in a water bath adjusted at 27 °C. Thereafter, the dough was sheeted via several reduction steps between 2 cylinders of a sheeter (model SM-250M from Picard Bakers LP, Victoriaville, Qc, Canada) for several reduction steps with the 15mm (1 passage), 7.5mm (1 passage), 4.5mm (1 passage) and 2.5mm (2 passages with folding of the dough between each passage through the sheeter) and 1.75mm (similar conditions as at 2.5mm) gap adjustment, successively. For the last reduction step at 1.75 mm, the dough was rested for 1 min between the two passages. The dough was then punched out with a square cutter of 60 x 60 mm and immediately baked at 280°C in an oven (serial No.4845, Picard bakers LP, Victoriaville, Qc, Canada) equipped with a balance. Baking (8.5 to 10 minutes for 10 to 12 doughs) was stopped when a weight loss of 20% was attained (approximately 4.5-5.5 min). The biscuits were then cooled down for at least 30 min on the baking tray, and finally packed into aluminium foiled pouches.

4.5- Rheological properties of the dough

Dough characteristics were evaluated by the texture profile analysis (TPA) method (Stable Micro Systems, Surrey, UK, 25 kg load cell). Cylindrical dough disks, 25mm diameter, and 20 mm thick were prepared with a circular shape cutter. The parameters for the TPA procedures were as follows: 10 cm diameter stainless steel plate, cross-head speed (Vz= 0.8mm/sec), compression of 40% of the original thickness, recovery period between the two strokes 5s. The real time plots of the doughs were analyzed for the following: 1) consistency (Ns), the combined area of the two resistance peaks (A1 and A2) ; 2) hardness (N), the peak force of the first compression peak of the product; 3) cohesiveness , the ratio of the area of the two resistance peaks (A2/A1); 4) springiness (distance of the detected height of the product of the second compression-original compression distance or L2/L1peak. The average from 6 pieces of dough was recorded on three dough preparations (c.v <11.5%).

Following the double compression test, additional dough disks were prepared from the dough rested for approximately 35 to 40 min into the hermetically closed bag. The disks were lubricated with paraffin oil (viscosity of 30 mPa.s) to prevent drying of the dough and adhesion to the probe for the lubricated squeezing flow test (LSF). The same operating conditions as for the double compression were adopted and the disk thickness was reduced by 40% of its initial value in a 25 s period. After stopping the cross-head, the decay of the compression (force) was followed during 180 s (25 data points /s.). The force at the start of the relaxation was denoted as Fmax, equivalent to dough hardness. The half relaxation time, denoted as Tla was measured as the time required for the force to decrease to a value Fmax/2 (Bartolucci and Launay (2000)

Wheat flour dough behaves in biaxial extension as a viscoelastic liquid and its behaviour is not linear: rheological parameters are strain and strain rate dependent. The equation proposed by Bartolucci and Launay (2000) that is based on a non-linear Maxwell Model was adopted. The relaxation curve after lubricated squeezing flow is shown in Fig 1 and the equation 1 can be written:

$$\sigma(t)=\sigma_i\left[1+k\left(\frac{1}{n}-1\right)t\right]^{\frac{n}{n-1}}$$

Where σ is the stress (Pa) and $\sigma = \sigma_I$ at $t = o$; k (s^{-1}) corresponds to the constant of relaxation speed and n is the flow behaviour index (dimensionless). This equation was used to fit (software Table curve 2D, Jandel) stress relaxation data obtained with the lubricated squeezing flow method.

Elongational viscosity of the dough was also monitored with the LSF compression method under the same conditions and was calculated as 2 Fh/R^2V_z, where F is the peak force recorded for dough deformation in the LSF test, h is the dough height after compression, R is the radius after compression and V_z is the cross-arm speed (Miller and Hoseney, 1997b).

4.6- Biscuit evaluation

4.6.1- Dimensions

Biscuits (length, width) were evaluated using a Vernier calliper on a set of 12 biscuits, after 2 weeks storage in aluminium pouches stored at 20° C. Cookie volumes were measured with a volumeter filled with rapeseed and density (g/cc) was calculated directly from the weight and volume data obtained by averaging values of 15 biscuits measurements. Thickness test was performed by placing the biscuits on top of each other in a stack unit measuring the thickness and restacking the 5 biscuits 3 times.

4.6.2- Water activity (Aw)

Biscuits were ground with a mortar and pestle and Aw were determined using a Water Activity meter CX2, Aqualab.Inc (Decagon Devices, Pullman, Wa) on 5 biscuits samples stored for15 days in aluminium pouches. The dough Aw was determined on the 30 min rested dough.

4.6.3- Moisture

Biscuit moistures were determined according to the AOAC 31.005 (1970), on five ground biscuits samples for each recipe.

4.6.4- Colour

The values of surface colour of biscuits in terms of brightness (L), redness (a) and yellowness (b) were measured using the Hunterlab (Labscan spectrophotometer V1-A30, Hunter Associates Lab Inc, Reston, VA) calibrated with the black glass and white tile (Illuminant D65, CIE 10 ^{0} observer, Ls 13593). The a scale varies from green (negative) to red scale (positive) and the b scale corresponds to a yellow-blue scale on which yellow is positive. The colour data correspond to averages of 5 biscuits surface colour measurements.

4.6.5- Mechanical characteristics

Biscuits stored 15 days in aluminium pouches at ambient temperature were punctured using the TAXT2 Texture Analyzer fitted with a 10 ^{0} stainless steel cone. The speed of penetration was 0.6mm.s^{-1}. All the biscuits were punctured for a distance of 6 mm and a minimum of 12 measurements were carried out for each sample. The acquired data were analyzed based on the peak analysis of the change in the force versus time or displacement curves as described elsewhere (Goullieux et al., 1995; Maache Rezzoug et al., 1998).

Work (S) was evaluated by multiplying the cross head speed (0.6mm/s) by the area of the force vs time curve presented in Fig 3.2, or directly from the area under the curves between 0 and 6 mm distance (Nm). The drop of force (ΔF), characterizing each peak and representing the resistance to the detachment of the grains or group of grains, and the number of peaks (N) during the displacement y, representative of the number of grains or group of grains detached were evaluated.

The parameters derived from these tests were the overall mean penetration force, Sm = S/y (N), number of spatial ruptures (the number of grains or group of grains per unit of penetration distance), No = N/y (mm^{-1}), the average drop-off (mean tearing force) Fs = Σ (ΔF)/N (N) and friability work Fs/No (N.mm).

4.7-Statistical analysis of data

The experimental design was a completely randomized design and the difference among treatments was tested using the Duncan multiple comparison test (Steele and Torrie, 1980). Analysis of variance using PROC GLM of the SAS system (version 8.01, SAS Institute, Cary, NC) was performed to determine the significant difference between flours. The Pearson's correlation test was also performed using PROC Corr of the SAS system to determine the significant linear correlations between quantitative continuous variables related to end-quality parameters and the rheological properties of the dough. All correlations were performed at a 5% significance level. Data reported are the average of three replications.

5. Results and Discussion

5.1. Physico-chemical characteristics of the flour streams and fractions

Table 3.1 shows the composition and physico-chemical properties of the patent, middle-cut and clear flour fractions. Protein content increased significantly from the patent, middle-cut and clear fractions from 7.41, 9.65 and 12.7% g/100g basis respectively, due to variation of the cytoplasmic origin of the products derived from distinctive soft wheat flour sets. This evolution of protein content could be attributed to increasing concentration of peripheral endosperm portion, which is reported to be rich in protein, lipids and ash (Prablasankar et al., 2000). Higher ash content, e.g. 0.99% in the clear fraction, could be due to the extraction of the aleurone layer adhering to the bran as it contains higher mineral levels. These results further confirm that the flours obtained at the beginning of the milling (patent) come mainly from the central portion of endosperm, which is rich in starch and low in protein, whereas at the end of grinding, the material is taken essentially from the periphery of the kernel (clear) which is rich in ash, pentosan and protein as reported by Berton et al. (2001) and Prablasankar et al. (2000). The cylinders moving closer and closer in the grain crushing operation also cause breaking the starch granules, thus the damaged starch content increased slightly from 3.6 to 4.9% from the patent to clear fractions, respectively.

The alveograph P values showed a significant increase in the dough resistance to deformation related to the elastic resistance of the clear flour with a concomitant diminution of the L value or extensibility parameter, so the «P/l» ratio almost doubled from 0.34 to 0.67 from the patent to clear fractions, an indication that the clear fraction produces a more elastic dough. The clear fraction also had higher water sorption capacity as reflected by a higher AWRC 74.9 versus 63.4% for the patent, due to higher damaged starch, pentosan and protein of this flour material. Higher AWRC has been reported to exert a negative impact on sugar-snap cookie spread (Nemeth et al., 1994; Labuschagne et al., 1996).

5.2. Recovery of the fractions

The results (not shown) indicated minor differences in the rheological properties of the dough and semi-sweet biscuit characteristics (i.e. dimensions and surface appearance) between the lipid-extracted and non-extracted flour samples, once the lipid fraction was re-incorporated into the flour fractions at the same proportion as in the native flours, confirming the observations of Sollars and Bowie (1966) who reported a small influence of the lipid fraction on cookie diameter when fully re-incorporated into the flour. Thus, the lipid extraction procedure with dichloromethane was subsequently omitted for this study. The composition data of Table 3.2 confirmed a progressive increase in gluten recovered from the patent to clear fraction breaks from 7.97, 11.3 and 15.6% due to the high protein content of the subaleurone endosperm portion which is not easily reduced during roller milling as the inner endosperm. As a result, increasing concentration of the peripheral endosperm portion, rich in protein, lipids and pentosans is noted as reported by Morrisson and Hargin 1981, Pomeranz (1988) and Ranhottra et al. (1990). In contrast, the crude starch content which consists of both the prime and starch tailing fractions decreased significantly from the patent, middle-cut and clear from 86.7, 82.3 and 75.8%, respectively. The starch tailings, or the top layer obtained by centrifugation of the starch and water solubles fraction, rose substantially from the patent, middle-cut and clear fractions from 6.8, 13.4 and 18.9%, respectively, with a concomitant drop in prime starch of 79.9, 68.9 and 56.9%, respectively. Starch tailings are reported to contain 87-94% starch (mainly small granules, large damaged starch particles, 1-2% protein, 4% pentosans, 0.7% fatty substances, 0.3% ash and 3% of cellulosic material (MacMasters and Hibbert, 194; Miller and Hoseney, 1997). The pentosans or non-starchy polysaccharides originating from the tailing portion are reported to contribute to the higher water binding capacity of this fraction (Sandsted, 1961). The prime starch, on the other hand, consists essentially of large granules and non-damaged starch. The ash concentration also increased from the native patent, middle and clear from 0.41, 0.57 and 0.99% and the lipids from 1.26, 2.07 and 3.23% respectively due to the large quantity of cell wall or non starchy polysaccharides originating from the grain envelop (Prablasankar et al., 2000; Berton et al., 2001). Damaged starch, which exerts a negative effect on cookie diameter (Abboud et al., 1985; Nemeth et al., 1994) varied little, i.e. 3.5 to 4.9% among the three native flours (Table 3.2). The water solubles fraction which contains proteins (essentially albumins and some globulins), starch, pentosans and several compounds of low molecular weight (Sollars 1959; Miller and Hoseney, 1997a) increased from 5.27, 6.43 and 8.59% from the central, middle and peripheral portions of the endosperm, respectively.

With low protein content flours such as those of soft wheat, researchers have reported difficulties in effecting good fraction separation especially with hand kneading (Pomeranz, 1977). During the hand kneading extraction of the three mill fractions, the gluten network structure obtained from the clear flour appeared to break down, as compared with the patent and middle-cut flours; the latter structure being much more cohesive. Moreover, the patent and especially the reconstituted patent tended to be sticky when incorporated into the biscuit recipe.

Table 3.2 shows that the proteins are mainly associated with the gluten fraction, and to a lower extent with the water-solubles fraction. The isolated gluten from the flour streams had approximately the same protein content. Compared to the results of MacRitchie (1985), the recovery of flour protein was higher than the hard wheat flour examined, likely due to the use of soft wheat flour fractions which have a weaker gluten structure. During aqueous fractionation of flour, the bulk of free lipid appeared to be primarily associated with the gluten fraction. This is in agreement with the observations of MacRitchie (1985) and Larsson and Eliasson (1996), since once the flour is brought in contact with the aqueous solution, the lipids can interact with the protein and starch matrix. The higher level of lipids found in the tail-end stream or clear fraction could be due to the extraction of the aleurone layer adhering to the bran that contains higher amount of oil, and also to its contamination with germ fragments. Flour pentosans were essentially associated with the water-solubles fraction and, to a lower extent with the crude starch fraction.

5.3. Solvent retention capacity (SRC)

To assess the sorption properties of the native versus the reconstituted flours, four solvents (water, 5% sodium carbonate, 5% lactic acid and 50% sucrose) were used to establish the fraction's functionality profile related to baking performance based on the weight of solvent held after centrifugation. Each solvent provides information on a different chemical and physical aspect of the sample, thus, helping at identifying the flour components contributing to end-use functionality (Slade and Levine 1994; Gaines, 2000). The solvent retention method (SRC) with sodium carbonate is associated with levels of damage starch; sucrose SRC with pentosan and, to some extent, to the gliadins; lactic acid to the glutenin characteristics and water SRC as an indicator of moisture sorption of all the flour constituents combined. Water hydration capacity of wheat flour components varies widely, e.g. granular starch can absorb between 39 to 87% of water in weight, damaged starch between 200 and 430%, pentosans between 500 and 1500% and proteins between 114 and 215% (Berton et al., 2002). Yamazaki (1955) concluded that any component that would absorb large amounts of water can affect sugar-snap cookie diameter negatively; e.g. starch tailings and pentosans-rich sub-fractions would decrease the cookie diameter. Low sodium carbonate and sucrose SRC values are desirable (Kaldy et al., 1991; Labuschane et al., 1996; Nemeth et al., 1994). Good quality cookie flour should retain water poorly, so more water is available to the sugar added to the recipe to form a syrup. Also, the dough viscosity should decrease during baking, and the dough must spread farther to yield cookies of large diameter. Moreover, flour samples with higher water retention require increased baking times in cookie manufacture and produce a less tender end-product.

When the lyophilized starch, gluten and water-solubles fractions were recombined to the original levels of the native flours, the overall water sorption increased slightly by ~ 6.0 to 7.0% with the three flour materials (Table 3.3) since the lyophilization process of the gluten, starch and water-solubles fractions, once reconstituted to their original levels make them more hydrophilic than their native flours counterparts. Thus, the gluten and starch fractions were both tempered and then recombined with the water-solubles fraction to adjust the water SRC and moisture contents as close as possible to the native flours. Sodium carbonate, sucrose and lactic acid SRC values of the reconstituted flour also increased by ~ 1 to 4% versus the native variants. Higher sodium carbonate SRC values were found for the clear flours, whereas the lactic acid SRC decreased substantially from the patent to the clear fraction from 92.9 to 70.9% for the native, and from 96.5 to 72.5% for the reconstituted flours, respectively. This may imply a pronounced reduction of the glutenin functional properties from the patent to the clear flour, in spite of the larger amount of protein found in the latter, indicating that the proteins from the patent and clear could be of different quality.

5.4. Rheological properties of the biscuit dough

Many aspects of biscuit processing and end-products are closely related to the rheological behaviour of the dough. Biaxial extension properties are most likely involved during dough shaping (moulding, sheeting and oven rise). The Lubricated Squeezing Flow (LSF) is one method used to study the biaxial extension for biscuit dough (Bartolucci and Launay, 2000). Thus, the doughs mixed for making biscuits were tested by both a double compression test and a biaxial compression via the LSF test. All stress relaxation curves were very well fitted with equation 1, and correlation coefficients were always higher than 0.99.

The Aw of the native and reconstituted doughs remained relatively constant among all the recipes, i.e. at 0.89 ±0.05. As expected, the texture profile analysis of the dough, presented in Table 3.4, demonstrated that the hardness, quantified by the double compression and also by the maximum force at 40% compression of the LSF method (Fmax), yielded the same trends, i.e. an increase of approximately 21 to 28% from the patent to the clear fraction, likely due to the higher protein content of the clear flour. Higher hardness values noted with the LSF method could be attributed to the 5-10 min additional resting time of the biscuit dough to complete both, the double compression and compression-relaxation rheological tests on the same dough materials. A similar raise was noted with the reconstituted variants from the central to the peripheral portion of the wheat endosperm; however, the reconstitution procedure lowered these values by ~ 17-28% versus the parent flour counterparts. The consistency increased considerably from 19.3 to 25.5 N.s and elongational viscosity from 0.41 to 0.55×10^{-5} Pa.s from the native patent to the clear. Moreover, the reconstitution procedure reduced the consistency by ~ 15-35% and elongational viscosity by ~17-27% as compared to their parent counterparts. Furthermore, the area under the 2nd peak of the double compression test was significantly lower than the first peak, indicating that less energy is required for the second compression because of lower elastic recovery. These results corroborated the observations reported by Graßberger et al. (2003) and Uthayakumaran and Lukow (2003) on reconstituted hard wheat flour, where a moderate to considerable decrease in extensibility and maximum resistance of dough and gluten of simple water/flour dough was recorded. The cause of direct change in these rheological parameters related to flour reconstitution could be explained by many factors, namely the aggregation of gluten proteins by mechanical denaturation due to hand kneading (Laszity,1984; Masi et al.,1998; Wu and Hoseney,1990), the new water distribution among components in the composite network (Köhler et al.,1999), the change in the original state of the components dispersion (Tolstoguzov, 1997, 2002), the possible intervention of endogeneous proteolytic activity, and many others unidentified factors.

Among the dough rheological parameters, springiness and cohesiveness showed the least difference among the flours, but the reconstitution procedure appeared to raise slightly the cohesiveness or internal interactions responsible for the dough structure; the reconstituted fractions might be more hydrophilic and, thus, absorb more free water during dough development, making the recombined patent especially more sticky than its native counterpart.

For the biscuit's dimensions resulting mainly from an elastic retraction of the dough as soon as it is sheeted and cut-out, three LSF rheological parameters were evaluated to predict their length as suggested by Renard and Théry (1998) and Bartolucci and Launay (2000) and Tla reflects the time necessary to reduce the final compression force by half, once the compression is removed due to elastic retraction of the dough. A higher Tla value is translated into a higher speed of recovery of the dough once laminated and cut and thus reduced length of the biscuits in the laminating direction. The Tla values ranged between 0.45 to 0.59 s from the native patent to clear and they were higher for the native compared with the reconstituted flours; the differences in Tla values were smaller for the clear flours (Table3. 4), probably due to higher protein content which result in higher viscoelastic properties. Figure 3 shows the direct relationship between the biscuit length and Tla values (r^2=0.97). In parallel, the relaxation rate constant (k) translated the internal constraints accumulated into the dough; a higher k values indicates that these constraints are relaxing more rapidly, and consequently the biscuit retraction should be reduced resulting in higher biscuit length as shown in Fig 3.4 (r^2=0.95). The Tla and k responses can be explained in terms of elastic recovery; dough compressed during the sheeting process stores mechanical energy in the dough, inducing partial strain recovery. High *k* values mean that the energy stored in the dough dissipates more rapidly, and as a result, the dough recovery is smaller and the dough retraction is minimized. The *k* parameter declined from 4.51 to 3.19 and to 3.09 s^{-1} from the patent, middle-cut and the clear fractions, respectively, whereas the Tla values rose from 0.45, 0.52 and 0.59 s for the same respective flours. The recombination procedure raised the relaxation rate constant for the patent and middle-cut fraction, but yielded almost unchanged *k* values in the case of the clear fraction, probably due to the greater concentration of gluten in this fraction that allows the dough to withstand more easily the reconstitution procedure. Smaller differences for the Flow behaviour index (*n*) were observed among the three flour materials (Table 3. 4).

5.5. Biscuit dimensional characteristics

For packaging, constancy of the biscuit dimensions is important to ensure conformity with the standard package size. Length and width of the semi-sweet biscuits were correlated (r= -0.83), suggesting that these parameters are dependent on the viscoelastic properties of the dough; following stamping, the biscuit length will tend to decrease along the lamination direction while its width and thickness will increase as a result of partial strain recovery.

The clear fraction induces a decrease in the biscuit length with both the reconstituted and the native flours. Higher protein and pentosans contents which enhance the hydration properties of the flour tend to produce sugar-snap cookies with a lower spread (Bushuk, 1966; De La Roche and Fowler, 1975; Donelson, 1990; Kaldy and al., 1991). In parallel, the semi-sweet biscuits showed the same tendency with length reduction. The reconstitution procedure had little influence on the biscuit final length (increase of 3-5% for the reconstituted patent and middle-cut versus the parent flour and a decrease of 3-7% for the clear). The clear flour contributes to the increase of the elastic nature of the dough, its shrinking following lamination and thereby results in lower biscuit length and higher thickness (Table 5). Moreover, the volume of the biscuits made with the patent and middle-cut fractions was only slightly altered by the reconstitution procedure. These data can be favourably compared to results obtained with bread volume data for products made with different reconstituted hard wheat flour cultivars, e.g. a decrease in loaf volume by ~2.5 to 9.0% (Uthayakumaran and Lukow, 2003; Graßberger et al., 2003). The density rose significantly from 0.32 to 0.35 g/cc, from the patent to clear flour fractions, respectively. This is attributed to the higher protein content which contributes to ensure a well formed protein matrix resulting in a more dense biscuit at comparable moisture contents (3.8-4.2%). Reconstitution had a marginal effect on the density parameter.

5.6- Biscuit colour and appearance

Colour expressed as the changes in the Hunterlab tristimulus attributes, L, a, b, of the biscuit surface confirmed that the biscuits made with recombined flour had a darker colour than those made of the native flours; i.e. a significant decrease in the brightness (L) value with a concomitant shift of the coefficient a towards the red component (a) or the red shade of the colour were observed (Table 6). The browning of biscuits is particularly noticeable with the use of the clear flour in the recipe. Instead the parameter b remained rather constant. A higher degree of Maillard reactions in the reconstituted products may be due to the starch and protein degradation during the fractionation leading to higher amounts of free amino acids and reducing sugars in the recombined flours, as suggested by Feather (1994).

All the biscuits made with both the native and reconstituted flours had normal surface appearance, with no cracking pattern due to the sugar crystallisation upon cooling, this is likely due to the lower level of sugar incorporated in the semi-biscuit recipe than in sugar snap cookie which generally tends to show a cracking pattern.

5.7- Biscuits mechanical properties

The mechanical properties of biscuits are salient quality attributes as they have a direct impact on the sensory perception and consumer acceptance. Semi-sweet biscuits are products with a slightly compact granular structure, consisting of an assembly of grains, trapping a small quantity of air not uniformly distributed. Assessment of the mechanical properties, as measured by conical penetrometry that couples both compression and shear forces, permits good characterization of the textural properties of biscuits since they are very sensitive to relatively small changes in the biscuit internal structure differences (Goullieux et al., 1995). Water, sugar and fat, for most cookie products, are the ingredients that most influence the mechanical properties, but little is reported on the role of flour quality, by itself, on the mechanical attributes of the end-products. The internal structure of biscuits is responsible for the textural quality and the gluten which forms the protein network surrounding the starch granules largely contributes to this internal structure .i.e. gluten is the major element in the technological value of the flour for biscuit manufacture (Yamazaki and Donelson, 1976). The analysis of fracture pattern on the force-distance or force-time graphs allows the quantification of several textural attributes of the products. Table3.5 indicated that the specific tearing force (Fs) for particles or group of grains in the biscuit made with parent flours increased from the patent, middle-cut and clear fractions from 1.61, 1.90 and 2.14 N, respectively. This implies that the clear flour creates stronger bonds between the particles after baking, presumably due to the higher protein and water-solubles contents, in agreement with the work of Gaines et al (1994) and Maache-Rezzoug et al. (1998). Such cohesion forces declined by approximately 11-13.7% with the reconstituted patent and middle-cut flour, but an inverse tendency was noted with the reconstituted clear fraction, where an increase of 17.3%, or 0.37 N was observed compared to its native counterpart. It is postulated that due to the higher protein content of this flour coupled with the fractionation/reconstitution treatment procedure, the creation of strongest bonds between particles is favoured. The spatial frequency (No) or the number of grains or group of grains per unit of penetration shows similar trend with that of the Fs values, rising from 7.86, 9.10 to 9.76 mm^{-1} with the native patent, middle-cut and clear mill stream, respectively. The fractionation and reconstitution procedure increased the No values by ~ 28 to 32% in the patent and middle-cut flours, respectively, but decreased it in the clear by ~ 22.5%. These data suggest that during baking and cooling of the biscuit, the clear flour formed fewer grains. The Fs/No ratio or friability effort, also referred to crunchiness remain relatively constant with the biscuits formulated with the original three flours (0.21 to 0.22 Nmm). A friable product should exhibit during its penetration a greater number of teared grains (high No) for a low tearing force (low Fs), thus a lower ratio Fs/No can reflect a more friable end-product. Friability of the biscuits made with the three native flours was quite similar. However, the reconstituted patent and middle-cut flours showed a pronounced decrease of the Fs/No ratio versus the native flour counterparts, but an inverse effect was noted with the clear reconstituted flour. The average puncturing force, referred to as Sm, which reflects the biscuit's resistance to solicitation, also rose from the patent to clear flour from 6.76 to 12.5 N, respectively.

5.8- Correlations between rheological and biscuits parameters

Many aspects of biscuit processing and end-product quality attributes are positively or negatively correlated with the dough rheological parameters (Table 3.7). None of the rheological parameters allow the prediction of biscuit thickness and volume which depend on oven rise during baking. However, the biscuit density was positively correlated with the Fs ($r= 0.973$) and Sm ($r= 0.935$, $p \leq 0.01$). The cohesion of the biscuit structure (Fs), reflecting interparticle interactions, correlated positively to the half-relaxation time ($r= 0.865$, $p\leq0.05$) and mean penetration force or Sm ($r= 0.954$, $p\leq0.01$) and negatively with the rate of relaxation ($r= -0.861$). The resistance to solicitation or Sm was negatively correlated with the rate of relaxation ($r= -0.856$), but positively correlated to Tla ($r=0.875$). These data confirm that the relaxation curves of the LSF method; particularly the Tla and k parameters are good predictors of semi-sweet biscuits quality characteristics such as biscuit length and also some textural properties. Certain rheological parameters are also correlated among themselves, e.g. the dough consistency correlated positively with Tla ($r= 0.944$, $p\leq0.01$), elongational viscosity ($r= 0.979$, $p\leq0.001$), hardness ($r=0.964$, $p\leq0.01$) and negatively correlated with k ($r= -0.934$, $p\leq0.01$). In parallel, the half relaxation time was negatively correlated with the rate of relaxation ($r=-0.962$) and positively correlated with the elongational viscosity ($r = 0.968$) and consistency ($r= 0.944$, $p\leq0.01$). This suggests that dough hardness, consistency and elongational viscosity could also be predicted via the determination of the viscoelasticity of the biscuit dough as expressed by the Tla and k indices.

6. Conclusions

There are major compositional and functional differences among the patent, middle-cut and clear fractions obtained from a commercial milling plant. These differences are also reflected in large variation in the rheological properties of the biscuit dough and biscuit characteristics. Incorporation of the central endosperm as flour material in a semi-sweet biscuit recipe usually produces biscuits of superior length in the direction of sheeting, with lower cohesion forces among the grains of the biscuit structure, and easier to break than the peripheral portion of the endosperm which, in turn, yields biscuits of higher density and higher cohesion forces among grains or group of grains in the biscuits made with native flours. The flour fractionation procedure influenced moderately all these attributes confirming that in spite of the gentle treatment (hand kneading with no use of chemical agents for the extraction of lipids) the functional properties of the native flours could not be restored completely. Biscuit quality indices such as length, volume and density were significantly less altered by this procedure than the rheological attributes such as dough hardness, consistency, Tla and elongational viscosity of their respective flours grades. However, the reconstitution promotes considerably the browning of the products.

The present study also confirmed that even if empirical methods are still used for assessing the rheological properties of wheat flour dough, the viscoelastic behaviour in biaxial compression and relaxation of biscuit dough provides interesting information to relate some dimensional and textural characteristics of the biscuits to the data of the stress relaxation phenomenon taking place during dough processing, especially the half relaxation time and relaxation rate constant which are useful predictors of the biscuit length, density and friability. The semi-sweet biscuit recipe with higher flour and lower sugar and fat contents yielded very valuable technogical data to characterize the flour functionality.

The work also demonstrated the potential of using different wheat endosperm fractions in biscuit making to obtain the sought biscuit dimensions, density and mechanical properties and that an appropriate fractionation and reconstitution procedure could serve as a means to further investigate the effect of individual flour constituents (starch, lipids and protein sub-fractions) on dough rheology and baking performance. However, the plasticizing effect of water on wheat endosperm proteins which may vary from the original to reconstituted flours and further depend on pentosans and damaged starch levels should be additionally considered in future studies. Moreover, it would be necessary to develop predictive models, which could explain from the dough's thermomecanical properties, the changes occurring in the dimensional characteristics during the different phases of semi-sweet biscuit production by employing the three fractions isolated from flours of different biochemical composition as this approach has already been considered with cookies.

7. Acknowledgements

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Table 3.1: Physico-chemical characteristics of the flour streams

Flour Grades	Protein content (%) *	Damaged starch (%)*	Ash content (%) *	$W.10^{-4}$ J	Alveograph L (mm)	P (mm)**	P/L	AWRC (%)
Patent	7.41	3.6	0.408	73	82	28	0.341	63.4
Middle-cut	9.65	3.9	0.571	92	99	38	0.384	65.7
Clear	12.7	4.9	0.995	80	75	50	0.666	74.9

Note: * on 14% moisture basis
** P: (height *1.1)mm

Table 3.2: Proportions and composition of freeze dried fractions isolated from their respective flours (g/100g dry matter)

Grades and Fractions	Proportions (%)	Moisture (%)	Total proteins (%)	Total pentosans (%)	Lipids (%)	Starch [d] (%)
Patent	100.0	12.2	7.41	0.99 [c]	1.26	89.9 [d]
Starch [a]	86.7 (0.79)[b]	4.73	0.26	0.45	1.13	
Gluten	7.97 (0.42)	4.66	71.8	-	8.27	
Water soluble	5.27 (0.21)	8.12	18.1	10.2	-	
Middle-cut	100.0	11.5	9.65	1.56	2.07	86.2
Starch	82.3 (1.83)	2.12	0.45	1.05	0.99	
Gluten	11.3 (0.34)	3.57	71.2	-	8.31	
Water soluble	6.43 (0.69)	6.64	18.3	11.1	-	
Clear	100.0	11.2	12.7	1.76	3.23	81.3
Starch	75.8 (1.56)	2.55	1.31	1.37	1.10	
Gluten	15.6 (0.71)	3.72	68.1	-	11.2	
Water soluble	8.59 (0.54)	9.59	22.1	6.94	-	

[a] Starch tailing constitutes 6.8, 13.4 and 18.9% and prime starch 79.9,68.9 and 56.9% of the crude starch content for the patent , middle cut and clear flours respectively

[b] Data are means of four repetitions, standard deviation (in parentheses)

[c] Total pentosans is constituted of both soluble and insoluble pentosans

[d] Starch+protein+pentosans +lipids+ash equivalent to 100% on d.w

Ash content: 0.408, 0.571, and 0.995 % and damaged starch: 3.5, 3.8 and 4.9% for the patent, middle- cut and clear respectively

Table 3.3: Solvent Retention Capacity (SRC) of the parent versus fractionated flour fractions

Flour grades	Water (%)		Sodium Carbonate (%)		Sucrose (%)		Lactic acid (%)	
	As is	Reconst	As is	Reconst	As is	Reconst	As is	Reconst
Patent	47.7 (0.17)	54.3 (1.41)	64.9 (2.53)	64.6 (0.98)	72.6 (0.85)	74.2 (0.54)	92.9 (1.05)	96.5 (1.78)
Middle-cut	49.2 (0.94)	55.5 (0.36)	63.4 (1.84)	66.4 (1.12)	76.9 (1.08)	79.5 (1.89)	81.3 (0.56)	79.8 (1.87)
Clear	53.5 (0.36)	59.6 (0.52)	74.5 (1.05)	78.9 (1.24)	93.5 (0.45)	91.7 (0.78)	70.9 (1.24)	72.5 (2.87)

Data are means of three replicates (number in parentheses are s.d)

Table 3.4: Means of dough rheological parameters of the three native and reconstituted flours

	Patent		**Middle- cut**		**Clear**	
	Control	Reconst	Control	Reconst	Control	Reconst
Hardness (N)	3.77 [c]	3.07 [d]	4.42 [b]	2.98 [d]	4.84 [a]	3.78 [c]
Cohesiveness	0.26 [c]	0.32 [a]	0.27 [bc]	0.30 [b]	0.30 [b]	0.33 [a]
Consistency (N.s)	19.3 [d]	13.5 [f]	23.8 [b]	15.3 [e]	25.5 [a]	21.6 [c]
Springiness	0.38 [b]	0.55 [a]	0.37 [b]	0.43 [b]	0.41 [b]	0.43 [b]
Fi (N)	4.36 [b]	3.11 [d]	4.57 [b]	3.22 [d]	5.27 [a]	3.94 [c]
Tla (sec)	0.45 [d]	0.37 [f]	0.52 [c]	0.42 [e]	0.59 [a]	0.56 [b]
k (s^{-1})	4.51 [c]	5.19 [a]	3.19 [d]	5.14 [b]	3.09 [e]	2.99 [f]
n	0.35 [a]	0.34 [a]	0.32 [b]	0.30 [c]	0.32 [b]	0.27 [d]
Elong. Vis (Pa.s*10^{-5})	0.41 [c]	0.30 [e]	0.46 [b]	0.34 [d]	0.55 [a]	0.46 [b]

Values within a row with the same letter are not significantly different at P<0.05

Table 3.5: Means of the biscuits parameters of the patents and clear flours and reconstituted flours

	Patent		Middle-cut		Clear	
	Control	Reconst	Control	Reconst	Control	Reconst
Length (cm)	6.39 ^{b}	6.56 ^{a}	6.24 ^{b}	6.54 ^{a}	6.19 ^{c}	5.96 ^{c}
Volume (ml)	35.9 ^{a}	35.7 ^{a}	34.8 ^{ab}	33.5 ^{bc}	35.8 ^{a}	32.1 ^{c}
Thickness (mm)	8.75 ^{b}	8.76 ^{b}	8.60 ^{b}	8.21 ^{c}	9.19 ^{a}	8.15 ^{c}
Density (g/cc)	0.32 ^{d}	0.32 ^{d}	0.33 ^{c}	0.33 ^{c}	0.35 ^{b}	0.38 ^{a}
Fs (N)	1.61 ^{d}	1.43 ^{e}	1.90 ^{c}	1.64 ^{d}	2.14 ^{b}	2.51 ^{a}
Sm (N)	6.76 ^{e}	6.50 ^{e}	10.7 ^{c}	9.21 ^{d}	12.5 ^{b}	14.2 ^{a}
No (mm^{-1})	7.86 ^{d}	10.1 ^{b}	9.10 ^{c}	12.0 ^{a}	9.76 ^{b}	7.56 ^{d}
Fs/No (N.mm)	0.205	0.141	0.209	0.136	0.219	0.332

Values within a row with the same letter are not significantly different at P<0. 05

Table 3.6: Moisture and colour attributes of the biscuits from the native and reconstituted flours fractions

	Patent		Middle-cut		Clear	
	Control	Reconst	Control	Reconst	Control	Reconst
Moisture (%)	3.68	3.48	3.62	3.32	3.65	3.57
Aw	0.234	0.231	0.234	0.236	0.228	0.229
Hunterlab L	84.4	66.5	82.1	63.4	76.0	55.8
Hunterlab a	2.91	6.33	3.45	6.98	5.22	9.10
Hunterlab b	19.2	19.9	19.5	19.5	20.4	19.8

Average of 5 readings. Results in bracket are the standard deviation

Table 3.7: Pearson correlation matrix for end-product quality parameters with rheological characteristics

	Fi	Tla	Ka	Elong.Vis.	Consis	Hardn	Vol	Thick	Dens	Fs	Sm	No
Fi	1.000	0.826 *	-0.768	0.938**	0.937**	0.974***	0.357	0.612	0.254	0.447	0.463	-0.423
Tla	0.826*	1.000	-0.962*	0.968**	0.944**	0.863	-0.185	0.208	0.750	0.865*	0.875*	-0.472
Ka	-0.768	-0.962*	1.000	-0.910*	-0.934**	-0.847*	0.230	-0.131	-0.739	-0.861*	-0.856*	0.565
E.Vi	0.963**	0.968**	-0.910*	1.000	0.979***	0.947**	0.046	0.402	0.565	0.715	0.739	-0.432
Cons	0.937**	0.944**	-0.934**	0.979***	1.000	0.964**	0.042	0.357	0.510	0.681	0.707	-0.473
Sprin	-0.713	-0.586	0.544	-0.665	-0.720	-0.626	0.013	-0.038	-0.149	-0.340	-0.358	0.304
Hard	0.974***	0.863*	-0.847*	0.947**	0.964**	1.000	0.290	0586	0.332	0.511	0.550	-0.412
Vol	0.357	-0.185	0.230	0.046	0.042	0.290	1.000	0.893	-0.706	-0.604	-0.592	0.057
Thick	0.612	0.208	-0.131	0.402	0.357	0.586	0.893	1.000	-0.331	-0.227	-0.189	-0.032
Den	0.254	0.750	-0.739	0.565	0.510	0.332	-0.706	-0.331	1.000	0.973*	0.935**	-0.392
Fs	0.447	0.865*	-0.861*	0.715	0.681	0.511	-0.604	-0.227	0.973**	1.000	0.954**	-0.511
Sm	0.463	0.875	-0.856	0.739	0.707	0.550	-0.592	-0.189	0.935**	0.954*	1.000	-0.265
No	-0.423	-0.472	0.565	-0.432	-0.473	-0.412	0.057	-0.32	-0.392	-0.511	-0.265	1.000

Fi: hardness (LSF test), Tla; half relaxation time; Ka: rate of relaxation; E. Vi: elongational viscosity; Cons: consistency Spring: springiness; Hard: hardness (double compression); Vol: volume; Thick: thickness, Den: density; Fs: mean tearing force Sm: mean penetration force ; No:number of spatial rupture.

*p<0.05, **p< 0.01, *** p< 0.001, ****p <0.0001 respectively

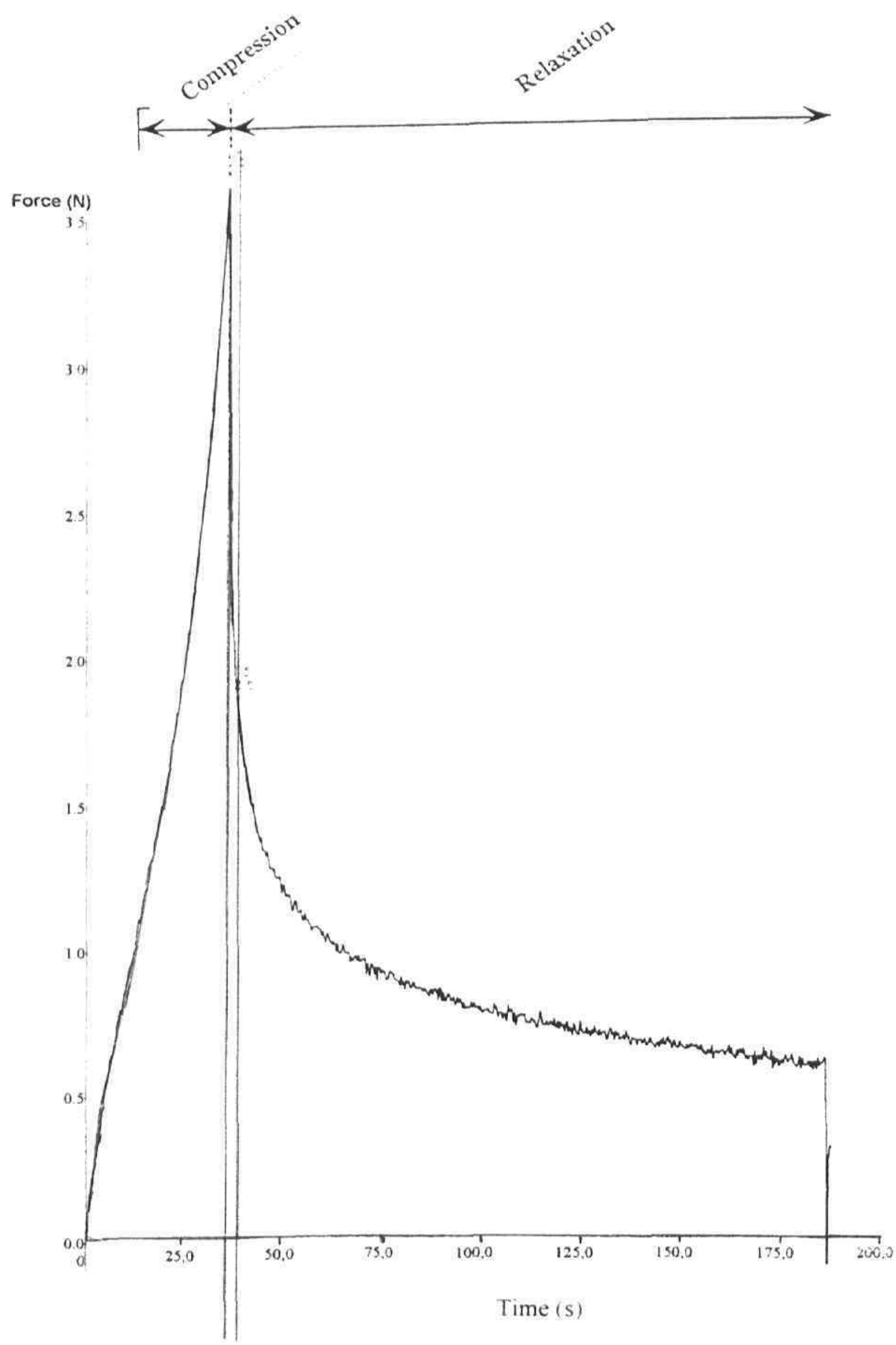

Figure 3.1: Typical compression –relaxation curve after lubricated squeezing flow

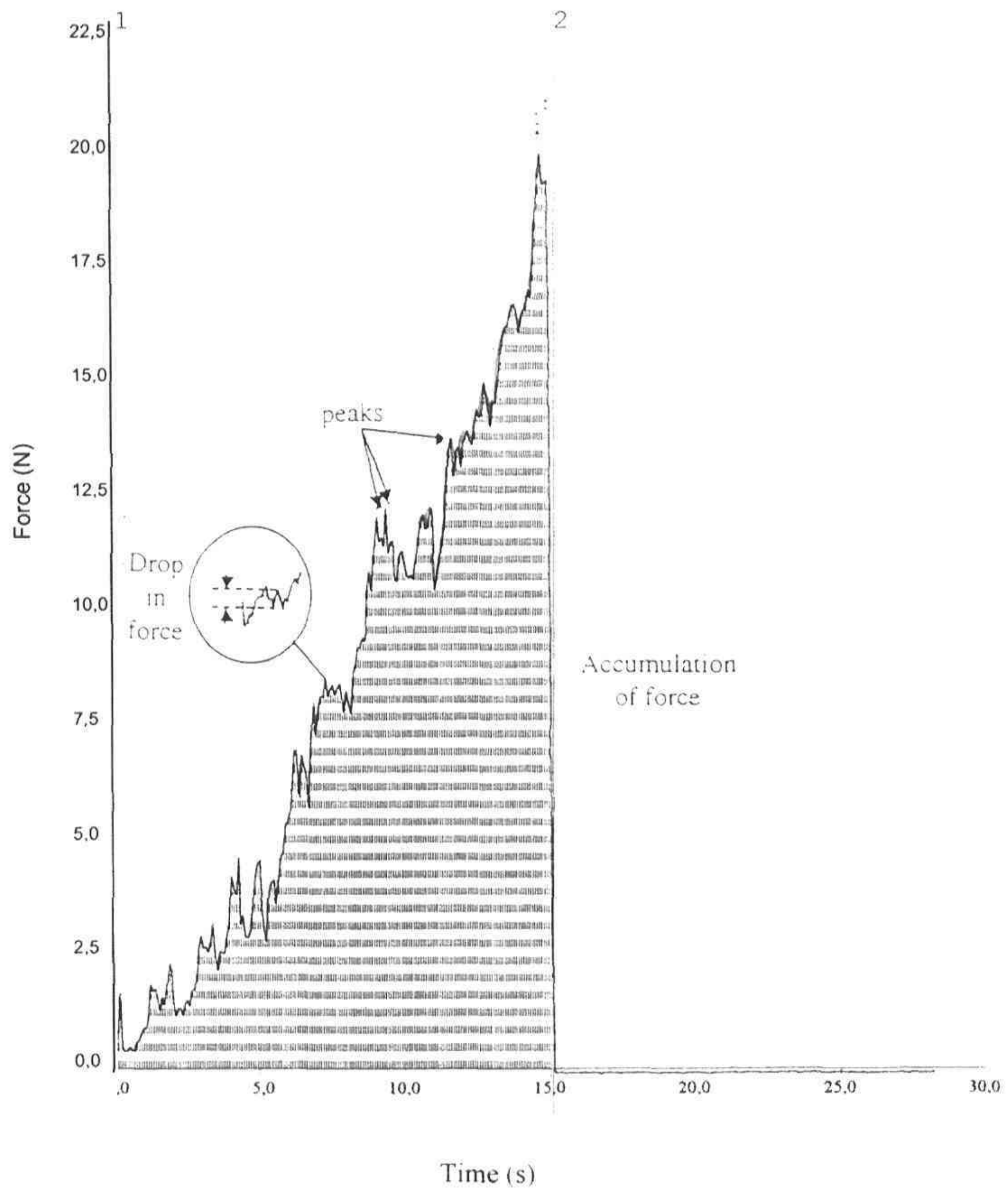

Figure 3.2: Typical evolution of force versus time by conic penetrometry on biscuits

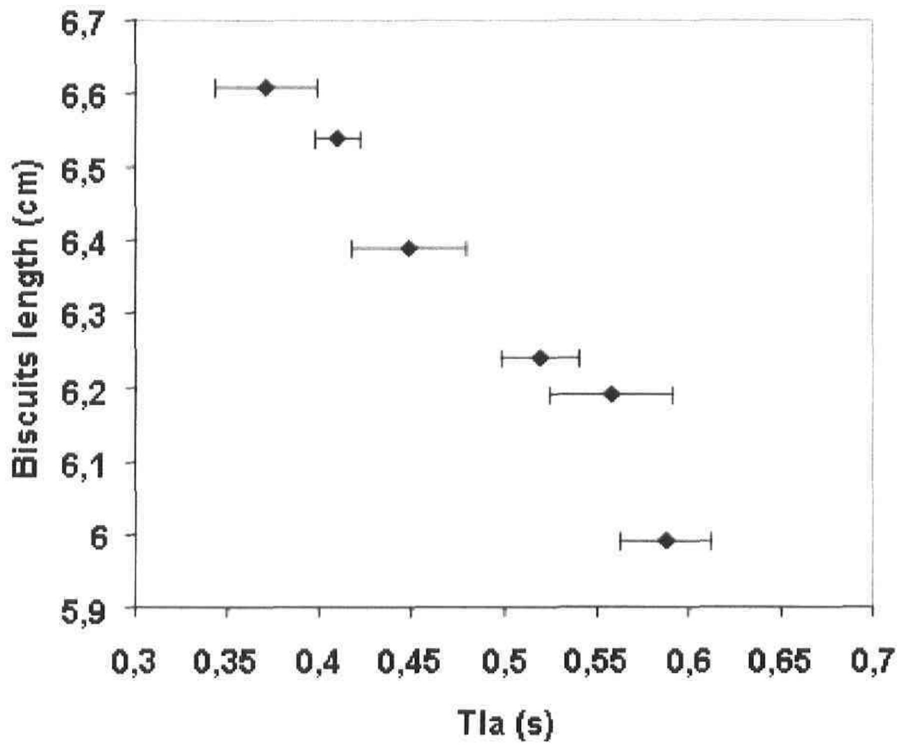

Figure 3.3: Relationship between Tla and biscuit length

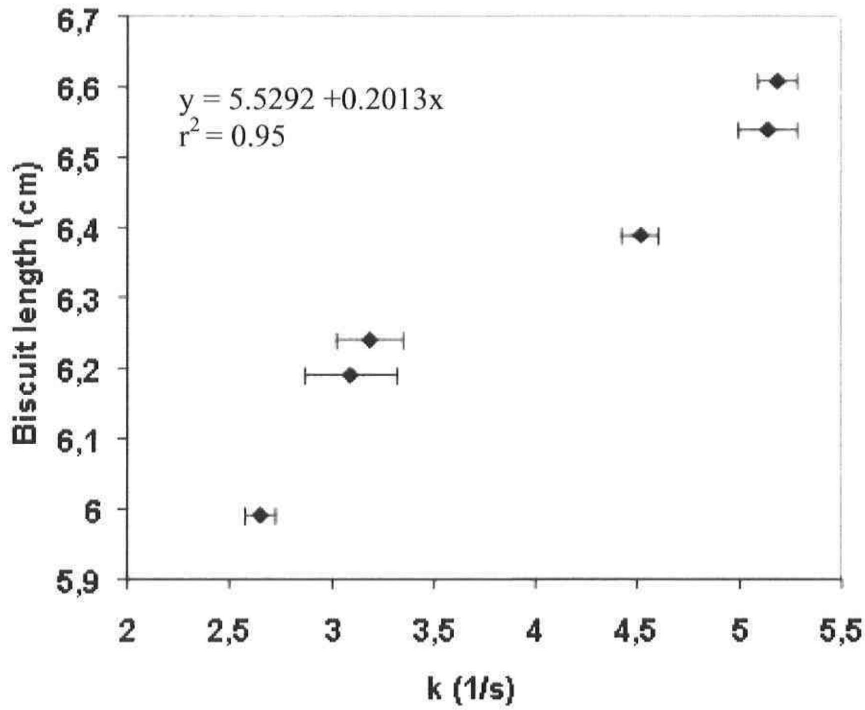

Figure 3.4: Relationship between k and biscuit length

« *I do not know what I may appear to the world; but to myself, I seem to have been only like a boy playing on the seashore, and diverting myself in now and then finding a smoother pebble or a prettier shell than ordinary, whilst the great ocean of truth lay all undiscovered before me*»

Isaac Newton (1642-1727)

CHAPTER IV

Flour constituent interactions and their influence on dough rheology and quality of semi-sweet biscuits: a mixture design approach with reconstituted blends of gluten, water-solubles and starch fractions isolated from different soft wheat flour streams

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1-Résumé

Cette étude consistait à identifier les paramètres biochimiques et technologiques qui modifient la fonctionnalité de la farine de blé tendre pour la biscuiterie; nous avons choisi un biscuit semi-sucré comme produit standard. Un simplexe centroïde à neuf points a été utilisé afin de déterminer l'effet de diverses proportions des fractions gluten, hydrosolubles et d'amidon isolées des trois grades de farine (patente, intermédiaire et basse) possédant une vaste gamme de caractéristiques compositionnelles et de fonctionnalités influent sur le comportement rhéologique des pâtes et les paramètres de qualité des biscuits. La teneur en protéines et pentosanes solubles et insolubles, et les lipides endogènes de chacune de « farines modèles» a été quantifiée. Pour chacune de ces farines modèles, la consistance, la viscosité élongationnelle, la dureté, le temps de demi relaxation, la constante de vitesse de relaxation, la cohésion et l'élasticité des pâtes, ainsi que la densité, la fermeté, la force de déchirement et la fréquence spatiale des biscuits ont été mesurées. Des modèles de régression ont été élaborés pour prédire comment les caractéristiques rhéologiques des pâtes et les paramètres de qualité du biscuit changent en fonction des changements de composition de ces mélanges de fractions; en plus des principaux termes linéaires (concentration des fractions d'amidon, de gluten et hydrosolubles). D'importants termes d'interaction qui ne sauraient être considérés comme négligeables lorsqu'il s'agit de prédire les propriétés des pâtes et biscuits ont ainsi été déterminés. Avec les modèles élaborés, nous avons tracé les courbes iso-contour et « trace plots» pour mieux comprendre le comportement des farines modèles sur les propriétés des pâtes et biscuits. Les coefficients de corrélation Pearson, qui servent à décrire les relations entre la composition biochimique des «farines modèles» et les propriétés rhéologiques de la pâte et les caractéristiques du biscuit, ont permis de mettre en évidence le rôle fonctionnel des protéines solubles et insolubles totales, des pentosanes totaux et des lipides totaux pour les biscuits secs.

2-Abstract

The biochemical parameters affecting soft wheat flour functionality in biscuit making have been explored using a semi-sweet biscuit product as a study system. A nine-point simplex centroid was used to investigate the effect of varying the ratios of gluten, water-solubles and starch fractions isolated from three different flour grades (patent, middle-cut and clear flours) which exhibited a wide range of compositional and functionality characteristics on the dough rheological behaviour and the biscuit quality parameters. The amounts of soluble and insoluble proteins and pentosans as well as the endogenous lipids in each flour fraction were quantified. Dough consistency, elongational viscosity, hardness, half-relaxation time, relaxation rate constant, cohesiveness and springiness as well as biscuit density, firmness, tearing force and spatial frequency for the different flour fraction combinations were assessed. Regression models have been applied to predict the responses of the rheological attributes of the dough as well as the biscuit quality characteristics to the compositional changes of the flour blends; in addition to the main linear terms (concentration of starch, gluten and water-solubles isolated from the different flour grades), significant interaction terms were identified which cannot be neglected in any prediction scheme for the dough and biscuit properties. Contour plots were drawn in an effort to better understand the overall property responses of the dough and biscuits. The Pearson correlation coefficients, describing the relationships between individual flour constituents in the combined fraction blends and the dough rheology and biscuit characteristics, were used to unravel the functional role of the total, soluble and insoluble proteins, pentosans and lipids in biscuit making.

3- Introduction

Soft wheat flours are complex mixtures of constituents such as starch, proteins (e.g. gluten proteins), lipids and various non-starch polysaccharides (e.g. pentosans). Studies on the relationships between composition and functionality of soft wheat flour components in relation to semi-sweet biscuit quality are limited and essentially conducted on the spread and the surface-cracking pattern of the standard sugar snap cookie recipe (flour: sugar: water: fat of 100:60:15:30 on 14% moisture basis) (Kaldy et al., 1991; Nemeth et al., 1994). Moreover, a complete quantitative and qualitative description of the effects of flour constituents on cookie quality still remains largely unresolved due to many complex interactions among the flour components and also the variations in their concentration among wheat cultivars and crop years. Cookie quality is generally associated with soft wheat flour of low protein content (Hoseney et al., 1988; Wade, 1995; Morris and Rose, 1996), smaller particle size (Rogers et al, 1993; Kaldy et al., 1993) and low alkaline water retention capacity (Kaldy and Rubenthaler, 1987).

Numerous works refer to the negative impact of protein content on cookie spread (Sollars, 1959; Souza et al., 1994). Abboud (1985b) found that the negative correlation was minimal among soft wheat flours, while Nemeth et al. (1994) found no correlation between cookie diameter and protein content for a group of soft wheat flours from different countries; however, within the same cultivar, the diameter depended mainly on the protein content ($r = -0.87$) when fertilization levels varied. A negative correlation was also established between the tail-end fraction of the milling process, rich in insoluble pentosans and higher in damaged starch, and the cookie spread (Yamazaki, 1955; Sollars, 1956; Upton and Hester, 1966; Abboud et al., 1985). The negative role of soluble pentosans on the cookie diameter was also reported in the literature (Cottenet, 1986; Kaldy et al., 1991). The water sorption capacity of pentosans is often referred to impact soft wheat flour functionality, with the soluble pentosans affecting protein hydration and thereby dough and biscuit properties (Jeltama et al., 1983). According to Sollars and Bowie (1966), the degree of the sugar snap cookie spread was mainly related to the amount of free water or the flour constituents capable of binding water, whereas in other studies (Rogers et al., 1993; Nemeth et al., 1994) the concentration of water controlling components in the flour (pentosans, proteins, damaged starch) was poorly correlated with cookie diameter. Free and polar flour lipids can also contribute to the intrinsic quality of soft wheat flour; once removed, cookie diameter is reduced along with a browner colour and a poorer surface appearance (Clements and Donelson, 1981). The starch, one of the major flour constituents, is often described as inert filler embedded in the continuous protein matrix (Wade, 1988). Nevertheless, the damaged starch sub-fraction exerts a significant influence on spread reduction (Abboud et al., 1985b). Further to variation in flour constituent composition among batches of soft wheat flours, the complexities of the cookie recipes, with varying amounts of sugar and fat, may impact the development of the gluten network (Flint et al., 1970; Abboud, 1985a) and modify the extent of starch gelatinization on baking (Varriano-Marston et al., 1980; Wade, 1995).

The role of gluten, starch, lipids and water-solubles has been often examined in cookie making by the fractionation/reconstitution technique and interchange studies (Yamazaki, 1955; Sollars, 1956a,b; Sollars and Bowie, 1966; Yamazaki et al., 1977; MacRitchie, 1985; Donelson, 1988). Cookies baked from the blends of such flour fractions may match closely the spread of those made with the native flour. Previous reconstitution studies conducted on gluten, crude starch and water- soluble fractions isolated from various flour streams with markedly different biochemical properties revealed that the biscuits exhibited acceptable dimensional characteristics and surface appearance compared to their native flour counterparts, in a semi-sweet biscuit recipe with a flour: sugar: fat: water ratio of 100:30:8:36 on 14% flour basis (unpublished). Compared to cookie where the spread is an index of flour quality, the absence of retraction of the dough after sheeting and baking, the development of a biscuit with low density and appropriate crunchiness are considered as criteria of flour quality for the semi-sweet biscuits (Tharrault; 1994a, b; Contamine et al., 1995). There has been already a lot of work done trying to understand the role of other cookie ingredients such as sugar and fat incorporated into the various cookie recipes on the quality of end-products (Abboud et al., 1985a; Maache Rezzoug et al., 1998b), but little attention has been paid to the role of gluten, water-soluble and starch fractions and, more specifically, on the effect of their constituents on dough rheology and biscuit quality.

The present investigation was therefore undertaken to examine the impact of gluten, starch and the water-soluble fractions isolated from commercial patent, middle-cut and clear flours via a mixture design on both the rheological attributes of the dough and the mechanical properties of semi-sweet biscuits. The experimental approach was based on inducing a variation in the concentration of these three fractions used in the reconstituted flour mixtures to yield different levels of protein, lipids, pentosans and starch, and to explore the impact these constituents have on the dough and biscuit characteristics.

4- Materials and methods

4.1- Wheat materials

The wheat flour grades used for this study are described in the section 4.1 of the chapter 3.

4.2- Flour grades and fractions analysis

Moisture contents of flour grades and flour fractions (gluten, starch and water solubles) were determined according to the AACC method 44-15A (2000). Total protein content was measured by the LECO PF-428 nitrogen/protein analyzer (LECO Instruments Ltd., Mississauga, Ontario, Can); the nitrogen content was multiplied by 5.7. Soluble and insoluble protein, referred to as insoluble glutenin, contents were carried out by the method of Sapirstein and Johnson (2000). Total, insoluble and water-soluble pentosans were determined according to the method of Rouau and Surget (1994). Determination of lipids was carried out by an acid hydrolysis method, AOAC 922.06 (2000).

4.3- Fractionation of flour

The experimental approach of MacRitchie (1985) was used as a basis for the fractionation protocol is described in the section 4.3 of the chapter 3.

4.4- Rheology of the dough and the biscuit evaluation

All these procedures were previously described in the chapter 3, sections 4.4, 4.5 and 4.6.

4.5- Experimental Design and data analysis

A mixture design was constructed to enable the study of the effect of ratios of gluten, water solubles and starch fractions. The ADX menu of the SAS/QC module was used to build the experimental design (2004). It was used to select the vertices of a sub-region of the simplex. The variables in the constituent mixtures were gluten (X_1), starch (X_2) and water-solubles (X_3) isolated from three commercial flour grades of a soft wheat mill operation (patent, mid-cut and clear fractions). Proportions of the independent variables in the mixtures were calculated in fractions or % and the sum was equivalent to 1, resp. 100%. The proportions were constrained, i.e. there is a minimum and a maximum for gluten and starch, and a maximum for the solubles-fraction to yield different flours of varying technological value, for instance dough that could be sheeted and not be sticky or biscuits with appropriate dimensional and textural properties. The sub-region corresponds to the nine-point simplex-centroid design of Cornell and Harrisson (1990, 1997), as shown in Table 4.1 and it was used to formulate the three-constituent mixture systems. Some design points were replicated in order to assess the model lack of fit. The response or dependent variables were the rheological parameters of the dough (hardness, consistency, elongational viscosity, etc.) and the biscuits dimensional and textural properties (volume, density, firmness, etc.)

In order to assess the predictive ability of the regression model, ideally a second dataset would be needed. As it is not possible, an alternative strategy commonly referred to as cross-validation was used. The principle consists of leaving out one observation from the model, fitting the model on the remaining observations and calculating the difference between the observed value and the predicted value for the one that was left out of the model and repeating this process over all observations in the initial data set. Using this approach the residual sum of squares (R^2 pred) can be obtained for each of the models. The R^2 pred values are used to compare the performance of several regression models; models with small predicted residual sum of squares are preferable. The adjusted coefficient of determination was also used (R^2 adj). It provides a measure of the variability in the response variable explained by the model.

The relationship between the dough/biscuit properties and the constituent composition can be represented using a mathematical equation in which the x_i represents the fractional constrained proportion of the i^{th} ingredient, $0 \leq x_i \leq 1$, $i=1,2,\ldots, q$. In the current experiment, there are 3 ingredients, thus q=3. The sum of the components $\sum_{i=1}^{3} x_i = x_1 + x_2 + x_3$ must be 1 or equivalent to 100%.

Data analysis of the flour mixtures provides information on the blending properties of the constituents. They can be linear or nonlinear trends depending on whether the value of the response changes linearly or not, given the composition of the ingredient(s) used in the blend. Different models provide information on the shape of the blend properties. They are mathematical representations of the response surface over the region defined by the mixture blends. The Sheffe's canonical special cubic model was also fitted to the data as recommended by Cornell and Harrisson (1997), in situation when the shape of the response surface is uncertain. The following specific analyses were performed: (1) Predicted residual sum of squares (R^2 pred) or the predictive ability of a model. It leaves out one observation, fits the model to the data and measures the difference between the prediction and the actual experimental result. (2) Adjusted coefficient of determination (R^2 adj) or the measure of the variation in the response variable explained by the constituent(s) and their interactions. The adjusted R^2 has a correction for the loss of degree of freedom associated with adding another predictor in the regression. This implies a penalty term, so that the value of the adjusted R^2 will only rise if the increase in the penalty is more than outweighed by the rise in the value of R^2. (3) P lack of fit test (P LOF) or the measure of the adequacy of the model, which provides an independent estimate of the experimental error variance (derived from repeated experimental runs). The null hypothesis is that the model does not lack fit (i.e. fits the data well) and H_1 that the model lacks fit; P values smaller than α=0.05 lead to the rejection of H_0, therefore lack of fit. Multiple regression analysis (Proc Reg, SAS Institute, 2004) was used to fit the following models:

Model	**Equation**
Linear	$Y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3$
Quadratic	$Y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3$
Scheffe's cubic	$Y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3$

where Y or response is the predicted dependent variable (e.g. TPA parameters), β_1 s are the parameter estimates for each linear and cross constituent terms for the prediction models, where x1=gluten, x2= starch and x3= water-solubles. Because of the restriction of the mixture design, it was not possible to estimate the intercept of the lines.

Even if all ingredients are expressed as percentages, the ratio of the raw values of the soluble-fraction and starch, for instance, is about 1/30. Pseudo-components x'_i are essentially used to bring the different ingredients in the mixture on a comparable scale, by standardizing them as follows:

$$x'_i = \frac{x_i - L_i}{1 - L}, \text{ where } L = \sum_{i=1}^{3} L_i \text{ and } x'_1 + x'_2 + x'_3 = 1$$

Regression models for mixture data have no intercept and use pseudo-components as explanatory variables. Pseudo-components are a new set of coordinates used in a mixture regression model. They allow the comparison of regression coefficients between models, which is only directly interpretable for linear coefficients.

Triangular contour plots were constructed based on the generated regression models. This is a graphical technique for representing a 3-dimensional surface by plotting constant z slices, called contours, on a 2-dimensional format. Display of contours side-by-side allows the comparison of the models.

The Pearsons's correlation was performed using PROC CORR of the SAS system to determine the significant linear correlation between the quantitative continuous variables related to end quality parameters and the rheological properties of the doughs.

5- Results and discussion

5.1- Composition of the fractions isolated from the flour streams

The proportions of starch, gluten and water-soluble fractions isolated by fractionation from the patent, middle-cut and clear flour grades are presented in Table 3.2 of the chapter 3.
The biochemical composition of the gluten, water-solubles and starch fractions revealed that the protein content in the native patent, middle-cut and clear flours increased from 7.41, 9.65 and 12.7 %, respectively (Table 4.2). The lipid content of the patent, middle-cut and clear flours also increased from 1.26, 2.07 and 3.23%, respectively, and this is related to the extraction of the aleuronic layer adhering to the bran, as it contains high amounts of oil due to its contamination with germ fragments (Morrison and Hargin, 1981; Pomeranz, 1988). The lipids can play an important role on dough development as they can interact with the protein and starch matrix. The total pentosan content of the native flours, which is related to the quantity of cell walls present in the flour, rose from 0.99, 1.56 to 1.76% in the native patent, middle-cut and clear fractions, respectively. The soluble pentosans, on the other hand, varied from 0.33 to 0.40%, and represent approximately 33.0, 25.0 and 19.3% of the total pentosans, respectively. Thus the insoluble pentosans increased substantially from 0.66, 1.16 and 1.42% in the same respective flours. The protein content of the gluten fraction remained relatively constant among the different flour grades (71.8 to 68.1%) and the protein content of the WS fraction, representing mainly the soluble globulins and albumins, increased slightly from the inner to the peripheral layer of the endosperm (18.1 to 22.1% on dry basis). The crude starch protein content remained relatively low (<1.31%). The insoluble proteins represent the protein portion in flour that is unextractable with 50% 1-propanol, but is extractable with a solvent having a reducing agent. It represents the polymeric glutenins, referred as insoluble glutenin, which is reported to be an important factor related to rheology and functionality of wheat flour in bread-making, despite the relatively low amounts of this protein fraction present in the flour (Sapirstein and Johnson, 2000). The insoluble protein (insoluble glutenin) increased from 1.14, 1.48 to 1.91%, while the soluble protein representing mainly the albumins, globulins and also the gliadins by this

method also rose from 5.12, 6.08 and 7.36% in the native patent, middle-cut and clear flour respectively. Thus the residual protein, deduced from the total protein content minus the combined soluble and insoluble protein fractions increased substantially from 1.15, 2.09 and 3.93% in the three respective native flours. The insoluble protein accounted for 21.8, 17.8 and 17.7 % and the soluble protein 54.6, 56.3 and 63.7 of the total protein content of the gluten isolated from the native patent, middle-cut and clear flour materials, respectively. As expected, the WS fractions contain essentially soluble proteins.

The separation technique used to isolate gluten, water-solubles and crude starch fractions from flours with water as the only extractant, without the use of a non-polar solvent to extract the lipid, resulted in the binding of lipids to the gluten component (8.27, 8.31 and 11.2% on gluten d.b) and to a lower extent to the crude starch (~1.0% on starch basis). These observations are in agreement with the findings of Yamazaki and Donelson (1976) and MacRitchie (1985) who found that lipids are essentially bound to gluten, if they are not extracted by solvent prior to flour fractionation. Contrary to our expectations, the soluble pentosans of the WS fraction accounted for only 70 to 80% of the total pentosans. These low values suggested that aggregation phenomena could have occurred during the drying of the WS fraction that make resolubilization of this material difficult prior to analysis. The starch tailings of the patent, middle- cut and clear fractions had 5.06, 5.54 and 4.99% of the total pentosans, respectively, with 95% of them being insoluble pentosans.

5.2- Influence of the starch, gluten and water soluble fractions and their ratios on the rheological properties of the dough

Many aspects of biscuit processing and biscuit characteristics are closely related todough rheolgical behavior (Manohar and Rao, 1999). During the manufacture of semi-sweet biscuits, the doughs are sheeted in several reduction steps. As a result, the gluten network is aligned in the direction of the machining (Levine and Drew, 1994) and this process induces important stresses to the dough and to its elastic component. Several rheological tests were used to monitor the cookie rheogical behavior. Dough consistency (Miller, 1985) and the lubricated uniaxial compression method (Miller and Hoseney, 1997) have been used to predict cookie diameter. Biaxial extension properties are likely involved in dough shaping, sheeting, moulding and also in bubbles growth during oven rise (Bartolucci and Launay, 2000). Stress relaxation in the lubricated squeezing flow (LSF) method is often adopted in biscuit-making since it gives a good assessment of the viscoelastic properties of the dough which are important to predicting biscuit dimensions (Renard and Théry, 1998; Bartolucci and Launay, 2000).

Tables 4.3 a, b and c display the hardness, consistency (obtained by the two bytes tests) and elongational viscosity (obtained by simple compression) responses to the various blend combinations for the fractions obtained from the three flour grades. These rheological attributes increased gradually with the gluten concentration. Thus, at 25% gluten in the clear flour i.e. 0.25 glu (gluten) / 0.75 st (starch), these attributes increased by a factor of approximately two versus the patent (15%) and middle-cut (18.3%gluten) counterparts . For the same concentration of gluten, higher levels of the WS fraction decreased significantly the values of these parameters, especially with the clear flour (0.25 glu / 0.61 st / 0.14 ws (water soluble) vs 0.25 glu / 0.75 st). This indicates that the WS fraction due to its high water binding capacity contributes to the rheological characteristics of the doughs by inhibiting the development of gluten. Pentosans, being the most hygroscopic flour constituents, absorb more rapidly and extensively the water incorporated into the biscuit recipe than the other flour constituents (gluten and starch), and possibly inhibit proper development of the gluten network. Similar trends are observed with the Tla values, and inverse tendencies with the k values. The Tla refers to the time necessary to reduce the final compression force by half, once the compression is removed due to the elastic recovery of the dough, whilst the k value reflects the internal constraints accumulated in the dough or relaxation rate constant. The relaxation following lubricated squeezing flow has also been used as an indicator of biscuit dimensions (Renard and Théry, 1998; Bartolucci and Launay, 2000). Following sheeting, biscuit length will decrease only in the direction of the laminating direction and its width will increase, as a result of strain recovery. The faster the internal constraints accumulated in the biscuit dough relax (corresponding to a high k value), the lower the recovery. As a result, the biscuit length will be less reduced and the biscuit thickness will be lower. With an increase in k, the elastic energy stored in the dough dissipated more rapidly, the recovery is smaller and as a result, the final length of the biscuit will be greater. In contrast, the higher Tla and the lower k values of the clear flour blends (0.25glu / 0.68st / 0.07ws and 0.25glu / 0.75/st) confirm the market elastic recovery of their doughs. This suggests that flours with high protein content favor the development of a stronger gluten network, yielding a more elastic dough (higher Tla and lower k). At 25% gluten concentration, difficulties in dough handling were encountered during the lamination process due to the pronounced retraction of the dough. In this case, the dough was laminated three times instead of two, using 1 min resting period with the final 1.75 mm roll gap to minimize the problem of retraction.

Blends made with higher WS concentrations yielded more cohesive dough. Lower gluten levels, coupled with higher WS contents, induce the development of very cohesive doughs (0.03 glu / 0.91 st / 0.06 ws and 0.09 glu / 0.85 st / 0.06 ws equivalent to standard patent flour). Springiness (derived by the two-byte tests) increased by higher concentrations of WS among all the flour blends (0.03 glu / 0.91 st / 0.06 ws vs 0.03 glu / 0.94 st / 0.03ws and 0.03glu / 0.97st of patent). Even though the parameters Tla and springiness imply elasticity, they do not provide the same information, due to differences in the testing methodologies. It is also of interest to note that even at low concentrations of gluten (~2.1% protein for the 0.03 glu / 0.91 st / 0.06 ws mixture of the patente), machinable doughs were produced, which resisted to the sheeting stress and could also be layered without breakage.

The Aw measurements (Table 4.3a, b and c) revealed that without or with the regular amounts of the WS fraction found in the native flours, the water activity did not substantially change. However, by doubling the concentration of the WS fraction in the clear (14%), a marginal decline of the dough Aw value was noted (0.05 glu / 0.81 / 0.14 ws, 0.15 glu / 0.71 st / 0.14 ws and 0.25 glu / 0.61 st / 0.14 ws). This could be related to the higher concentration of pentosans present in the WS fraction that could enhance stronger water 'binding' in the respective dough systems.

5.3- Influence of the starch, gluten and water solubles fractions and their ratios on the biscuit characteristics

The biscuit moisture contents, ranging between 3.8-4.1 % (Aw of 0.230 ± 0.007) for all the flour blends, were controlled by monitoring carefully the biscuit dough moisture loss at ~ 20% during the baking process and cooling period of the biscuits on the baking tray. Tables 4.4 a ,b, c show that thickness of the biscuits changed marginally with the three flour fraction mixtures at lower gluten content, while a considerable thickness increase was noted with higher gluten and lower levels of the water soluble the clear flour. Higher gluten content contributes to rapid shrinking of the dough following lamination and cut-out. Doughs with smaller recovery (higher k) values resulted in thinner biscuits. Similar observations have been made by Bartolucci and Launay (2000). All the flour mixtures in which the protein content did not exceed 12.9% were easily sheeted and biscuit's thickness easily controlled. A lower density, commonly reported as a quality index for biscuits (Tharrault, 1994a,b), was attained with the patent and middle-cut flour blends, as density did not exceed 0.370g/cc. Biscuit density gradually rose from the patent, middle-cut to clear flour blends from an average of 0.309, 0.333 to 0.415g/cc, respectively, for the reconstituted mixtures of the parent flours; this is likely attributed to the higher gluten concentration of the clear flour, as evidenced by the higher density of the clear containing 25% gluten. The concentration of the WS fraction also appeared to influence biscuit density, as denser biscuits were generally obtained with lower WS fraction levels, especially when combined with higher gluten contents (0.25 glu / 0.61 st / 0.14 ws, 0.25 glu / 0.68 st / 0.07 ws and 0.25 glu / 0.75 st of the clear flour).

The mechanical properties of biscuits have a direct impact on the consumer perception of these products. Biscuit structure is made of a number of cavities, which form in the biscuits during baking as the leavening gases and water vapor are released. These cavities are irregular holes formed by the expansion of air pockets trapped in the dough during mixing. The walls of the cavities are formed from pieces of the endosperm linked together partly by contact with each other and partly with a sugar glass to give grains or group of grains, similar to bricks and cement in a wall (Burt and Fearn, 1984; Sharp, 2001). Penetrometry gives a good characterization of the textural attributes of biscuits since it is very sensitive to relatively small structural changes; in this case, textural measurements are made using a 10^{O} cone angle which produces the greatest penetration distance before fracture of the biscuits occurs. Analysis of the fracture pattern on the force-distance graph allows the quantification of several textural attributes (Goullieux et al., 1995; Maache Rezzoug et al., 1998b, c), namely firmness, the force of bonds between cavities or grains (Fs) and the number of grains or group of grains (No) in the biscuit internal structure.

At constant concentration of the WS fraction, biscuit firmness increased gradually with increased gluten level independently of the flour grades. Moreover, the biscuits prepared with blends of the clear fractions were significantly harder than the patent and middle-cut flour mixtures. These results are in agreement with the work of Kiger and Kiger (1967), as the biscuit firmness is reported to increase with protein content, especially above 10%, due to gluten elasticity which causes retraction of the dough on sheeting and oven rise resulting in smaller biscuits. It is also noteworthy that the biscuits made with the higher concentration of WS (14%) and the low concentration of gluten (5%) in the 0.05 glu / 0.81 st / 0.14 ws blend of the clear mixtures produced unacceptable biscuit firmness (~ 104 N); probably a «case hardening» situation due to of sugar concentration level in the water soluble fraction. In general, regardless of the fraction combinations in the blends (flour grades and ratio of the isolated fractions), the addition of WS fraction raised the biscuit firmness, suggesting a synergistic influence of both the gluten and the WS. The specific tearing force (Fs), which reflects the strength of the interparticle interactions between cavities (grains) and/or group of grains in the biscuit structure, remained relatively constant among the different flour mixtures, although with higher concentrations of WS and gluten this force rose marginally. The spatial frequency (No) or the number of grains or group of grains encountered per unit distance of cone penetration indicated that biscuits with lower gluten concentration contained more grains or group of grains in the internal structure, whereas the addition of the WS fraction contributed to higher No values. Lower Fs/No ratios that are related to friability or biscuit's crunchiness (Maache Rezzoug et al., 1998) were obtained with flour fraction mixtures containing lower levels of gluten (Fs/No ratio inferior to 0.110). At gluten concentrations exceeding 15%, coupled with low levels of the WS fraction, a significant loss of crunchiness was observed, as evidenced by the higher Fs/No ratios (Tables 4a, b and c). In parallel, denser biscuits were observed from these blend combinations, implying that denser biscuits are less crunchy. These data further suggest that the textural attributes of biscuits could be controlled by the judicious combination of the WS and the gluten fractions incorporated into flour mixtures.

The biscuit color expressed by the Hunterlab Tristimulus Lightness or L-value for the various combinations of WS, gluten and starch fractions is also presented in Tables 4a, b and c. The WS fraction was the principal fraction controlling the lightness parameter. Without incorporation of the WS coupled with higher starch levels, the biscuit's surface appeared whiter, i.e., higher L values (patent 0.03 glu / 0.97 st and 0.09 glu / 0.91 st and the middle-cut blend 0.03 glu / 0.97 st). Additionally, an increase of the gluten concentration coupled with higher WS fraction (clear flour blends 0.15 glu / 0.71 st / 0.14 ws, 0.25 glu / 0.61 st / 0.14 ws) produced darker biscuits. The higher degree of the Maillard reactions usually reflects higher amounts of free amino acids and carbonyls groups of reducing sugars (Camire et al., 1990). High concentrations of soluble proteins, pentosans and low molecular weight compounds present in the WS fraction (sugars) as well as the hydrolysis of starch and non-reducing sugars (e.g. sucrose) can contribute to a darker biscuit color (Feather, 1994).

5.4- Regression models and prediction of properties of flour mixtures on the dough rheological behavior and biscuit characteristics

Many literature reports indicated that wheat flour functionality cannot be described by simple summation of individual constituent effects (Köehler et al., 1999; Graßberger et al., 2003). Although gluten, water-solubles and starch fractions of the flour have their own specific biochemical characteristics, the flour technological value depends primarily on the structure/function relations of these constituents, their concentration as well as on numerous interactions among them which occur during dough development. Such effects are important determinants of the overall dough rheological behavior and this in turn influences the dimensional and textural characteristics of the end-product. In this context, a specific attribute such as dough consistency of native or reconstituted flour blends cannot be predicted by a simple additive approach; instead, equations need to be developed to include complex interactions involving flour constituents like gluten, water-solubles and starch fractions isolated from different flour grades to better predict the consistency of the composite mixtures made of these fractions.

The proportions of the fractions in each flour mixture were determined using a mixture experimental design with multiple constraints on the concentrations of the three isolated fractions. This mixture experimental design corresponds to a specific response surface methodology in which the independent variables are the isolated fractions and the response or the dependent variable becomes a function of the blend composition (Cornell and Harrisson, 1997). For instance, for the patent, the variation in the percentage of the water-soluble fraction is 6% (0-6%), versus 12% (3-15%) for the gluten and 18% for the starch (79-97%). The fractional composition of the blends under study also varied significantly as shown in Table 1, e.g. 6.0, 9.0% and 85% for the water-solubles, gluten and starch, respectively, for the native patent flour. In this experimental design, the gluten level was increased or decreased by approximately 66% from the initial values found in the three flour streams blends, so the overall protein content of the flour blend across all the flour grades varied from as low as 2.1 % to a high 16.9%; such a variation in the protein content can be considered as rather extreme for Canadian soft wheat flours. In contrast, the water-solubles fraction could not be largely increased in the patent and middle-cut fraction blends as it altered severely the dough stickiness and the lamination process was not feasible. In contrast, for the clear fraction mixtures the WS concentration could be increased by 50% of its original content, without the stickiness problem, likely due to the fact that the higher gluten levels overcome the addition of the WS fraction. Finally, the starch content was balanced accordingly to ensure that all flour fraction combinations were adjusted to 100%. Regression analyses were carried out to establish the relationships between the rheological properties of the doughs and the biscuit characteristics and the composition of the blends obtained using the isolated fractions of the three flour grades. Quadratic as well as special cubic terms, i.e. «non-linear» terms were significant ($P<0.05$) in many of the regression models derived (Table 4.5 a, b, c), implying that the interactions between the flour fractions were important. In these models, positive values of partial regression coefficients for the non-linear terms indicate a constituent synergistic effect in the binary or ternary blends, while negative values suggest antagonistic effects. For example, in the model for cohesiveness of the patent flour the positive non-linear terms indicated that interactions between starch and water-solubles (x_2x_3 of 3.28) as well as between gluten and water-solubles (x_1x_3 of 2.97) result in higher cohesiveness of the dough than expected in the absence of interactions (linear terms). In contrast, the negative non-linear terms in the model for dough hardness of the same flour indicated that interactions between gluten and water-solubles (x_1x_3 of -19.55), and between starch and water-solubles (x_2x_3 of -17.44), suggest that the effect of the water soluble fraction on the dough hardness is not the same for different values of starch. The adequacy of each model was verified by the lack of fit test and the R^2 of prediction. With the lack of fit test, the analysis of variance showed that these models in the patent, with the exception of biscuit volume and thickness, appeared to be adequate with no significant lack of fit ($P>0.05$) and with a satisfactory R^2 (pred) among the three flour grades used for comparison purposes. The values of the R^2 (pred) and P (LOF) of the biscuit properties were not as adequate as for the dough rheological parameters, i.e., lower R^2 (pred). This could be explained by the variation in the biscuits dimensional properties which sometimes are difficult to control under bench top conditions at low speed of production, due to the retraction of the dough during the sheeting, cutting and baking processes which may alter significantly these properties, especially when higher gluten concentrations are used in the biscuit formulation. Furthermore, when different equations of prediction are generated, i.e. linear, cubic, and quadratic models, it is difficult to ascertain the influence of each fraction from different flour grades and their interactive effects on the rheological behavior of the dough and the biscuit quality attributes, except if the same regression model is employed for comparison purposes; i.e. a quadratic model for predicting dough cohesiveness and biscuits specific tearing force for all the 3 flour grades One must also recognize that the relationship between factors and responses are very complex and could also be affected by factors such the ability to control water 'binding' and water retention during baking which depend upon the concentration of the pentosans, proteins, damaged starch, the granulometry differences in each fraction mixture, the glass transition phenomena (plasticization behavior of constituents by water and sugars) and the dough viscosity as well as upon some non-identified genetic factors, etc.

5.5- Contour plots for dough rheological parameters and biscuit quality attributes

In order to compare the effects of the isolated flour fractions in the blends, contour plots were generated by the SAS program with the approach of transforming all the factors in such a manner to bring them to the same scale. This was carried out by using the pseudo-components plot analysis. Such plots were generated using the «mixture» models, i.e., the non-linear regression models which identified the mixtures or fraction blends that are expected to produce an estimated yield value within some given interval of values (Tables 4.5 a, b, c). These equations are graphically presented in a ternary diagram and the area of experiment (compositional range) is limited into a parallelogram defined by the experimental plan for each flour grade. Only the independent variables which were found significant at $P<0.05$ with acceptable (R^2 pred) in the models were retained for the contour graphs of the flour grade mixture design. The contour of response surface for dough's consistency showed increasing consistency values from 10 N.s for the patent to 50 N.s for the clear flour, due to the increasing gluten concentration from the inner to outside layer of the endosperm (Figure 4.1). The patent flour presented a maximum towards the gluten-starch edges and was mainly influenced by the interaction of both the WS and gluten since the addition of gluten induced higher consistency, whereas the addition of the WS fraction decreased it, probably due to their competition for water. The contour of the middle-cut and clear flour grades showed a maximum region parallel to the WS fraction, re-confirming that lower levels of the water-solubles fraction raise substantially the dough consistency. This attribute is also subject to an antagonist influence of the significant negative interaction for all the binary terms of the different components. This may suggest that the protein quality of the patent can also have an influence on the dough behavior, in comparison to the blends made from fractions of the mid-cut and the clear flour grades, since the patent had the lower protein content. By tracing a perpendicular line to the angle formed by the higher starch content, this line is almost parallel to the consistency ellipses of the three flour grades mixtures combinations, suggesting that starch has a relatively minor effect on dough consistency. This observation is in agreement with the works of Lineback and Wongsrikasen (1980) and Wade (1995) who found that starch is mainly acting as filler in the cookie dough, with its granules occupying voids within the continuous structural network of the proteins and additionally starch gelatinization is limited in cookie/biscuit systems (Varriano Marston et al., 1980). The contour plots for dough hardness and elongational viscosity, despite of the differences in the mechanical tests employed for analysis, exhibited similar patterns to those of consistency (graphs not shown). This is further evidenced by the Pearson correlation coefficient matrix (Table 4.6) describing the relationships between these three attributes for the three types of flour grade mixture combinations tested (n=15); the rheological attributes were highly correlated ($r>0.90$, $p<0.05$). The attribute of cohesiveness that reflects the structural strength of the dough network was also influenced by the WS fraction; e.g. a higher concentration of this fraction resulted in increasing cohesiveness for the patent, middle-cut and clear flour blends. The dough Tla increased from patent, middle-cut and clear flour by 0.4, 0.6 and 1.4 s, respectively, and seemed to be mainly related to gluten content levels. As anticipated, a higher gluten concentration induced an increment of elasticity to the biscuit's dough. The linear independent variables showed that gluten has an impact on the Tla, i.e. x_1 of 0.578, 0.909, 3.02 for the patent, mid-cut and clear, respectively; also, significant interaction effects between gluten and the water-solubles fraction were observed (x_1x_3 of 0.412, -0.698, -4.999 for the different blends of the three flour grades). The Tla contours of the response surface plots shown in Figure 4.2 are parallel and facing the gluten, confirming that the gluten fraction exhibits almost a linear effect; i.e. with an increase in gluten content there is an increase of Tla, whereas inverse trends are observed with the k parameter.

Biscuit density of the patent flour blends is affected positively by the interactions of gluten-starch and starch-water solubles, but negatively by the cubic terms. The contour plots of Figure 4.3 revealed that increasing the contents of the WS fraction in the mixtures lowered the biscuit density. However, for the middle-cut fraction blends, the curves tended to be closer with decreasing the WS and increasing the gluten fractions, implying a synergy between these two. The higher densities obtained with the fraction blends of the clear flour appear to be related to the compositional parameters of this flour grade; i.e the higher protein, pentosans and some damaged starch contribute to the enhancement of water "binding" properties of these materials. The predicted firmness parameters although not very accurate (low R^2 pred) could give an estimate of how much the gluten and WS fractions will alter this attribute; high WS levels resulted in hard biscuits. The mean tearing force contour plots in Figure 4.3 revealed that higher gluten contents slightly increased the tearing force (Fs) with both the patent and middle-cut fraction blends. In the case of the clear flour blends, there was a large raise of the Fs with increasing levels of the WS fraction. This may imply that with the higher sorption capacity of the WS constituents, especially at higher concentrations, when coupled with high gluten content of the clear grade, there is a major impact on the textural properties of the biscuits. Thus, it is important that a certain ratio of gluten/WS should be kept in the formulation of a biscuit recipe in order to obtain products of acceptable organoleptic properties; e.g, a ratio of 5/81/14 for gluten/starch/WS gives unacceptable textural properties. Finally, the contour plots for the spatial ruptures (No) showed a minimum region parallel to the starch/gluten axis, suggesting that the WS fraction plays a critical role in the creation of cavities in biscuit internal structure; higher WS levels tend to raise the number of grains, whereas there is a decrease in No with increasing higher gluten concentration, regardless of the flour grade.

5.6- Role of the flour constituents on the rheological attributes of the dough and biscuit quality parameters

By quantifying the amounts of soluble and insoluble proteins, pentosans and lipids found in various blends of flour fractions isolated from each flour grade, the influence of these constituents on dough rheology and biscuit textural parameters can be assessed. The matrix of correlation coefficients describing the linear relationships between flour major constituents and dough rheological attributes are shown in Table 4.7. Dough elongational viscosity, consistency and hardness are highly (negatively) correlated with the total pentosans of the clear flour (r= -0.944, -0.909 and -0.889, respectively) and to a lesser extent to the middle-cut flour. This might be related to the high water retention capacity of the fractions rich in pentosans or the starch tailing sub-fraction. In a biscuit dough, where water is incorporated at low levels in the recipe, it is possible that the more hygrospopic components such as the pentosans (10g water/g of pentosans) will absorb more rapidly and extensively the water than those components with lower binding capacity (e.g. gluten 2.8g water/g; damaged starch 1.5g water/g) and therefore largely influence the rheological behavior of the dough (Bloksma and Bushuk, 1971).

In contrast, dough hardness and consistency of the patent flour were positively related to the total protein of the patent. This positive correlation decreased abruptly from the patent, middle-cut and clear flours, respectively. A possible explanation is that protein quality of the inner endosperm and not the amount of protein itself largely affects end-product quality, as suggested by Abboud and al. (1985). The bulk of flour lipids, mainly bound to gluten, showed similar as the protein component, i.e., with the patent flour blends significant relationships between lipids and the consistency and hardness of the doughs were identified. The latter might suggest a synergistic effect between the two components (proteins and lipids) that becomes important during dough development, originating from hydrophobic interactions.

The cohesiveness of the clear flour blends, on the other hand, was positively correlated to the total, soluble and insoluble protein ($r= 0.789, 0.834, 0.866, p\leq 0.001$, respectively), implying that the higher concentration of the external parts of the endosperm which are more hydrophilic could favor the formation of stronger internal bonds responsible of the dough structure. Moreover, the total protein as well as the soluble and insoluble proteins was positively correlated with the Tla. As expected, inverse correlations were obtained between the three protein fractions and the k parameter.

Biscuit density and thickness demonstrated low and inconsistent correlation coefficients with the flour constituents. However, the density of the middle-cut flour fraction combinations showed strong correlations with total protein ($r=0.725$) and lipids ($r=0.797$) and a negative correlation with insoluble pentosans ($r= -0.891$) (Table 4.8). This is in agreement with the findings of Jeltama et al. (1983) who reported that the insoluble pentosan content is correlated with crunchiness, which in turn is related to lower biscuit density. Biscuits made with either the patent or the middle-cut flour blends exhibited positive correlations between firmness and the total, soluble and insoluble proteins, whereas the firmness of biscuits made with the clear flour fractions were correlated to a lower extent to the pentosans. Total pentosans and especially their insoluble fraction were highly correlated with the number of grain or group of grains per unit of penetration or the spatial frequency (No) for all three flour grades (Table 4.8). In parallel, the specific tearing force (Fs) is correlated positively with the total and insoluble pentosans of the clear flour ($r=0.747$ and 0.756, $p\leq0.01$), implying that the pentosans could foster stronger binding among the grain particles in the product after baking and thereby increase its overall firmness.

6- Conclusions

There are distinct compositional and functional differences among the native patent, middle-cut and clear flours as well as among the isolated fractions of gluten, starch and water-solubles derived from these flours by a wet fractionation protocol. Such differences in constituent concentration and functionality of each fraction largely affect the rheological behavior of the dough and the biscuit baking performance. Interaction effects among starch, gluten and water-solubles fractions are also significant and seem to influence the rheological properties of the dough and the quality attributes of biscuits made of the three flour grades. Neglecting such interactions could result in significant deviations of the expected responses in the mixtures. For such multicomponent flour blends, neither the rheological parameters of the dough (consistency, half relaxation time, elongational viscosity) nor the biscuit characteristics (density, mean tearing forces, spatial frequency) could be assumed to be weighted averages of those characteristics of the individual constituents; instead, mathematical models incorporating interaction terms can yield reliable regression equations to describe the dough and biscuit characteristics as a function of the flour blend composition.

This study demonstrated the potential of using the various wheat endosperm portions of the wheat grain in biscuit making to achieve appropriate dimensional and textural properties of the end-product. It also confirmed that an appropriate fractionation/reconstitution procedure to isolate some flour sub-fractions could be a viable means to develop new biscuit prototypes of low gluten contents by better exploiting the potential of other major flour constituents, especially the pentosans in order to control the dimensional and textural attributes of the biscuits. Similar to proteins that are often reported to alter significantly the dough and biscuit properties (higher protein content flours produce denser, firmer and less crunchy biscuits), pentosans can also affect these properties and assist in developing products with acceptable quality.

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Table 4.1. Experimental design with three independent variables for the three flour grades (actual composition of the mixtures on d.b.)

Flour Grades	Recipes no.	Fractions proportions		
		X_1	X_2	X_3
Patent	1 *	0.030	0.910	0.060
	2	0.030	0.940	0.030
	3 *	0.030	0.970	0.000
	4 * [a]	0.090	0.850	0.060
	5 *	0.090	0.880	0.030
	6 *	0.090	0.910	0.000
	7 *	0.150	0.790	0.060
	8	0.150	0.820	0.030
	9 *	0.150	0.850	0.000
Middle-cut	1 *	0.037	0.899	0.064
	2	0.037	0.931	0.032
	3 *	0.037	0.960	0.000
	4 [a]	0.110	0.826	0.064
	5 *	0.110	0.858	0.032
	6	0.110	0.890	0.000
	7 *	0.183	0.753	0.064
	8 *	0.183	0.785	0.032
	9 *	0.183	0.817	0.000
Clear	1 *	0.050	0.810	0.140
	2	0.050	0.880	0.070
	3 *	0.050	0.950	0.000
	4	0.150	0.710	0.140
	5 * [a]	0.150	0.780	0.070
	6	0.150	0.850	0.000
	7 *	0.250	0.610	0.140
	8 *	0.250	0.680	0.070
	9 *	0.250	0.750	0.000

X1: Gluten **X2: Starch** **X3: Water-solubles**

[a] Weight proportions equivalent to the original flour
Recipes were carried out in a random order
* Duplicate runs

Table 4.2. Proportion and composition of the freeze-dried fractions isolated from the respective flours grades (g/100g d.b.)

Grades and Fractions	Total protein (%)	Soluble protein (%)	Insoluble [d] protein (%)	Total [e] pentosans (%)	Soluble pentosans (%)	Lipids (%)
Patent	7.41	5.12	1.14	0.99 [c]	0.33	1.26
Crude starch [a]	0.26	-	-	0.45	0.06	1.13
Gluten	71.8	39.2	15.7	-	-	8.27
Water solubles	18.1	17.6	3.38	10.2	8.16	-
Middle-cut	9.65	6.08	1.48	1.56	0.40	2.07
Crude starch	0.45	-	-	1.05	0.10	0.99
Gluten	71.2	40.1	12.7	-	-	8.31
Water solubles	18.3	16.8	3.73	11.1	8.87	-
Clear	12.7	7.36	1.91	1.76	0.34	3.23
Crude starch	1.31	-	-	1.37	0.11	1.10
Gluten	68.1	43.4	12.1	-	-	11.2
Water solubles	22.1	19.5	4.56	6.94	4.79	-

[a] Starch tailings constitute 6.8, 13.4 and 18.9.% and prime starch 79.9, 68.9 and 56.9 % of the crude starch content values for the patent, middle-cut and clear, respectively. The total pentosan content of the respective starch tailings was 5.06, 5.54 and 4.89% (with more than 95% being soluble pentosans)

[c] Total protein: Soluble + insoluble + residual

[d] Insoluble protein referred to insoluble glutenin

[e] Total pentosans consist of both soluble and insoluble pentosans

Ash content: 0.41, 0.57, and 0.99 % and damaged starch: 3.6, 3.8 and 4.9% for the patent, middle-cut and clear, respectively.

Table 4.3a. Influence of the flour fraction combinations on the dough rheological properties and Aw for the patent flour

Fractions (Patent)	Hardn (N)	Spring.	Cohes.	Consist. (N.s)	Elong. Visc. 10^{-5} * (Pa.s)	Tla (s)	k (s^{-1})	Aw
.03/.91/.06	2.43	0.891	0.405	8.33	0.182	0.222	5.05	0.880
.03/.91/.06	2.11	0.872	0.378	8.62	0.169	0.231	4.46	0.883
.03/.94/.03	2.41	0.821	0.400	10.8	0.185	0.290	6.45	0.894
.03/.97	2.94	0.511	0.347	14.0	0.339	0.320	6.99	0.895
.03/.97	2.83	0.560	0.312	13.7	0.319	0.321	6.58	0.898
.09/.85/.06*	2.85	0.735	0.411	12.2	0.219	0.307	5.17	0.893
.09/.85/.06*	2.59	0.749	0.423	12.3	0.209	0.304	5.02	0.893
.09/.88/.03	2.26	0.544	0.325	12.3	0.188	0.347	5.46	0.898
.09/.88/.03	2.45	0.555	0.314	11.3	0.163	0.340	5.13	0.896
.09/.91	3.30	0.342	0.234	16.1	0.352	0.368	4.83	0.888
.09/.91	2.75	0.400	0.276	14.5	0.330	0.342	4.44	0.886
.15/.79/.06	3.52	0.475	0.303	15.9	0.314	0.432	3.63	0.880
.15/.79/.06	3.63	0.500	0.325	16.4	0.297	0.433	3.45	0.881
.15/.82/.03	3.04	0.526	0.351	16.9	0.309	0.431	4.19	0.894
.15/.85	3.98	0.382	0.273	19.7	0.439	0.450	3.25	0.885
.15/.85	4.01	0.371	0.266	19.9	0.418	0.448	2.95	0.886

Hard, hardness; Spring, springiness; Cohes, cohesiveness; Consist, consistency; Elong.visc., elongational viscosity; Tla, half relaxation time; k, relaxation rate constant

X1: gluten X2: Starch X3: Water soluble

* equivalent to the native flour

Table 4.3b. Influence of the flour fraction combinations on the dough rheological properties and Aw for the middle-cut flour

Fractions (middle-cut)	Hardn (N)	Spring.	Cohes.	Consist. (N.s)	Elong. Visc 10^{-5} * (Pa.s)	Tla (s)	k (s^{-1})	Aw
.037/.899/.064	1.65	0.514	0.379	7.84	0.199	0.288	7.25	0.886
.037/.899/.064	1.64	0.513	0.367	8.61	0.179	0.274	6.89	0.885
.037/.931/.032	2.85	0.511	0.311	15.7	0.297	0.327	7.07	0.889
.037/.96	3.45	0.437	0.281	21.5	0.386	0.383	7.82	0.903
.037/.97	3.63	0.428	0.296	20.1	0.347	0.364	7.35	0.905
.11/.826/.064*	2.73	0.430	0.322	11.8	0.210	0.410	5.14	0.889
.11/.856/.032	3.09	0.420	0.310	16.7	0.271	0.437	5.22	0.895
.11/.856/.032	3.18	0.430	0.298	17.0	0.279	0.428	4.91	0.902
.11/.89	3.84	0.343	0.257	23.1	0.358	0.432	5.76	0.906
.183/.753/.064	2.69	0.483	0.358	15.6	0.307	0.537	3.71	0.895
.183/.753/.064	2.40	0.490	0.365	15.7	0.283	0.510	3.45	0.896
.183/.785/.032	4.79	0.438	0.334	25.5	0.488	0.628	3.32	0.899
.183/.785/.032	4.78	0.442	0.331	26.6	0.463	0.579	3.16	0.901
.183/.817	4.91	0.404	0.321	27.5	0.523	0.643	3.65	0.908
.183/.817	5.00	0.412	0.325	26.7	0.497	0.598	3.48	0.905

Hardn, hardness; Spring, springiness; Cohes, cohesiveness; Consist, consistency; Elong.visc., elongational viscosity; Tla, half relaxation time; k, relaxation rate constant

X1:gluten X2: Starch X3:Water soluble

* equivalent to the native flour

Table 4.3c. Influence of the flour fraction combinations on the dough rheological properties and Aw for the clear flour

Fractions (clear)	Hard (N)	Spring.	Cohes.	Consist. (N.s)	Elong. Visc 10^{-5} * (Pa.s)	Tla (s)	k (s^{-1})	Aw
.05/.81/.14	2.91	0.583	0.356	15.2	0.255	0.424	4.62	0.844
.05/.81/.14	2.99	0.609	0.366	14.2	0.267	0.456	4.52	0.847
.05/.88/.07	4.81	0.381	0.248	16.8	0.449	0.500	5.30	0.881
.05/.95	9.56	0.267	0.201	52.6	0.892	0.561	4.91	0.896
.05/.95	9.57	0.268	0.202	54.2	0.981	0.560	4.24	0.894
.15/.71/.14	2.73	0.538	0.389	14.1	0.310	0.480	3.78	0.854
.15/.78/.07*	3.72	0.434	0.328	20.9	0.439	0.633	3.92	0.889
.15/.78/.07*	3.41	0.432	0.315	18.8	0.458	0.570	3.73	0.887
.15/.78	7.50	0.344	0.287	42.2	0.808	0.778	3.77	0.891
.25/.61/.14	4.85	0.571	0.443	25.9	0.385	0.880	2.66	0.865
.25/.61/.14	4.81	0.541	0.454	25.0	0.346	0.853	2.65	0.868
.25/.68/.07	7.04	0.436	0.351	42.3	0.678	1.11	2.67	0.886
.25/.68/.07	7.16	0.410	0.367	41.6	0.612	1.12	2.57	0.887
.25/.75	9.58	0.393	0.351	60.9	1.16	1.45	2.19	0.893
.25/.75	9.71	0.386	0.352	58.6	1.04	1.47	2.21	0.895

Hardn, hardness; Spring, springiness; Cohes, cohesiveness; Consist, consistency; Elong.visc., elongational viscosity; Tla, half relaxation time; k, relaxation rate constant

X1:gluten X2: Starch X3:Water soluble

* equivalent to the native flour

Table 4.4a. Effect of the flour fraction combinations on biscuit characteristics for the patent

Fractions (Patent)	Dens (g/cc)	Thick. (mm)	Firm. (N)	No (mm^{-1})	Fs (N)	Fs/No (N/mm)	Hunter L
.03/.91/.06	0.308	9.66	9.72	12.9	1.12	0.087	66.63
.03/.91/.06	0.319	9.44	9.54	12.7	1.19	0.094	65.69
.03/.94/.03	0.343	9.52	8.99	10.5	1.01	0.096	79.51
.03/.97	0.339	8.54	9.41	10.1	0.82	0.082	89.51
.03/.97	0.334	8.32	9.21	9.62	0.86	0.089	87.59
.09/.85/.06*	0.301	8.42	10.5	11.9	1.18	0.099	66.78
.09/.85/.06*	0.317	9.02	10.7	11.7	1.13	0.966	66.75
.09/.88/.03	0.338	8.76	8.09	10.1	1.12	0.111	78.35
.09/.88/.03	0.348	8.63	8.38	10.6	1.03	0.097	81.46
.09/.91	0.348	8.98	7.63	9.68	1.04	0.107	84.26
.09/.91	0.351	10.02	7.94	9.86	1.08	0.111	82.65
.15/.79/.06	0.351	9.85	12.9	10.7	1.19	0.111	66.14
.15/.79/.06	0.354	9.28	12.4	10.9	1.17	0.107	68.19
.15/.82/.03	0.348	8.78	11.5	9.51	1.01	0.101	75.27
.15/.85	0.349	11.1	9.47	8.38	1.26	0.151	78.65
.15/.85	0.337	11.2	9.83	7.42	1.26	0.169	76.49

Den, density., Thick, thickness., Firm, firmness., No, spatial frequency., Fs, Mean tearing force., Fs/No, Friability's effort

X1: gluten X2: Starch X3: Water soluble

* equivalent to the native flour

Table 4.4b. Effect of the flour fraction combinations on biscuit characteristics for the middle –cut flour

Fractions (middle-cut)	Den (g/cc)	Thick. (mm)	Firm. (N)	No (mm^{-1})	Fs (N)	Fs/No (N/mm)	Hunter L
.037/.899/.064	0.306	8.81	10.3	12.2	1.16	0.095	74.56
.037/.899/.064	0.308	8.69	10.3	11.9	1.19	0.100	73.45
.037/.931/.032	0.315	9.23	9.76	12.2	1.10	0.090	78.51
.037/.96	0.319	9.15	8.28	10.9	1.14	0.100	88.16
.037/.97	0.322	9.36	8.68	10.8	1.05	0.097	87.09
.11/.826/.064*	0.333	8.36	16.7	12.4	1.15	0.093	64.7
.11/.856/.032	0.318	8.96	13.8	11.4	1.02	0.089	75.86
.11/.856/.032	0.311	8.90	12.3	10.3	1.10	0.107	76.51
.11/.89	0.342	9.04	11.3	9.31	1.04	0.111	79.51
.183/.753/.064	0.340	9.15	20.4	10.3	1.34	0.130	64.09
.183/.753/.064	0.334	8.97	19.5	10.4	1.35	0.128	62.55
.183/.785/.032	0.349	9.75	16.6	10.3	1.29	0.125	66.80
.183/.785/.032	0.357	9.96	16.4	9.91	1.34	0.135	68.10
.183/.817	0.370	11.3	16.1	7.38	1.12	0.155	76.12
.183/.817	0.368	10.9	15.4	6.98	1.16	0.166	74.79

Den, density., Thick, thickness., Firm, firmness., No, spatial frequency., Fs, Mean tearing force., Fs/No, Friability's effort

X1: gluten X2: Starch X3: Water soluble

* equivalent to the native flour

Table 4. 4c: Effect of the flour fraction combinations on biscuit characteristics for the clear

Fractions (clear)	Den (g/cc)	Thick. (mm)	Firm. (N)	No (mm^{-1})	Fs (N)	Fs/No (N.mm)	Hunter L
.05/.81/.14	0.418	8.43	105.5	17.0	8.99	0.527	64.71
.05/.81/.14	0.414	8.39	103.2	15.3	9.82	0.642	65.45
.05/.88/.07	0.391	8.92	33.8	13.8	1.56	0.113	69.45
.05/.95	0.423	8.68	28.3	13.1	1.13	0.086	78.20
.05/.95	0.419	9.13	26.8	10.4	1.12	0.108	80.49
.15/.71/.14	0.390	9.21	27.6	13.1	1.50	0.115	48.42
.15/.78/.07*	0.403	8.69	23.5	9.48	1.11	0.117	67.12
.15/.78/.07*	0.427	8.45	24.1	8.91	1.28	0.143	66.66
.15/.78	0.389	9.69	21.9	7.11	0.95	0.134	73.44
.25/.61/.14	0.384	9.89	28.8	12.1	1.55	0.128	47.74
.25/.61/.14	0.379	10.0	30.1	11.8	1.60	0.136	49.58
.25/.68/.07	0.429	10.4	23.6	8.88	1.48	0.160	55.14
.25/.68/.07	0.414	10.5	23.5	8.64	1.39	0.160	53.73
.25/.75	0.439	11.5	24.0	7.16	1.24	0.173	68.89
.25/.75	0.448	11.6	23.0	7.44	1.29	0.173	67.60

Den, density., Thick, thickness., Firm, firmness., No, spatial frequency., Fs, Mean tearing force., Fs/No, Friability's effort

X1: gluten X2: Starch X3: Water soluble

* equivalent to the native flour

Table 4.5a. Predictive regression models describing the relationships between rheological attributes of the dough and biscuit characteristics with the mixture composition (Pseudo components) of the patent flour

Dependent variables	Independent variables			Nonlinear blending terms				R^2(pred)	R^2 (adj)*	P(LOF)
	x1	x2	x3	x1x2	x1x3	x2x3	x1x2x3			
Hardness	**5.393**	**2.916**	**12.928**	**-2.962**	**-19.550**	**-17.449**		0.7999	0.9952	0.7574
Springiness	**0.598**	**0.549**	1.060	**-0.936**	-1.160	0.826	**3.223**	0.9001	0.9952	0.9849
Cohesiveness	**0.312**	**0.320**	**-1.690**	-0.225	**2.974**	**3.282**		0.5727	0.9921	0.9899
Consistency	**25.947**	**13.795**	**29.946**	**-10.230**	**-48.666**	**-46.184**		0.9378	0.9977	0.2485
Tla	**0.578**	**0.321**	-0.141	**-0.215**	**0.412**	0.278		0.9753	0.9994	0.9982
k	2.212	**6.839**	**-7.207**	-3.080	**20.159**	**12.071**	30.672	0.9070	0.9972	0.7164
n	**0.404**	**0.301**	**1.174**	-0.079	**-1.497**	**-0.892**		0.9228	0.9996	0.9990
Elong.visc	0.612	0.330	1.814	-0.389	-3.152	-2.899		0.9828	0.9970	0.9951
Volume	**36.715**	**30.416**	**128.038**	5.923	**-159.389**	**-114.776**	**-98.397**	0.5694	0.9980	0.0207
Density	**0.312**	**0.336**	**-0.206**	**0.100**	**0.956**	**0.710**	**-0.917**	0.7431	0.9997	0.9989
Thickness	**76.023**	**43.263**	**198.313**	-26.775	**-332.325**	**-198.900**		0.5043	0.9948	0.0004
Firmness	**12.617**	**6.063**	**25.969**	-2.246	-24.732	-14.445		0.7588	0.9952	0.4180
Spatial.rupt	**7.392**	**10.038**	**17.898**					0.8665	0.9981	0.4813
Tear. Force	**1.213**	**0.844**	**3.550**	0.579	**-3.850**	-2.753		0.5186	0.9959	0.9519

X1, X2, X3 refer to the proportions of gluten, starch and water-solubles fraction, respectively, in the formulation
In bold: significance at a probability level of α =0.05
R^2 (adj) *:1-((n-1)/ (n-p))*SSE/ SSTO, where n= number of observations used to fit the model, p=number of parameters in the regression model, SSE is the error or residual of squares (including lack of fit and pure error sum of squares), SSTO is the total sum of square which is a measure of overall variability in the response variable Y

Table 4.5b. Predictive regression models describing the relationships between rheological attributes of the dough and biscuit characteristics with the mixture composition (Pseudo components) of the middle-cut flour

Dependent variables	Independent variables			Nonlinear blending terms				R^2(pred)	R^2 (adj)	P(LOF)
	x1	x2	x3	x1x2	x1x3	x2x3	x1x2x3			
Hardness	**5.695**	**3.602**	**-2.771**	0.102				0.8115	0.9854	0.8015
Springiness	**0.546**	**0.430**	0.134	**-0.508**	0.294	**0.814**		0.8570	0.9990	0.9990
Cohesiveness	**0.428**	**0.283**	**0.659**	**-0.283**	**-0.638**	-0.164		0.8130	0.9989	0.9976
Consistency	**37.097**	**20.510**	**-83.965**	**-18.057**	**78.987**	**91.674**		0.9146	0.9945	0.0513
Tla	**0.909**	**0.374**	0.144	**-0.543**	**-0.698**	-0.078	**1.949**	0.9616	0.9985	0.9980
k	**1.429**	**7.511**	**13.121**	1.009	-6.846	-10.621	-8.909	0.9756	0.9988	0.9723
n	**0.261**	**0.262**	0.226	0.027	0.131	0.197		0.4063	0.9994	0.9995
Elong.visc	**0.815**	**0.356**	**-1.195**	**-0.687**	0.567	**1.436**		0.9485	0.9939	0.9922
Volume	**33.492**	**24.951**	27.488	4.684	-35.270	1.484		0.2771	0.9956	0.0156
Density	**0.396**	**0.323**	**0.659**	-0.025	**-0.650**	**-0.538**		0.8247	0.9997	0.9996
Thickness	**62.920**	**35.363**	**137.357**	-8.350	**-225.136**	**-138.912**		0.7673	0.9969	0.0028
Firmness	**27.564**	**8.487**	**82.724**	-0.408	**-129.037**	**-91.708**		0.6453	0.9812	0.0257
Spatial.rupt	**5.290**	**10.726**	-8.697	1.259	**44.970**	**34.389**		0.8758	0.9982	0.5271
Tear. Force	**1.469**	**1.078**	0.600	**-0.832**	0.380	0.993		0.3842	0.9978	0.9640

X1, X2, X3 refer to the proportions of gluten, starch and water-solubles, respectively, in the formulation

In bold: significance at a probability level of α =0.05

Table 4.5c. Predictive regression models describing the relationships between rheological attributes of the dough and biscuit characteristics with the mixture composition (Pseudo-components) of the clear flour

Dependent variables	**Independent variables**			**Nonlinear blending terms**				**R^2(pred)**	**R^2 (adj)**	**P(LOF)**
	x1	**x2**	**x3**	**x1x2**	**x1x3**	**x2x3**	**x1x2x3**			
Hardness	**22.563**	**9.614**	**13.961**	**-30.606**	**-58.231**	**-35.037**	**38.153**	0.9526	0.9982	0.8034
Springiness	**0.448**	**0.270**	**1.568**	0.069	**-1.500**	**-0.875**		0.9420	0.9981	0.9958
Cohesiveness	**0.426**	**0.203**	**1.006**	0.066	**-0.917**	**-0.725**		0.9683	0.9991	0.9985
Consistency	**143.991**	**53.725**	**78.076**	**-192.011**	**-375.501**	**-201.797**	**256.393**	0.9483	0.9982	0.0717
Tla (s)	**3.021**	**0.560**	**0.727**	**-2.319**	**-4.999**	-0.729		0.9904	0.9991	0.9833
Ka (s^{-1})	-0.151	**4.685**	0.605	1.446	**10.080**	6.739		0.9016	0.9961	0.7393
Na	**0.199**	**0.282**	**0.628**	-0.051	**-0.494**	**-0.493**	**0.648**	0.8178	0.9993	0.9990
Elong.visc	**2.051**	**0.937**	**1.781**	**-2.083**	**-6.513**	**-4.182**	**4.482**	0.8417	0.9954	0.9731
Volume	**26.888**	**22.759**	11.243	26.608	28.985	22.442		0.4342	0.9941	0.1001
Density	**0.565**	**0.415**	0.417	-0.192	-0.563	0.050		0.8888	0.9966	0.9920
Thickness	**45.395**	**32.224**	-3.340	33.542	58.784	60.335		0.5438	0.9926	0.0001
Firmness	**107.134**	**26.629**	**331.156**	**-184.259**	**-706.937**	-230.239		0.7121	0.9428	<.0001
Spatial.rupt	**12.774**	**11.683**	**32.196**	**-21.660**	**-36.209**	-15.312		0.8407	0.9941	0.8600
Tear. Force	**10.025**	0.986	**32.443**	-17.934	**-73.190**	-22.525		0.6379	0.8824	0.3533

X1, X2, X3 refer to the proportions of gluten, starch and water-solubles, respectively, in the formulation

In bold: significance at a probability level of α =0.05

Table 4.6. Pearson correlation matrix showing the linear relationships between dough rheological parameters

	Hardness			**Consistency**			**Elong.Visc** *		
	Patent	Mid	Clear	Patent	Mid	Clear	Patent	Mid	Clear
Hardness		1.000		0.922	0.973	0.993	0.877	0.955	0.956
Consistency	0.922	0.973	0.993		1.000		0.912	0.967	0.971
Elong.Visc.	0.877	0.955	0.956	0.912	0.967	0.971		1.000	

Significant value at the level of significance α=0.05 (two-tailed test) for all the parameters.
*Elong.visc; elongational viscosity

Table 4.7. Pearson Correlation Matrix describing the linear relationships between the rheological attributes of the dough and flour components of each flour grade

	Grades	**Total protein**	**Soluble protein**	**Insoluble protein**	**Total pentosans**	**Soluble pentosans**	**Insoluble pentosans**	**Total lipids**
Hardn	Patent	0.727**	0.640**	0.706**	-0.384**	-0.345	-0.648*	0.770*
	Mid-cut	0.495	0.442	0.402	-0.842***	-0.751**	-0.885***	0.612*
	Clear	0.036	-0.041	-0.103	-0.889***	-0.873***	-0.589*	0.275
Cohes	Patent	-0.423	-0.335	-0.401	-0.423	0.385	0.498*	-0.471*
	Mid-cut	0.292	0.339	0.374	0.714**	0.767***	0.247	0.172
	Clear	0.789***	0.834***	0.866***	0.448	0.675**	-0.268	0.613*
Consis	Patent	0.759***	0.633**	0.728***	-0.573*	-0.533*	-0.739**	0.826*
	Mid-cut	0.482	0.426	0.384	-0.881***	-0.792***	-0.897***	0.606*
	Clear	0.089	0.011	-0.052	-0.909***	-0.877***	-0.639**	0.330
Tla	Patent	0.889***	0.791***	0.862***	-0.385	-0.338	-0.587*	0.925*
	Mid-cut	0.903****	0.876****	0.854****	-0.472	-0.305	-0.935***	0.951*
	Clear	0.725**	0.672**	0.626*	-0.659**	-0.444	-0.925***	0.852
k	Patent	-0.837***	-0.845***	-0.841***	-0.088	-0.338	-0.587	-0.818*
	Mid-cut	-0.989***	-0.985***	-0.979***	0.128	-0.055	0.784***	-0.981*
	Clear	-0.928***	-0.901***	-0.875***	0.382	0.109	0.892	-0.967
El. Vis	Patent	0.519	0.343	0.449	-0.703**	-0.676**	-0.799***	0.673*
	Mid-cut	0.509	0.457	0.419	-0.815***	-0.720**	-0.879***	0.622*
	Clear	0.012	-0.071	-0.135	-0.944***	-0.933***	-0.606*	0.267

*, **, ***, **** $p \leq 0.05, 0.01, 0.001, 0.0001$ respectively.Hardn, hardness; Cohes, cohesiveness; Consis, consistency; Tla, half relaxation time; Ka, rate of relaxation; El.Vis, Elongational viscosity.

Table 4.8. Pearson Correlation Matrix describing the linear relationships the relationships between the biscuit quality attributes and flour components of each flour grade

	Grades	**Total protein**	**Soluble protein**	**Insoluble protein**	**Total pentosans**	**Soluble pentosans**	**Insoluble pentosans**	**Total lipids**
Dens	Patent	0.692	0.632	0.658	-0.517	-0.494	-0.609*	0.506
	Mid-cut	0.725**	0.688**	0.662**	-0.596**	-0.462	-0.891***	0.797
	Clear	-0.206	-0.245	-0.275	-0.428	-0.485	-0.127	-0.083
Thick	Patent	0.234	0.176	0.218	-0.316	-0.303	-0.363	0.273
	Mid-cut	0.574*	0.538*	0.514*	-0.556*	-0.449	-0.762***	0.644
	Clear	0.707**	0.667**	0.632*	-0.507	-0.297	-0.816***	0.803
Firm	Patent	0.719**	0.822****	0.748****	0.588*	0.624**	0.396	0.637*
	Mid-cut	0.842****	0.830****	0.821***	0.439*	0.321	0.401*	0.854
	Clear	-0.410	-0.354	-0.309	0.684**	0.639**	0.708**	-0.561
No.	Patent	-0.448	-0.275	-0.403	0.844****	0.819****	0.927****	0.553*
	Mid-cut	-0.602*	-0.555*	-0.520*	0.738**	0.632*	0.873****	-0.700
	Clear	-0.495	-0.425	-0.367	0.832****	0.682**	0.874****	-0.685
Fs	Patent	0.596*	0.647**	0.611*	0.596*	0.341	0.356	0.551
	Mid-cut	0.596*	0.617*	0.632*	0.693	0.632*	0.873****	-0.700
	Clear	-0.353	-0.299	-0.253	0.747**	0.639*	0.756**	-0.493

*, **, ***, **** $p \leq 0.05, 0.01, 0.001, 0.0001$ $p \leq 0.05, 0.01, 0.001, 0.0001$ respectively.

Dens, density; Thick, thickness; Firm, firmness; No, number of spatial ruptures; Fs, mean tearing force

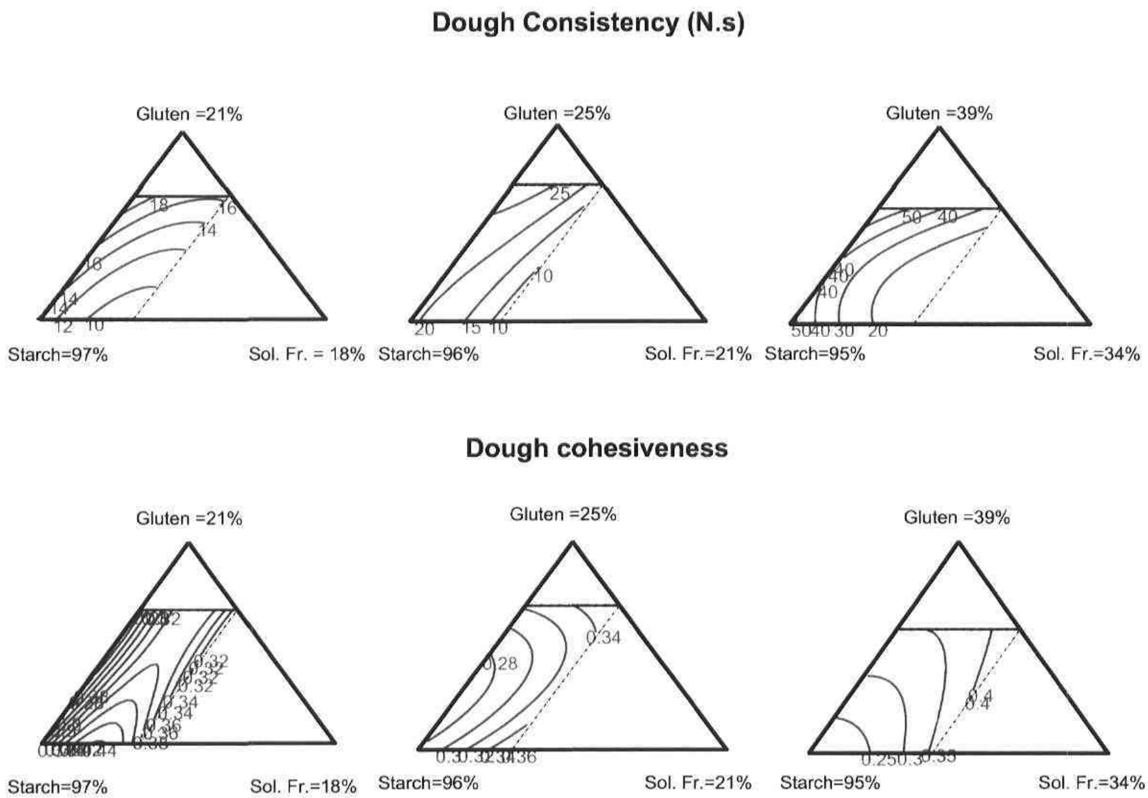

Fig.4.1. Contour lines of predicted consistency and cohesiveness based on mixtures where X_1(gluten) + X_2 (starch) + X_3 (water solubles) are normalized to 100%. From left to right: the fractions isolated from the patent, middle cut and clear flour grades; the parallelogram figure (inset) outlines the «valid area».

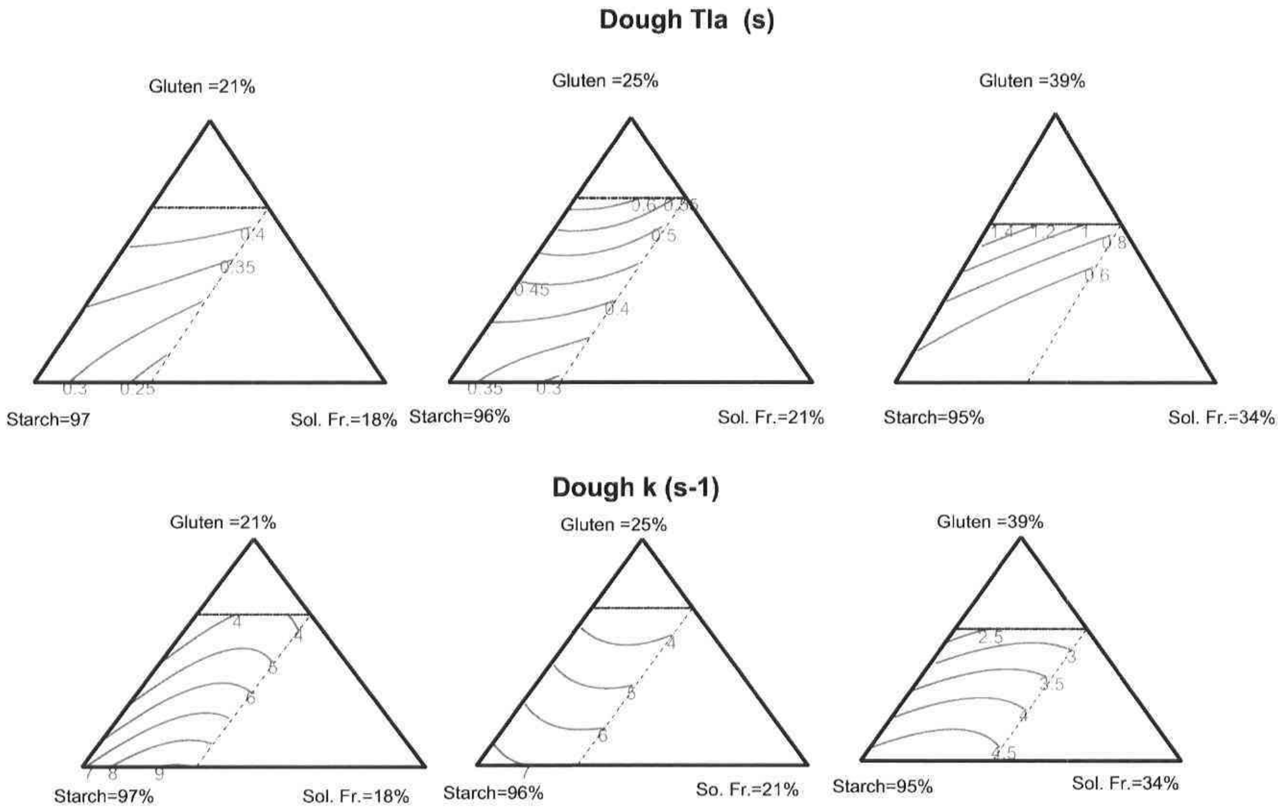

Fig.4.2. Contour lines of predicted relaxation time (Tla) and relaxation rate constant (k) based on mixtures where X_1(gluten) + X_2 (starch) + X_3 (water solubles) are normalized to 100%. From left to right: the fractions isolated from the patent, middle cut and clear flour grades; the parallelogram figure (inset) outlines the «valid area».

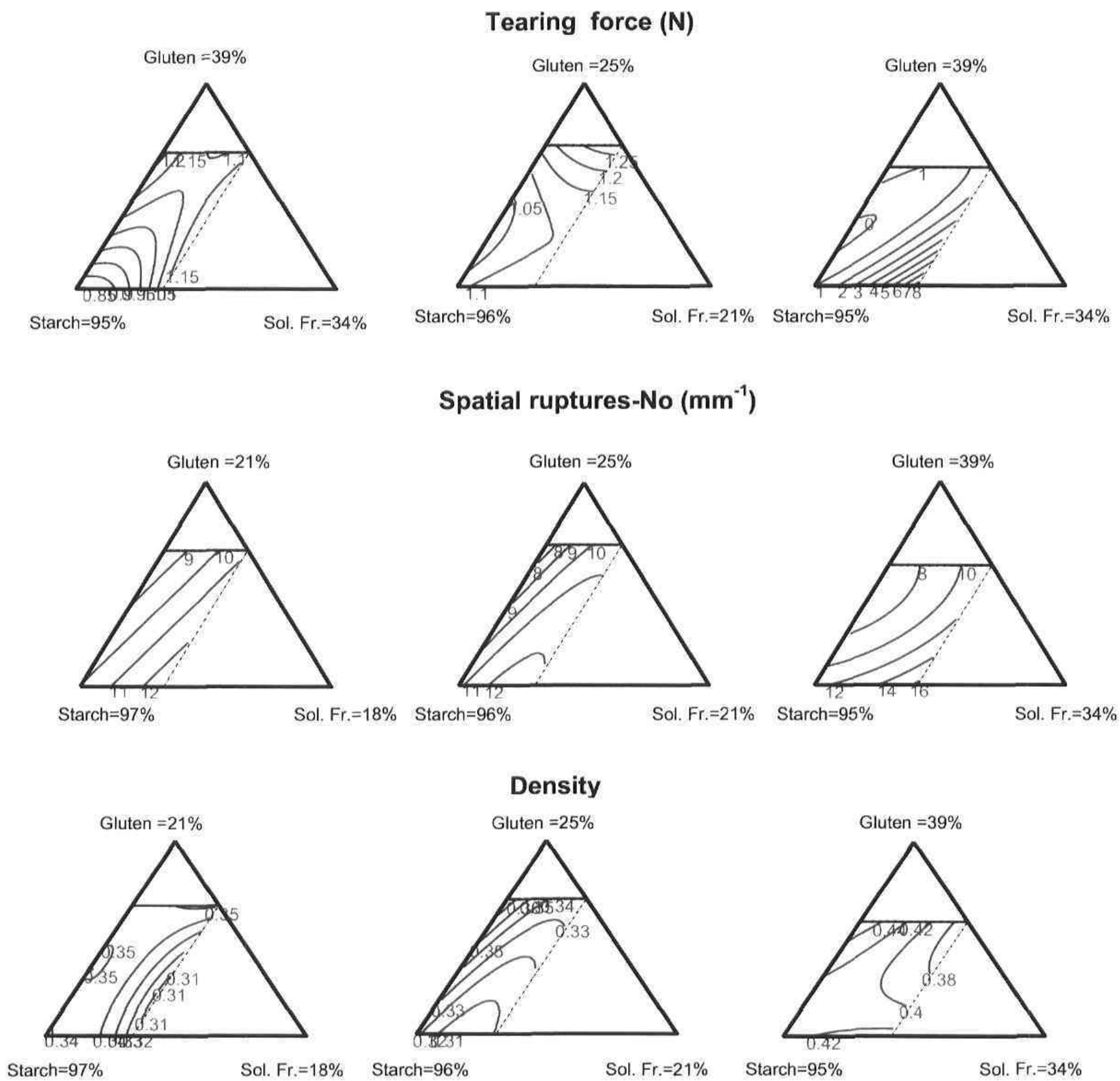

Fig.4.3. Contour lines of predicted biscuits Tearing force, Spatial ruptures and Density based on mixtures where X_1(gluten) + X_2 (starch) + X_3 (water solubles) are normalized to 100%. From left to right: The fractions isolated from the patent, middle cut and clear flour grades; the parallelogram figure (inset) outlines the «valid area».

«Nothing is more dangerous than an idea when it's the only one you have»

Emilie Chartier

CHAPTER V

Incorporation of patent and clear flour streams and their isolated gluten, water solubles and starch tailings fractions into commercial soft wheat flour: impact on dough rheology and semi-sweet biscuit characteristics

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1-Résumé

Une méthode de fractionnement et de reconstitution pour déterminer l'impact de la fortification d'une farine de blé tendre commerciale problématique (pâte collante, biscuits fragiles avec surface rugueuse) par des fractions d'amidon principal et de refus, du gluten et fractions hydrosolubles isolées des farines patente et basses sur la rhéologie et les caractéristiques des biscuits a été évaluée.

L'addition de 10, 20 et 30% de farines patente et basses à la farine commerciale a donné lieu à un accroissement proportionnel de la consistance, de la dureté et de l'élasticité des pâtes biscuitières ainsi que de la densité, et autres propriétés texturales des biscuits. Le collant de la pâte, caractéristique a diminué considérablement, et les biscuits présentaient une surface lisse, sans aucune fissure. La substitution de la farine contrôle par 9.5, 11.5 et 13.5% de gluten isolé des farines patente et basses a exercé un effet important sur la consistance, la dureté et l'élasticité de la pâte. En outre, le gluten isolé de la patente a eu plus d'effet sur ces paramètres que celui de la farine basse, à des teneurs en protéines pratiquement équivalentes, probablement parce qu'il était d'une meilleure qualité protéinique. Par ailleurs, l'ajout de gluten à la farine contrôle a entraîné une augmentation modérée de la densité et de la fermeté du biscuit, mais en a abaissé sa friabilité. La substitution de la farine contrôle par l'amidon de refus à 3,6 et 9% a exercé un effet encore plus marqué sur la consistance et la dureté de la pâte et a aussi influencé de façon très prononcée la densité et la fermeté des biscuits tout en abaissant considérablement leur friabilité, probablement en raison de la forte contribution de pentosanes associées à cette fraction. La substitution par la fraction hydrosoluble et amidon principal à 1, 2.5 et 5% ont exercé un effet négligeable sur le comportement rhéologique des pâtes, mais la fermeté des biscuits est influencée par la teneur en pentosanes de ces fractions.

Les paramètres rhéologiques et les caractéristiques de biscuiterie se sont révélés essentiellement reliés à la composition des fractions, et les résultats indiquent qu'en plus de la quantité et qualité du gluten, d'autres facteurs, comme la teneur en pentosanes influencent aussi les caractéristiques des biscuits semi-sucrés.

2-Abstract

A flour fractionation-reconstitution procedure was used to study the influence of the addition of gluten, water solubles, prime starch and starch tailing fractions isolated from patent and clear flours mill streams on the dough rheology and semi-sweet biscuit characteristics of a problematic commercial soft wheat flour (dough too sticky, biscuits too fragile with cracks). The addition of 10, 20 and 30% on d.w.b of the native patent and clear flour streams to the control soft wheat flour raised the dough's consistency, hardness and elastic properties as well as the biscuit textural attributes (density, firmness and friability). The dough stickiness associated with the control flour was reduced and the biscuits exhibited good surface appearance and were free of cracks. Substitution of the control flour with 9.5, 11.5 and 13.5% of the gluten isolated from the patent and clear streams induced a even more pronounced effect on the dough's consistency, hardness and elastic properties. Moreover, the gluten isolated from the patent stream had more effect on these parameters than the clear stream's gluten, at almost equivalent protein content, likely due to its superior protein quality. Additionally, with increased levels of gluten protein to the control flour there were moderate increases in the biscuit's density, firmness, and lower friability. The substitution of the control flour with 3, 6 and 9% starch tailings produced the largest impact on consistency and hardness of the dough. This fraction also exerted a very pronounced effect on the biscuit's density and firmness, while it lowered the friability, presumably due to the presence of higher amounts of pentosans in this fraction. Both the water solubles at 1, 2.5 and 5.0% and prime starch fractions at 3,6 and 9.0% had negligible effects on dough rheology, but the biscuit's hardness was essentially related to the pentosans content of the resulting flour. The rheological and biscuit properties were related to the composition of the fractions used for the substitution of the base flour and the results suggested that besides wheat gluten quantity and quality, others constituents such as pentosans, as well as the overall composition of the flour blends can largely affect the quality of the semi-sweet biscuits.

3-Introduction

The bakery industry requires flour with appropriate functionality parameters were used to produce different products. The stream splitting method where a given group of similar or complementary streams are blended for specific end-use has already found application (Dubé et al., 1987). The physico-chemical characteristics of these flour streams depend on the wheat cultivars selected, the type of streams that are extracted in the mill, and the flow sheet of the milling operation (Nelson and MacDonald, 1977; Kruger, 1981; Berton et al., 2001).

Flour functionality cannot be described by simple summation of individual constituent's effects (Köehler et al., 1999, 2003; Graßberger et al., 2003). Although the gluten, water solubles and starch sub-fractions have their own specific biochemical characteristics, the flour functionality depends mainly on the structure-function relationships of the constituents inherent to these fractions, their concentration as well as on the numerous interactions among them during the dough development process.

Wheat fractionation-reconstitution procedures have been developed to allow the direct use of dried-ground gluten, crude starch and sub-fractions, and flour water solubles for bread and cookie making to determine wheat functionality for many years (Sollars, 1956a and 1958; Yamazaki and Donelson, 1976; Yamazaki et al., 1977; MacRitchie, 1985; Czuchajowska and Pomeranz, 1995; Miller and Hoseney, 1997). A successful fractionation scheme implies that cookies baked from a blend of the isolated fractions should be identical to those baked from the parent flour. Most studies on the relationships between flour composition and functionality with respect to cookie quality are essentially conducted on a sugar-snap cookie recipe (flour/sugar/fat/water ratio of 100:60:15:30, on 14% moisture basis) and focussed mainly on the cookie spread and top grain. Several authors have discussed the negative impact of protein content on the spreading of cookies (Kaldy et al, 1991), while other studies found no significant relation between cookie diameter and protein content (Donelson, 1988; Nemeth et al., 1994). According to Sollars and Bowie (1966), the reduction of cookie diameter observed with the use of prime starch is mainly related to the content of damaged starch. Moreover, the severe spread depressing effect of starch tailings was nearly proportional to their quantity added to the control flour (Yamazaki, 1955; Sollars, 1966; Sollars and Bowie, 1966). On the other hand, the water solubles fraction is reported to affect dough rheology (Mattern and Sanstead, 1957), and to exert a small effect on the cookie diameter reduction (Sollars, 1959). The non-starch polysaccharides (pentosans) inherent to the starch tailings and the water solubles fraction are hypothesized to influence the baking performance of wheat flour due to their high water binding capacity (Bushuk, 1966). On the basis of previous studies, it appeared that semi-sweet biscuits (flour/sugar/fat/water of 100:30:8:326 on 14% moisture basis) made with reconstituted flour containing starch, water solubles and gluten fractions isolated from the patent, middle-cut and clear flour streams had almost equivalent dimensional characteristics and good surface appearance than their native counterparts, even if the fractionation-reconstitution procedure reduced moderately the consistency and viscoelastic properties of the reconstituted dough.The inner and peripheral layers of the wheat endosperm seemed to have a marked influence on these properties. By creating a variation in the concentration of the gluten, starch and water solubles fractions in the reconstituted flour, major changes in the rheological properties of the dough and biscuit characteristics were observed. It was also found that besides proteins that are often reported to alter significantly the dough and biscuit properties, pentosans originating from the water solubles and starch tailing fractions can also modify substantially these properties.

There has been already a lot of work done trying to assess the effect of other cookie ingredients such as fat, sugar and water on the rheological behaviour of the dough and the dimensional and textural characteristics of the final product using various cookie recipes (Abboud et al., 1985a; Kulp, 1994; Maache Rezzoug et al., 1998b), but little work has been conducted on the role of the flour fractions to modify these properties in a semi-sweet biscuit. Cookie manufacturers can modify the dough rheological behaviour by adding water and other flours to minimize stickiness, reduce consistency of the dough and also to produce desirable cookie texture with soft wheat flour of inappropriate functionality for cookie making. Hence the objective of this study was to evaluate the potential of adding the gluten, starch tailings, prime starch and water solubles at different concentrations into a commercial soft wheat flour, as well as to substitute part of the flour with native patent and clear flours as an alternative to modify the dough and biscuit characteristics.

4-Materials and methods

Previously described in chapter 3 and 4, except for the statistical analysis.

The problematic commercial soft wheat flour used to produce sugar-snap type of cookies was provided by Biscuit Leclerc, St-Augustin de Desmaures, Qc.
The physico-chemical properties of the commercial (control) flour were as follow: protein 7.64%, ash 0.512%, lipids 2.03%, pentosans 1.34% on (g/100g basis) basis. The Water SRC 44.7%, Sucrose SRC 79.1%, Lactic Acid 71.6% and Na_2CO_3 SRC 64.2%
The physico-chemical properties of the patent and clear flour streams, as well as the proportions and composition of the fractions isolated from the patent and clear flour streams are presented in the Tables 3.1, 3.2 and 3.3 in the chapter 3.
The levels of the native patent and clear flour streams added directly to the commercial flour were expressed as g/100g basis. The substitution levels of the commercial flour with various fractions (gluten, starch tailing, prime starch and water solubles) isolated from the two flour streams were also in g/100g basis.

4.1-Statistical Analysis

The analysis of the influence of the native flour streams and their isolated fractions (gluten, water soluble and starch tailing) on soft wheat functionality was performed using one-way analysis of variance; comparisons were made among the fortified samples, and with the control base flour. When a significant F test was found in the one-way ANOVA, the means were compared with the Duncan's test to identify differences among them at the specified significance levels. All analyses were performed using the GLM procedure of the SAS System (SAS version 8.0, SAS Institute Inc, Cary, NC, 1999).

5-Results and discussion

5.1-Influence of the addition of patent and clear flour streams

The commercial flour used in this study exhibited some production difficulties; the dough was too sticky, and the cookies had non uniform surface appearance (with many visible cracks) and were too thin, when made with a productionl recipe closely resembling the sugar snap formula, AACC 10-52. A semi-sweet biscuits recipe was selected to assess the effect of native flour stream substitution and fortification of the control flour on both the dough rheological behavior and the biscuit quality. It is assumed that the flour effect in this latter recipe will have a greater impact on the rheological properties of the dough than the sugar snap-cookie, since these properties are likely more sensitive to the consequences of the formation of a viscoelastic gluten matrix due to higher water availability and lower fat and sugar contents (Tharrault, 1995,1997)

It is known that changes in wheat flour functionality are often related to the dough rheological parameters. For example, it is possible to inter-relate biscuit dimensions to data of the stress relaxation phenomena taking place during dough processing, especially the half-relaxation time (Tla) which is useful to predict biscuit length (Renard and Théry, 1988; Bartolucci and Launay, 2000) and sugar-nap cookie diameter to dough consistency (Gaines et al., 1994). The substitution of the native patent and clear flours streams to the control flour had a significant influence on the dough consistency, as measured by a double compression test, which increased from 19.3, 21.5 and 23.9 N.s for the patent and from 19.9, 21.2 to 25.4 N.s for the clear flour from the control dough consistency of 19.2 N.s, with substitution levels of 10, 20 and 30 wt % in the control flour, respectively ($p<0.0001$), as summarized in Table 5.1. Similar trends are observed with the dough hardness, which increased with the levels of addition of both native flour streams. In parallel, the Tla, determined by the compression-relaxation test which reflects the time necessary to reduce the final compression force by half, once the compression is removed due to the elastic recovery of the dough (retraction phenomenon), rose almost proportionally from 1.76, 1.82

and 1.96 s for the patent flour material from the original 1.48 s of the control flour. A higher Tla value is translated into a lower speed of recovery of the dough once laminated due to the dough viscoelastic properties. For the semi-sweet biscuit, absence of retraction is considered as a criterion of good quality as it determines the potential of biscuit to retract after baking (Contamine et al., 1995). Both flour streams when added to the control base flour demonstrated similar rheological behavior suggesting that either the native clear or the patent streams incorporated at the specified levels would influence similarly the rheological properties of the dough. However, the addition of these flour streams would alter the flour composition. Thus, the added flour streams contribute to a moderate increase of the protein content of the control flour from approximately 0.74 and 1.27% for each 10% increase of the native patent and clear flours, respectively, whereas the total pentosans increase marginally from approximately 0.10% and 0.17% with each 10% increase of the respective flour streams. Moreover, the lipids which are bound essentially to the gluten proteins increased very substantially with the three levels of substitution in the case of the both flour streams (i.e. the overall lipid content was raised by 6.4, 10.9 and 18.7% for the patent and 16.7, 33.0 and 49.3% for the clear, respectively), since the lipids content in the native clear stream is 2.6 times higher than in the patent. These changes in flour composition brought about some variations in the rheological properties of the doughs made with the substitution levels of the patent and clear streams. Control of biscuit's thickness and density are prevalent problems to the commercial production of biscuits. The biscuit dimensions (length, width, thickness) are summarized in Table 5.1. Contrary to the plant production recipe which demonstrated significant technical difficulties, the semi-sweet biscuits produced with the control flour had a smooth surface appearance, but exhibited several surface cracks as shown in Fig 5.1 and broke easily under the effect of pressure, whereas the dough demonstrated a strong stickiness tendency during the sheeting process. The width and thickness of the biscuits made with the fortified flours were not significantly different from that of the control flour, whereas the biscuit length was significantly lowered with the addition of the clear flour stream ($p<0.0001$). As the protein content rises with the level of substitution (9.86 and 11.4% for the patent and clear substitution at 30% substitution level, respectively, versus the control 7.64%), a well developed gluten matrix favors the dough retraction and thus the biscuit's final length. The higher Tla values are also consistent with the higher retraction of the dough and the lower biscuit lengths. It is an indication that the length is mainly dependant on the dough viscoelastic properties; following stamping, the biscuit's length will decrease along the rolling direction and its width will also increase, as a consequence of the strain recovery. Similar observations on these properties have been made by Renard and Théry (1998) and Bartolucci and Launay (2000).

Density is often referred as an important quality parameter for biscuit, in particular for predicting friability (crunchiness). Lower density is often suggested as a quality index for biscuit since it is related to a better development of the biscuit (Tharrault, 1994). The densities were strongly affected by the higher levels of fortification with both the patent and clear flour streams, ranging between 0.35 to 0.40 g/cc ($p<0.0001$); lower densities were attained with the lowest levels of substitution. In parallel, the biscuit firmness almost doubled from 14.3 N to 26.4 N at 30% substitution level with the patent flour stream. It was also of interest to note that for the substituted flours with the clear fraction there was much lower influence on the biscuit firmness parameter than their patent flour containing counterpart, at similar protein concentrations. The puncturing force, which is equivalent to the product resistance to break and corresponds to a reduced biscuit crunchiness, showed similar tendencies with the biscuit firmness, regardless of the type of flour stream used for fortification of the control flour. These results could be related to the superior quality of the gluten proteins present in the patent stream, as suggested by the stronger gluten strength (higher lactic acid SRC value), compared to the native clear flour stream, which instead contains higher amounts of lipids and pentosans. These observations are consistent with the findings of Gaines et al. (1994) who reported an increase in fracture force with increasing protein content of the flour, and that earlier mill streams produce softer cookies. It has been also postulated that these effects are related to the amount of water that the gluten network absorbs. The biscuits produced with the three levels of substitution, all exhibited similar smooth surface appearance and the surface's cracks disappeared at 20 and 30% substitution levels, for both the native patent and clear stream fortified flours. It is important also to note that the dough stickiness of the native control flour was considerably reduced at higher levels of substitution with the patent or clear stream flours.

5.2-Influence of the isolated gluten on dough rheology and biscuits properties

The influence of the isolated gluten addition to the control flour on dough rheological attributes and biscuits characteristics is shown in Table 5.2. The total protein content increased from 7.64 (control flour) to 13.7, 15.0 and 16.2% with the addition of 9.5, 11.5 and 13.5% gluten fraction from the patent flour and 13.4, 14.6 and 15.8% with gluten from the clear flour, whereas the pentosans concentration decreased by .12% for each 2.5% of gluten substituted to the control flour. Moreover, the lipoprotein complex formation on flour doughing during the fractionation procedure resulted in raising very substantially the lipids content in the fortified flours (i.e. 28.6% and 40.4% increase with 9.5% gluten isolated from the patent and clear streams, respectively) as lipids concentration rose by ~ 0.13 and 0.23% for each additional 2% gluten originating from the patent and clear streams. Higher gluten levels resulted in a significant increase of both consistency and hardness; i.e. an increase of consistency from 19.2 N.s to 62.6 N.s and hardness from 1.44 N to 4.01N at 13.5% substitution levels with the gluten isolated from the native patent was noted. Similar trends were observed with the clear gluten flour variants. As expected, the Tla values were also affected by the total concentration of gluten in the fortified flour blends, increasing from 1.48 sec (control) to 1.82, 2.12 and 2.42 sec with increasing levels of the patent's gluten from 9.5, 11.5 and 13.5%, respectively. Moreover, the gluten from the patent flour yielded slightly higher dough consistencies and Tla values than its gluten counterpart from the clear flour, at approximately the same protein concentration. This further supports the notion that besides the amount of protein present in a dough system, its quality largely contributes to the rheological responses of the dough as reported by Donelson, 1988.

A higher gluten concentration in the flour blends also resulted in significantly lower biscuit length than the control flour, especially with the isolated patent gluten fraction ($p < 0.001$); instead, the biscuit length of the flour variants with the clear gluten was only affected at higher concentrations of gluten. In parallel, the biscuit width was not significantly different from the control, but the biscuit's thickness increased with the concentration of gluten, as a consequence of the strain recovery. Increasing the gluten protein content also produced denser, firmer and less crunchy product as reflected by the higher puncturing force. Compared to the direct addition of native patent and clear flour streams at 10, 20 and 30% levels, which is equivalent to a protein contents between ~ 8.4 to 9.9% for the patent and 8.9 to 11.4% for the clear, the values for biscuit density, firmness and product resistance to puncture did not show additional increases with the concentrations of protein in the gluten fortified flours. In contrast to the biscuit quality parameters, the dough rheological behavior was dramatically altered by the higher protein content of the gluten fortified flours. This observation suggests that once a certain protein level in the composite flour is reached, the protein network is properly developed in the semi-biscuit dough, and any additional amounts of gluten proteins present will not modify visually the biscuit structure. All the biscuits produced with the gluten-fortified flours had a smooth, free of cracks surface appearance and did not break easily, and the doughs were all easily laminated. Additionally, the biscuits inner structure appeared more compact with less visible holes at the higher gluten concentrations, and the biscuits were free of cracks pattern (Figure 5.1).

5.3- Influence of the isolated water solubles fraction on dough rheology and biscuit properties

The solubles fraction obtained by centrifugation of the crude starch is made up of soluble proteins (albumins and globulins), amino acids, peptides, pentosans and several other low molecular weight components (Sollars, 1959; Mattern and Sandstead 1957; Miller and Hoseney, 1997a). This fraction is reported to exert little effect on the cookie diameter reduction. The incorporation of the isolated water solubles fractions from the patent and the clear streams into the control flour at 1.0, 2.5 and 5.0% rose slightly the protein content from 7.64 to 8.18 for the patent and 8.36% for the clear at 5.0% substitution level, whereas the pentosans content increased from 1.34 (control) to 1.78 (W.S patent) and to 1.62% (W.S clear) in the respective fortified flour materials (Table 5.3). In accordance with the studies of Chen and Hoseney (1995), the dough stickiness increases drastically with increasing concentrations of this fraction, and a maximum level of 5% addition was still judged acceptable to manipulate the dough in the mixing and sheeting units. Higher amounts of the water solubles fraction incorporated into the control flour did not change significantly the dough consistency, although it increased slightly its hardness value. In contrast, the elastic recovery, expressed by the Tla value, decreased with increasing concentration of water solubles. The high water sorption properties of this fraction could explain this trend since it could preferentially reduce the water availability normally necessary to favor a proper gluten network development. The biscuit's dimensional characteristics remained relatively unchanged, whereas the density slightly declined with higher concentration of the water solubles. In contrast, the adddition of 5% water solubles fraction induced a significant increment of firmness of the biscuits, raising the values by 5.2 N (W.S patent) and 3.7 N (W.S clear) of the control flour, likely due to the contribution of pentosans. No notable differences were noted with the biscuit surface appearance for all products made with the W.S fortified flours.

5.4-Influence of the prime starch and starch tailings fraction on the dough rheology and biscuits properties

Previous studies have suggested that the isolated crude starch had little impact on dough rheology and biscuit quality in comparison to the gluten and water solubles fractions. Thus the prime and starch tailings were studied separately to assess their role on the semi-sweet biscuit recipe. Literature data indicated that prime starch consists mainly of large, undamaged starch granules and according to Sollars and Bowie (1966), the cookie diameter, depends, above all, on the damaged starch content; the higher its content, the smaller the biscuit diameter. Both the prime starch and starch tailings were added at 3, 6 and 9% levels in the control flour. The prime starch did not affect neither the dough rheology nor the biscuit quality significantly when used to fortify the control flour (results not shown). Moreover, all the biscuits made with the prime starch had the same surface cracking appearance than the control flour as shown in Figure 5.1.

Starch tailings, on the other hand, is reported to consist mainly of fragmented and small starch granules, and parts of endosperm cells walls with adhering aleurone and bran tissues;

this material is rich in pentosans (Yamazaki, 1955). According to Mac Masters & Hilbert, 1944, the average composition for the starch tailings is starch 87-94%, pentosans 4%, cellulosic materials 3%, protein 1-2%, and fatty materials 0.3%. The reported effect for declining cookie diameter on fortification of flour with the starch tailings fraction in a sugar-snap cookie has been attributed mainly to the pentosan-rich sub-fractions of the wheat flour (Cottenet, 1986) and to the damaged starch, as well as to some minor contributing effects from lipids and the small starch granules present in this fraction (Sollars, 1966). Such behavior is generally thought to originate from the physicochemical properties of the constituents of this material and more specifically from their water absorption properties (Jelaca and Hlynka, 1971; Jeltema et al., 1983)

Compared to gluten, the starch tailings addition had an even more significant influence on the dough's consistency; the consistency of the control flour rose by a factor of ~1.5, 2.5 and 4.5 times with the three levels of fortification, and this effect was independent of the source of the isolated stream fraction (Table 5.4). Similar pattern was observed with the hardness attribute. This behavior may be explained by the increased concentration of pentosans in the fortified flours which rose by 0.15% for every 3% increase of the starch tailings fraction, whereas both the protein and lipids contents remained relatively constant. Contrary to our expectations, the dough Tla values also increased significantly with increasing concentration of starch tailings ($p < 0.001$), in spite of the minor elevation of the protein content of the S.T fortified variants. One possible explanation is that in spite of the limited water availability and the high amounts of fat and sugars in the biscuit recipe, pentosans might contribute to the formation of an elastic composite gluten network as these constituents have the potential to interact with the protein (Cottenet, 1986).

The biscuit's dimensional parameters were strongly affected by the starch tailings concentration ($p < 0.001$). Biscuit firmness showed a ~1.5, 1.9 and 2.3-fold increase with the starch tailings isolated from the patent and clear flour, confirming that this fraction exerts the largest impact on the biscuit textural attributes among the fractions studied. This is further evidenced by the puncturing force values which also largely increased with the level of S.T substitution; the values for the latter parameter indicate that the overall crunchiness declines substantially for cookies made with S.T fortified flours. Moreover, the biscuits produced with added starch tailings, had a very compact inner structure with little holes and darker, especially at 6 and 9% levels as shown in Figure 5.1 and they were difficult to break and quite hard organoleptically.

6-Conclusions

Partial substitution of commercial soft wheat flour with native patent and clear mill streams and/or fortification with gluten and starch tailing fractions isolated from these two streams had a significant impact on the dough rheological behavior and also the semi-sweet biscuit dimensional and textural properties. Among the fortifications with isolated fractions from the patent and clear flour streams, the starch tailings fraction had the largest impact in changing the dough and biscuit characteristics; the starch tailing variants produced the stronger biscuit structure and the products did not crack easily. The gluten fraction also showed major changes in the rheological attributes, but it affected the biscuit characteristics to a lower extent than the starch tailings, whereas the prime starch and water solubles fractions had a limited influence on these properties. These data also suggest that satisfactory semi-sweet biscuit quality appear to be the net result of a number of contributing functionalities from different constituents and flour fractions; e.g. up to a certain level pentosans and proteins may act as effective modifiers of the dough and biscuit properties, thus eliminating excessive stickiness of the dough and abnormal fragility of the end-product.

This study also confirmed that quantitative effects of dough components on dough characteristics can be manipulated, and important information of the relationships between composition and functional behavior can be obtained for any cookie recipe, using a simple reconstituted flour system. With this knowledge, biscuits of improved quality can be prepared by exploiting the functional potential of some endogenous flour constituents, namely the pentosans, lipids and proteins of each flour fraction originating from various mill streams sources, rather than by modification of the water/sugar/fat ratio in a recipe. The plasticizing effect of water on wheat endosperm proteins, which may vary with the type of flour fraction added, may also depend on the concentration of the pentosans and other water solubles as well as on the starch damage levels. Additionally, the variation in the concentration and functionality of proteins (glutenin and gliadin fractions), pentosans and lipids of the various flour streams within a given milling operation need to be better understood.

The results of the present study indicated that a simple reconstituted dough system can provide an unambiguous assignment of the quantitative and qualitative effects of the various flour constituents on dough characteristics and biscuit making quality. It has the advantages that any aspect of the flour constituent's functionality can easily be modified to improve the flour technological value for a particular end-product

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Table 5.1: Compositional aspects and means of biscuit-making parameters of the native patent (P1) and clear (CLR) flour streams

Sample	Composition			Dough			Biscuit					
	Protein (%)	Pentosans (%)	Lipids (%)	Consis. (N.s)	Hardn. (N)	Tla (s)	Length (mm)	Width (mm)	Thick. (mm)	Density (g/cc)	Firmn. (N)	Punct. (N)
Control	7.64	1.34	2.03	19.2^{c}	1.44^{e}	1.48^{e}	62.2^{a}	64.0^{a}	10.6^{a}	0.35^{f}	14.3^{d}	7.01^{c}
10% P1	8.38	1.44	2.16	19.3^{c}	1.66^{d}	1.76^{cd}	62.6^{a}	64.2^{a}	10.2^{ab}	0.36^{ef}	23.9^{b}	10.2^{b}
20% P1	9.12	1.54	2.28	21.5^{b}	1.96^{bc}	1.82^{bc}	62.1^{ab}	63.9^{a}	10.1^{bc}	0.37^{cd}	26.9^{a}	11.3^{a}
30% P1	9.86	1.64	2.41	23.9^{a}	2.02^{abc}	1.96^{ab}	60.9^{cd}	64.6^{a}	10.2^{ab}	0.39^{b}	26.4^{a}	10.6^{ab}
10% CLR	8.91	1.51	2.37	19.9^{bc}	1.85^{c}	1.48^{e}	61.5^{bc}	63.8^{a}	10.0^{bc}	0.36^{dc}	14.7^{d}	6.56^{c}
20% CLR	10.2	1.69	2.70	21.2^{b}	2.12^{ab}	1.66^{d}	60.8^{cd}	63.7^{a}	9.92^{bc}	0.38^{bc}	17.1^{c}	7.33^{c}
30% CLR	11.4	1.87	3.03	25.4^{a}	2.18^{a}	2.02^{a}	60.5^{d}	64.4^{a}	9.69^{c}	0.40^{a}	18.7^{c}	7.36^{c}
p-value				<0.0001	<0.0001	<0.0001	<0.0001	<0.1013	<0.005	<0.0001	<0.0001	<0.0001

Values within each column with the same letter are not significant at the given p-value

Consis, consistency; Hardn, hardness; Tla, rate of relaxation, Firm, firmness; punct, puncturing force

Table 5.2: Compositional aspects and means of biscuit-making parameters of the Gluten fraction isolated from the patent (P1) and the clear (CLR) flour streams

	Composition			Dough			Biscuit					
Sample	Protein (%)	Pentos (%)	Lipids (%)	Consis. (N.s)	Hardn. (N)	Tla (s)	Length (mm)	Width (mm)	Thick. (mm)	Density (g/cc)	Firmn. (N)	Punct. (N)
Control	7.64	1.34	2.03	19.2^{e}	1.44^{d}	1.48^{e}	62.2^{a}	64.0^{b}	10.6^{bcd}	0.35^{e}	14.3^{d}	7.01^{cd}
Gluten P1 9.5%	13.7	1.22	2.61	37.4^{d}	1.97^{d}	1.82^{d}	60.9^{bc}	64.0^{b}	10.6^{bcd}	0.37^{c}	15.8^{c}	7.35^{bc}
Gluten P1 11.5%	15.0	1.18	2.74	58.1^{ab}	2.62^{c}	2.12^{bc}	59.8^{cd}	65.0^{a}	10.9^{abc}	0.38^{b}	21.2^{a}	9.97^{a}
Gluten P1 13.5%	16.3	1.15	2.85	62.6^{a}	4.01^{b}	2.42^{a}	58.2^{e}	64.2^{b}	11.3^{a}	0.39^{a}	22.3^{a}	10.2^{a}
Gluten CLR9.5%	13.4	1.22	2.85	22.5^{e}	1.72^{d}	1.84^{d}	61.1^{ab}	64.9^{a}	10.5^{cd}	0.35^{de}	13.9^{d}	5.53^{e}
Gluten CLR11.5%	14.6	1.18	3.08	44.8^{c}	2.68^{c}	2.03^{c}	61.4^{ab}	65.0^{a}	10.2^{d}	0.36^{cd}	15.9^{c}	6.74^{d}
Gluten CLR 13.5%	15.8	1.15	3.26	57.6^{b}	5.42^{a}	2.30^{ab}	59.7^{d}	64.6^{ab}	11.0^{ab}	0.39^{ab}	18.5^{b}	7.83^{b}
p-value				<0.0001	<0.0001	<0.0001	<0.0001	<0.0059	<0.0068	<0.0001	<0.0001	<0.0001

Values within each column with the same letter are not significant at the given p-value

Table 5.3: Compositional aspects and means of biscuit-making parameters of the Water Solubles (W.S) fraction isolated from the patent (P1) and the clear (CLR) flour streams

Sample	Composition			Dough			Biscuit					
	Protein (%)	Pentosans (%)	Lipids (%)	Consis. (N.s)	Hardn. (N)	Tla (s)	Length (mm)	Width (mm)	Thick. (mm)	Density (g/cc)	Firmn. (N)	Punct. (N)
Control	7.64	1.34	2.03	19.2^{a}	1.44^{b}	1.48^{a}	62.2^{a}	64.0^{a}	10.6^{a}	0.35^{a}	14.3^{e}	7.01^{bc}
W.S P1 1%	7.74	1.43	2.00	19.8^{a}	1.79^{a}	1.43^{ab}	61.9^{a}	63.9^{a}	10.2^{ab}	0.33^{bc}	17.4^{bc}	6.93^{c}
W.S P1 2.5%	7.90	1.56	1.98	19.8^{a}	1.89^{a}	1.35^{bc}	60.9^{b}	63.7^{a}	9.16^{c}	0.33^{bc}	18.1^{ab}	7.55^{b}
W.S P1 5%	8.18	1.78	1.92	20.3^{a}	1.89^{a}	1.29^{cd}	61.3^{b}	63.9^{a}	9.81^{ab}	0.32^{c}	19.5^{a}	7.51^{b}
W.S CLR 1%	7.78	1.40	2.00	18.9^{a}	1.83^{a}	1.51^{a}	61.2^{b}	64.1^{a}	9.94^{b}	0.34^{ab}	15.6^{dc}	7.38^{bc}
W.S CLR 2.5%	8.00	1.48	1.98	19.6^{a}	1.76^{a}	1.28^{cd}	61.1^{b}	64.0^{a}	9.89^{b}	0.34^{ab}	16.4^{cd}	6.93^{c}
W.S CLR 5.0%	8.36	1.62	1.92	18.3^{a}	1.90^{a}	1.22^{d}	61.1^{b}	63.8^{a}	10.0^{b}	0.33^{bc}	18.0^{ab}	8.18^{a}
p-value				<0.14	<0.0001	<0.0001	<0.0001	<0.81	<0.0001	<0.0268	<0.0001	<0.0001

Values within each column with the same letter are not significant at the given p-value

Table 5.4: Compositional aspects and means of biscuit-making parameters of the Starch Tailings (S.T) fractions isolated from the patent (P1) and the clear (CLR) flours streams

	Composition			Dough			Biscuit					
Sample	Protein (%)	Pentosans (%)	Lipids (%)	Consis. (N.s)	Hardn. (N)	Tla (s)	Length (mm)	Width (mm)	Thick. (mm)	Density (g/cc)	Firmn. (N)	Punct. (N)
Control	7.64	1.34	2.03	19.2^{d}	1.44^{e}	1.48^{d}	64.2^{a}	64.0^{ab}	10.6^{a}	0.35^{d}	14.3^{d}	7.01^{d}
S.T P1 3%	7.46	1.45	1.97	29.7^{c}	3.28^{d}	1.50^{d}	64.3^{a}	64.3^{a}	10.0^{b}	0.38^{c}	21.4^{c}	9.27^{c}
S.T P1 6%	7.29	1.57	1.91	48.9^{b}	5.11^{c}	1.88^{bc}	64.3^{a}	64.3^{a}	9.68^{b}	0.40^{b}	27.5^{b}	10.9^{b}
S.T P1 9%	7.12	1.67	1.85	90.4^{a}	8.92^{b}	2.14^{a}	65.5^{bc}	63.5^{bc}	8.96^{c}	0.42^{a}	32.8^{a}	14.2^{a}
S.T CLR 3%	7.50	1.45	1.97	28.1^{c}	3.25^{d}	1.59^{d}	62.6^{d}	62.6^{d}	8.78^{cd}	0.37^{c}	16.4^{d}	7.71^{d}
S.T CLR 6%	7.37	1.56	1.91	48.8^{b}	5.12^{c}	1.77^{c}	61.7^{e}	61.7^{e}	8.36^{d}	0.38^{c}	27.8^{b}	10.9^{b}
S.T CLR 9%	7.23	1.66	1.85	87.9^{a}	10.6^{a}	2.00^{b}	63.2^{cd}	63.2^{cd}	9.13^{c}	0.40^{b}	34.6^{a}	13.3^{a}
p-value				<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001

Values within each column with the same letter are not significant at the given p-value

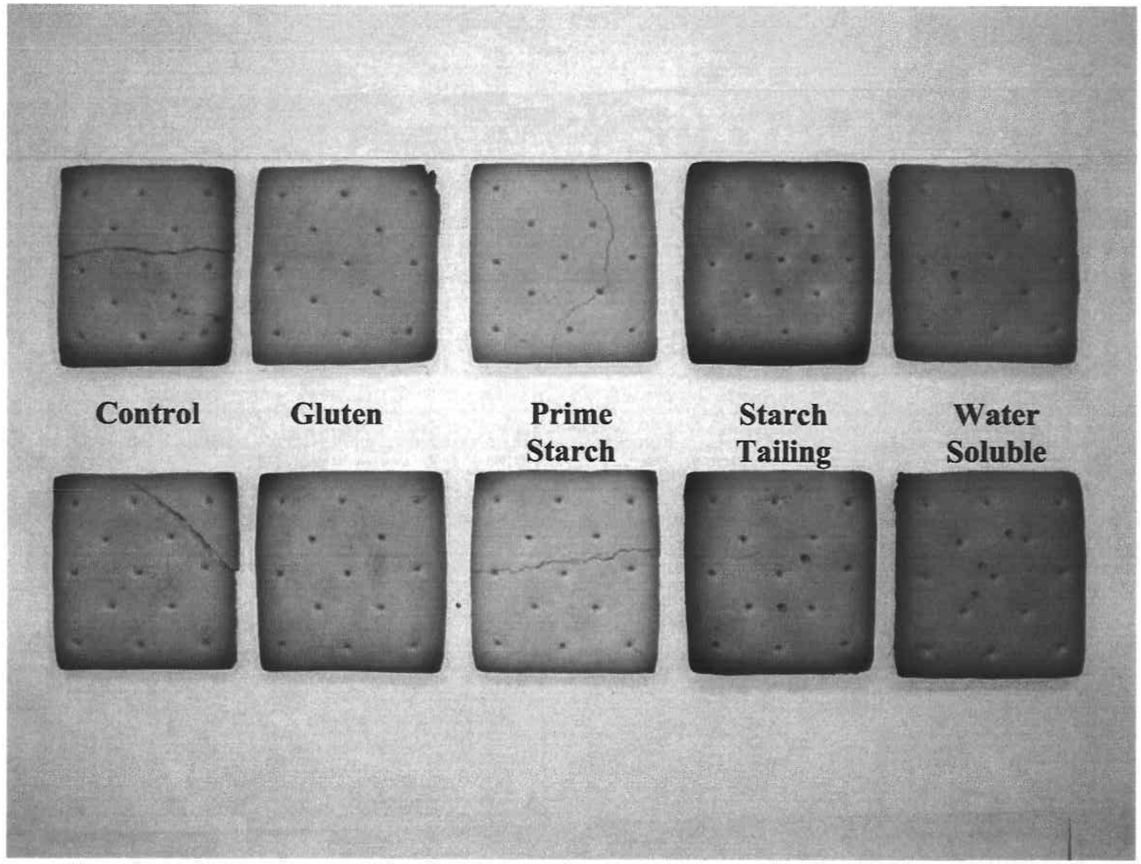


Figure 5.1. Cracking and surface appearance of the control flour supplemented with gluten (11.5%), prime starch (6%), starch tailing (6%) and water soluble (2.5%).

« The most exciting phrase to hear in Science, the one that heralds new discoveries, is not Eureka! ., but That's funny »

Isaac Asimov (1920-1992)

CHAPITRE VI

Conclusions et perspectives de recherche

1- Discussion générale.

Lors de sa transformation, la farine passe par un état pâteux, dont les propriétés complexes sont déterminantes, en terme de valeur d'utilisation. Les méthodes rhéologiques, fondamentales et empiriques permettent de caractériser cet état. Les approches physico-chimiques et rhéologiques constituent deux voies privilégiées pour comprendre, ou du moins savoir prévoir, la variabilité de la valeur technologique des farines. En effet, le procédé biscuitier soufre de l'absence de corrélations significatives entre la qualité de la matière première, la farine notamment, et la qualité du produit fini. C'est pour remédier à cet état qu'une étude a été menée, analysant le comportement rhéologique de la pâte d'une part, et les caractéristiques dimensionnelles et texturales du produit fini, tout en identifiant les critères analytiques susceptibles de prédire la variabilité de la valeur biscuitière des farines.

Dans ce travail nous avons démontré le potentiel des trois courants de mouture qui constituent la farine commerciale, et de leurs constituants sur les propriétés rhéologiques des pâtes, lesquelles sont considérées, à priori, comme étant la base des mécanismes qui déterminent les changements de dimensions et qualité des biscuits. Les éléments présentés ci-dessous devraient contribuer à apporter quelques réponses à cette demande, qui concerne aussi bien les industriels que les minotiers, soucieux d'améliorer la consistance de leur production et qualité des biscuits sans ajout d'ingrédients non amicaux.

Dans la première partie de notre étude, nous nous sommes particulièrement intéressés à cerner les problèmes de disparités de la fonctionnalité des farines selon le type de produits et procédés avec des recettes typiques des précédés coupe-fil, à la rotative et laminé. Cette étude a été réalisée avec l'objectif de prédire leur comportement rhéologique et biscuitier lorsqu' ils sont produits avec des farines présentant de moyennes et larges variations physico-chimiques obtenues par des mélanges de ces courants de mouture dans des proportions potentiellement rencontrées dans les minoteries. Rappelons, à cet effet , que la plupart des résultats publiés à date ont été acquis avec les anciennes normes AACC 10-52 ou sugar-snap cookie et AACC 10-53 ou micro-wire cookie. Une comparaison de trois recettes très différentes, quant aux procédés de fabrication et aussi à leurs proportions farine/sucre/gras et eau semblait donc légitime pour adresser l'aspect disparité de la fonctionnalité des farines selon la recette choisie. Cette disparité est bien réelle à l'usine. Elle se traduit par des pâtes collantes et non machinables, des cookies aux dimensions demeurées, trop fragiles ou trop dures, produits finis trop cassants. Fait intéressant : un problème spécifique peut apparaître pour une recette donnée, mais non pour une autre et cela avec la même farine provenant d'un silo unique, lors de la même journée de production.

L'utilisation de la farine basse dans les trois recettes biscuitières provoque une augmentation de la consistance des pâtes, une augmentation de la fermeté et densité des trois produits. De plus les biscuits secs se caractérisent par une structure interne bien cohésive et ils sont peu croustillants. Par contre, on observe une corrélation inverse avec ces mêmes attributs avec l'utilisation de la farine patente. La patente adoucit la pâte, la rend plus collante et induit une décroissance de sa consistance et elle contribue à l'augmentation de la longueur des biscuits secs, lesquels se caractérisent par une structure croustillante, susceptibles à la rupture. Quant à la farine de coupure, elle produit des pâtes aux consistances intermédiaires et des biscuits secs d'une fermeté et friabilité acceptables. Toutes les combinaisons de courants de mouture induisent de larges variations dans la composition en pentosane, minérale, et protéique et provoquent, comme les fractions individuelles, des changements considérables sur les caractéristiques des pâtes et biscuits. Ainsi les variations de la consistance des pâtes produites avec des combinaisons des fractions de mouture sont essentiellement en relation avec le niveau de la farine basse. Toute augmentation ou diminution de cette dernière dans la farine s'accompagne d'une croissance ou décroissance de la consistance des pâtes. De plus, les réponses de ces attributs rhéologiques et produits finis varient d'une recette à l'autre qu'il devient difficile d'établir un lien de causalité entre une réponse à un paramètre donné et une fraction de mouture. Ainsi, les combinaisons des trois courants de mouture donnaient des cookies coupe-fil et à la rotative aux densités plus élevées que la farine standard pour la saison (40 :48 :12 pour la farine patente, de coupure et basse, respectivement), alors que dans le cas des biscuits secs, laminés, ces densités étaient plus faibles que la farine standard de la récolte saisonnière. Il est donc impossible d'extrapoler les observations d'une recette à l'autre et cela, à cause principalement de la contribution des ingrédients de la recette tels les gras et sucres qui est loin d'être négligeable. Comprendre cet aspect variabilité de la fonctionnalité de la farine exige donc une bonne connaissance des changements qui se produisent avec tous les attributs rhéologiques des pâtes et produits finis. A cet égard, Wainright (1985) démontrait que la densité des biscuits secs Richtea diminuait avec une augmentation du diamètre médian, mais pour les biscuits à la rotative Lincoln, une corrélation inverse était notée.

L'étude des régressions «PLS» permet de mieux expliquer l'influence des trois fractions de mouture et de leurs interactions sur la consistance des pâtes, un paramètre souvent utilisé pour prédire le diamètre des cookies et les caractéristiques des biscuits et les paramètres densité, fermeté et surface des produits finis. Cette approche statistique permet de décrire l'impact des variations des fractions de mouture incorporées individuellement ou en combinaison ainsi que leurs interactions sur les caractéristiques des pâtes et produits finis pour mieux appréhender les relations qui prévalent entre les farines individuelles et recombinées et les réponses de la pâte et des produits, ou plus spécifiquement, entre les variables indépendantes (les fractions de mouture individuelles ou combinées) et les variables dépendantes (attributs des pâtes et produits finis). Cette méthode de régression permet de corréler les variables indépendantes aux variables dépendantes pour fournir une synthèse descriptive des relations qui prévalent entre la densité, par exemple, et l'influence de la farine basse, patente ou toute combinaisons possible. Cette méthode a confirmé la complexité extrême de l'effet farine pour la production des cookies et biscuits, surtout lorsque des modifications sont apportées dans les proportions de ces trois grades de farine. La précision des modèles est améliorée significativement en considérant les interactions qui existent entre ces trois fractions, selon la recette biscuitière. De plus, l'analyse des coefficients de corrélation permet de déterminer l'importance relative de ces fractions de mouture, leur interaction et leur effet carré sur les caractéristiques des pâtes et produits finis et aussi déterminer quelles sont les combinaisons de fractions les plus déterminantes sur les paramètres consistance des pâtes, volume, densité et fermeté des cookies et biscuits secs.

L'étude de plusieurs caractéristiques indépendantes des pâtes nous a permis d'établir certaines relations qui existent entre les propriétés physico-chimiques des farines et les caractéristiques des pâtes et produits finis. Ainsi, si l'on considère les corrélations linéaires simples, il apparaît que les paramètres physico-chimiques (protéines, granulométrie, cendres et alvéographe P) permettent d'expliquer les variations de la consistance des pâtes à cookies coupe fil et à la rotative. De plus, les propriétés de viscoélasticité exprimées par les indices Tla et k sur des pâtes simples (farine dans l'eau saline) complémentent bien les paramètres physico-chimiques pour prédire l'attribut consistance de la pâte des biscuits secs, laminés.

La densité est un important paramètre pour prédire le croustillant des cookies. Pour les recettes des cookies à la rotative et coupe-fil, les meilleures corrélations simples obtenues avec la densité font apparaître les mêmes paramètres explicatifs : il s'agit des paramètres rhéologiques mesurés à hydratation constante tels que le P de l'alvéographe, la force à 50% de compression (Fi), le temps de demi relaxation (Tla) après compression en conditions lubrifiées. On trouve également la faible granulométrie et les protéines pour appréhender la densité des cookies. Pour les biscuits secs, ces mêmes paramètres explicatifs apparaissent, mais à des degrés moindre (valeurs de p plus élevées). L'association du facteur protéine et la fraction granulométrique pour prédire la densité des produits biscuitiers a déjà été mis en évidence dans la littérature. Plus cette taille des particules est réduite, plus la densité est faible et une faible densité est une caractéristique recherchée par le biscuitier. Ces résultats nous laissent supposer que la distribution granulométrique peut servir comme traceur indirect de la qualité des protéines qui expliquerait à la fois des différences de la fracturabilité du grain et de la détermination de la densité des biscuits. Cette hypothèse rejoint celle de Slade et Levine (1994) qui dans le cas des cookies, attribuent les différences de comportement des farines aux propriétés thermodynamiques du gluten : son comportement avant la transition vitreuse serait apparenté à un matériau rendu caoutchouteux par thermodurcissement ou à celui d'un polymère fondu, selon qu'il s'agit d'une farine donnant une qualité médiocre ou satisfaisante, respectivement. De plus, le fait que l'on retrouve une bonne corrélation entre la force à 40% de compression et la densité des cookies milite aussi en faveur de l'idée d'un mécanisme rhéologique. Quant aux paramètres surface, volume et fermeté des trois produits biscuitiers, aucun des paramètres physico-chimiques et rhéologiques ne générait de bonnes valeurs prédictives (p trop élevé).

L'étude des corrélations linéaires a aussi permis de démontrer que les biscuits secs suivi par les cookies coupe-fil génèrent plus de réponses prédictives que les cookies à la rotative, à cause de leur plus grande teneur en eau qui permet d'induire des changements plus notables avec la variation de la composition biochimique de la matière première.

D'un point de vue pratique, cette étude a aussi démontré que la teneur en protéines et granulométrie ainsi que l'usage de la méthode empirique de l'alvéographe sur des pâtes, lesquelles sont couramment utilisés pour la sélection des cultivars de blé tendre et dures, sont effectivement utile pour éventuellement prédire la consistance des pâtes à cookies. Pour la pâte à biscuits secs, les propriétés viscoélastiques des pâtes réalisées par compression bi-axiale en conditions lubrifiées sont d'un intérêt particulier pour prédire certaines caractéristiques des produits finis.

Le fractionnement et la reconstitution des fractions gluten, amidon et fraction soluble ou autres sous fractions de la farine a permis de mettre en évidence les liens de cause à effet sur le comportement des pâtes et caractéristiques des biscuits secs. Une telle procédure apporte des informations intéressantes sur le rôle des constituants tels que les protéines, pentosanes, lipides et autres constituants déterminants dans la qualité des biscuits. Cela implique, avant tout, que l'isolation de ces fractions n'altère pas la fonctionnalité de la farine reconstituée. Ces fractions, lorsque re-incorporées dans les mêmes proportions que la farine originale doivent posséder les mêmes caractéristiques que la farine native. Plusieurs études de fractionnement et reconstitution réalisées sur les cookies sugar-snap cookies ont démontré que le diamètre et le « top grain» des produits finis ressemblaient aux cookies produits avec la farine originale, seulement si les lipides extraits par le dichloromethane (ou un solvant équivalent) étaient réincorporés à la farine délipidée, à la même concentration que la farine originale correspondante. Cependant, aucune référence ne discute de l'impact du fractionnement sur certains attributs rhéologiques des pâtes des cookies et biscuits, tels la consistance et viscoélasticité des pâtes. Des travaux récents sur le fractionnement des farines de blé dure confirment que l'extensibilité et la résistance de la pâte farine+eau mesurée par le test Kiefer ou la hauteur du pic enregistrée par un mixographe sur les farines reconstituées diminuaient très significativement. Ces résultats laissent présager que la procédure de fractionnement peut altérer la fonctionnalité des farines en provoquant une agrégation des fractions protéiques par dénaturation mécanique, due aux interactions non covalentes qui se développent entre les différents constituants de la farine durant son hydratation, en modifiant l'état de dispersion des constituants endogènes à la farine, en modifiant leurs capacité d'absorption de l'eau suite à la lyophilisation des fractions, etc. L'explication la plus plausible pour interpréter que l'étalement des cookies sugar-snap n'est pas affecté par la procédure de fractionnement est que la mise en forme des cookies dépend d'autres facteurs, et surtout celui impliquant un phénomène de transition vitreuse qui est encore mal expliqué pour les produits biscuitiers.

Notre étude a permis de démontrer qu'une simple procédure de fractionnement très similaire à un précédé de fractionnement industriel tel que le procédé Martin ou Raisio, bien décrit par Feillet (2000), sans extraire les lipides endogènes, permettaient de produire des biscuits secs, ayant des surfaces lisses et longueurs légèrement plus élevées, que les biscuits produits avec les farines originales correspondantes. Cependant, une diminution substantielle des attributs viscosité élongationnelle, consistance et vitesse de relaxation a été notée sur toutes les pâtes reconstituées par rapport aux farines natives. Cette observation confirment que certains paramètres rhéologiques n'expliquent pas les caractéristiques dimensionnelles et que d'autres facteurs non identifiés peuvent intervenir dans la mise en forme du biscuit. Au niveau du test biscuitier pour les biscuits secs, dont le protocole a fait ses preuves tant en matière de répétitivité que de pouvoir discriminant en Europe pour la sélection des farines à biscuits, il serait important de faire un suivi rigoureux des caractéristiques dimensionnelles, de la découpe à la mise au four, et pendant la cuisson. A cet égard, les travaux de Slade et Levine (1994) peuvent servir de référence. Ces auteurs ont étudié la fabrication des cookies, en couplant l'étude des propriétés thermomécaniques du gluten aux cinétiques de changement de dimension en cuisson, par une technique de photographie séquentielle. Le système des biscuits secs constitue un système très différent des cookies, à cause des teneurs réduites en sucres et en gras, ce qui rend les transferts directs de connaissance difficile. C'est la raison pour laquelle, il nous semblerait utile d'appliquer ce type d'approche aux biscuits secs pour appréhender l'influence des paramètres rhéologiques sur les caractéristiques dimensionnelles.

Par rapport aux cookies sugar-snap, dont l'étalement est principalement contrôlé par la consistance des pâtes, les pâtes à biscuits doivent être assez extensibles pour être laminées, sans être trop élastiques pour éviter une rétraction trop importante après la découpe. Ce sont ces variations dans la recouvrance élastique qui expliquent les variations des dimensions et poids des biscuits lors de la fabrication et l'absence de rétraction est considéré comme un facteur de qualité. Le test de compression/relaxation en conditions lubrifiées donne systématiquement de bonnes corrélations avec ce test biscuitier car il est bien adapté aux déformations réelles et essentiellement élongationnelles que subit la pâte biscuitière lors de son laminage. Notre choix de travailler en extension bi-axiale, à grande déformation, plutôt qu'en cisaillement et aux petites déformations, c'est-à-dire dans des conditions plus proches de celles rencontrées en technologie industrielle a donc été justifié pour les biscuits secs. La prééminence des phénomènes viscoélastiques a été mise en évidence : plus les contraintes accumulées dans la pâte se relaxent vite, plus la recouvrance est faible, et donc plus la longueur est élevée. Cela se traduit par une corrélation hautement significative, entre la longueur des biscuits, et la constante de la vitesse de relaxation (k). Cette constante issue d'un modèle semi empirique proposé par Launay (1990) en interprétant les rhéogrammes de compression/relaxation par le modèle de Maxwell convecté, permet de donner une bonne interprétation rhéologique du phénomène. En parallèle, le niveau de corrélation du temps de demi -relaxation ou Tla donne aussi d'excellents résultats avec la longueur finale des produits. Ainsi ce temps de demi-relaxation apparaît comme un paramètre robuste, bien lié à la valeur industrielle intrinsèque de la farine. Dans le cas où les pâtes possèdent des propriétés élastiques prononcées (un k élevé), l'épaisseur de la pâte va atteindre une limite. Ceci peut être expliqué en termes de recouvrance élastique:la pâte, comprimée durant le laminage, entrepose momentanément de l'énergie mécanique, induisant une recouvrance partielle de la déformation. Une valeur k élevée correspond à un procédé de relaxation rapide et par conséquent à un faible niveau d'énergie entreposée et par la suite à un phénomène de recouvrance de la déformation qui vanit.

Une compréhension des mécanismes mis en jeu lors de la détermination des dimensions des biscuits nécessiterait sans doute une approche plus fondamentale. Elle impliquerait, par exemple, une étude des propriétés thermodynamiques des pâtes et de celles du gluten et en interaction avec les autres fractions de la pâte. Ces propriétés sont en effet pressenties comme étant un facteur clé dans la mise en place des dimensions des biscuits, en particulier leurs propriétés mécaniques tels la densité et la résistance à la rupture. Avec les biscuits secs, les caractéristiques finales des produits sont assez différents des cookies, ainsi une faible densité et faible rétraction des biscuits et une texture friable sont des propriétés recherchées, alors que pour les cookies, on réfère essentiellement à leur diamètre et apparence de surface et aussi parfois à leur «snap» qui est associé principalement à la recristallisation du sucre. Les propriétés mécaniques des biscuits telles la force des liens entre les grains et la cohésion de la structure des biscuits peuvent être aussi prédites grâce à la détermination des paramètres de viscoélasticité des pâtes. Ces paramètres mécaniques, quant à eux, sont aussi fortement corrélés avec la densité des biscuits.

Pour définir le rôle fondamental des constituants de la farine, les fractions gluten, amidon de refus et principal ainsi que la fraction hydrosoluble ont été quantifiées pour leur teneur en protéines et pentosanes solubles et insolubles et lipides. En induisant des différences majeures dans la composition des farines reconstituées, il est alors possible de mieux cerner le rôle de chaque fraction et de trouver lesquelles de ces fractions sont responsables des différences dans la valeur technologique de la farine et ainsi déterminer l'impact des constituants déterminants qui vont altérer les caractéristiques des pâtes et biscuits. Cette approche s'est avérée très utile pour déterminer les relations structure/fonction des protéines du blé dur destiné à la panification. En effet, à cause des faibles variations de la teneur en protéines, pentosanes que l'on rencontre avec différents lots de farines, la complexité des recettes biscuitières dont la teneur en gras et sucres peut interférer avec le développement du réseau gluténique et modifier le taux de gélatinisation, il est difficile de bien cerner l'effet « farine». De plus, même si les farines reconstituées produisent des biscuits de taille similaires aux farines natives, elle peut être altérée par une lixiviation manuelle du mélange farine eaucomme discuté antérieurement.

L'utilisation d'un plan de mélange dans lequel les proportions gluten, amidon et fraction soluble répondent à des critères technologiques appropriés d'une farine (machinabilité des pâtes, dimensions et propriétés mécaniques des biscuits acceptables) s'est avérée d'une grande utilité pour mieux cerner «l'effet farine». Nos travaux ont démontré que toute augmentation du gluten, quelque soit la source d'isolation augmente substantiellement les paramètres dureté, consistance, viscosité élongationnelle et viscoélasticité des pâtes. Une confirmation que la teneur en protéines des farines favorise le développement du réseau glutineux et ainsi assure leur élasticité. Cependant la concentration du gluten dans une farine reconstituée ne doit pas dépasser les 25%, sinon de sérieux problèmes de rétraction sont notés. Ces résultats confirment donc que les protéines semblent jouer un rôle important dans l'expression biscuitière des biscuits secs, dû à une teneur en eau plus élevée que les cookies, ce qui favoriserait le développement partiel du réseau gluténique. Les fractions hydrosolubles isolées des trois farines, quant à elles, réduisent considérablement ces mêmes paramètres rhéologiques et ce, à la même concentration du gluten, tout en augmentant l'intensité de la cohésion des pâtes.

Cette étude a aussi démontré, sans équivoque, que la fonctionnalité des farines de blé ne peut être expliqué par la simple sommation des effets individuels de ces constituants. En effet, même si chaque fraction gluten, amidon et fraction soluble possède des caractéristiques biochimiques spécifiques, la valeur technologique de la farine dépend essentiellement des relations structure/fonction des constituants endogènes aux «farines modèles», à leur concentration et aussi aux nombreuses interactions qui prévalent entre eux.

Les différents modèles de régression (linéaire, cubique de Scheffé, quadratique) expliquent les interactions qui prévalent entre les fractions isolées sur les caractéristiques des pâtes et des biscuits secs. Les diagrammes de contour fournissent des synthèses graphiques éloquentes de ces interactions et de l'impact de ces trois fractions sur les paramètres étudiés. Ainsi, par exemple, les paramètres de viscoélasticité Tla et k et de consistance des pâtes produites à partir des farines modèles sont principalement associés à la teneur en gluten et toute addition de la fraction soluble réduisait ces paramètres, à cause de la compétition pour l'eau. L'amidon, de son coté, exerçait un rôle minime sur ces mêmes paramètres. Ces diagrammes nous ont permis de démontrer que les paramètres consistance, dureté et viscosité des pâtes démontraient les même tendances avec les différentes proportions de gluten, amidon et fraction soluble, confirmant que l'usage de l'une ou l'autre de ces méthodes rhéologiques s'avérait approprié pour démontrer l'effet des combinaisons de ces fractions sur le comportement rhéologique des pâtes. Ces trois tests sont, en effet, utilisés individuellement par plusieurs chercheurs pour corréler leurs résultats avec l'étalement des cookies.

La contribution relative de ces trois fractions peut être aussi représentée par l'étude de la section longitudinale d'une iso courbe référée comme les «trace plots». Ces figures démontrent que les réponses rhéologiques et caractéristiques des biscuits des farines reconstituées sont assez similaires; alors que la portion périphérique de la farine ou farine basse génère des tendances très différentes, à cause des propriétés physico-chimiques de beaucoup plus éloignées de celle de la portion centrale.

Le rôle des constituants endogènes associées aux trois farines reconstituées sur les paramètres rhéologiques et caractéristiques des biscuits a aussi permis d'associer le rôle des protéines et pentosanes solubles et insolubles et lipides à ces paramètres. Ainsi la consistance, viscosité élongationnelle et dureté des pâtes produits par la farine basse, étaient surtout expliquées par les pentosanes (corrélation négative), et ces mêmes paramètres démontraient une corrélation positive avec les protéines de la farine patente. Ces observations suggèrent que la qualité et non la qualité des protéines de la portion centrale de l'endosperme semblent jouer un rôle crucial sur ces attributs rhéologiques. Les lipides, étant essentiellement associés aux protéines présentent les mêmes corrélations que ces dernières. Tel qu'anticipé, la viscosité des pâtes est bien expliqué par la teneur en protéines. Quant à l'élasticité des pâtes des farines patente et intermédiaire (Tla), elle est corrélée positivement aux protéines (soluble et insoluble) et aussi négativement aux pentosanes insolubles.

Même si les protéines jouent un rôle important sur la fermeté des biscuits, les pentosanes exercent un effet majeur sur leurs caractéristiques mécaniques, en contrôlant leurs propriétés mécaniques (leur friabilité). Les pentosanes sont donc d'une importance majeur dans l'expression de la qualité des biscuits, probablement à cause de leur forte capacité de sorption de l'eau. Une bonne compréhension de l'effet quantitatif et qualitatif de ces deux constituants permet d'ajuster les caractéristiques des pâtes et aussi la texture des biscuits secs, et cela sans modifier les proportions des ingrédients sucre et gras de la farine dans la recette.

Ces résultats démontrent aussi que les variations dans les caractéristiques dimensionnelles et texturales des produits peuvent être réalisées à l'aide des variations compositionnelles extrêmes, par exemple avec des teneurs en protéines variant de ~ 2 à 14.0%.La formulation de biscuits sans gluten semble donc envisageable.

La teneur en pentosanes des farines est d'une importance capitale pour la qualité des biscuits secs, et en conséquences devrait être inclus dans le cahier de charge des farines de blé biscuitier. Cependant, il reste à mieux définir l'effet du taux de ramification de ces pentosanes sur les caractéristiques étudiées. Mentionnons qu'il aurait été aussi désirable de bien différencier l'effet physique (propriétés de sorption globale pour chaque farine modèle à l'aide du test SRC) des effets de la composition biochimique (concentrations des protéines, pentosanes, amidon endommagé) sur les propriétés des pâtes et caractéristiques des biscuits.

La substitution de la farine commerciale avec les farines patente et basses originales a aussi été évaluée. Cette confrontation pratique a servi à confirmer les effets déterminants des fractions et de leurs constituants, tels que rapportées dans nos études précédentes à l'aide de systèmes «farines modèles». En parallèle, l'impact de la fraction amidon principal et de refus aété comparé, cette dernière étant si souvent rapportée par la littérature comme la fraction responsable de la réduction du diamètre des cookies sugar-snap. La quantification des protéines, lipides et pentosanes de la farine commerciale fortifiée et ou modifiée par substitution a supporté les conclusions précédentes, à l'effet que la teneur en gluten induit une augmentation prononcée des attributs consistance et viscoélasticité pâtes, et que le gluten isolé de la patente se comporte différemment du gluten de la farine basse et cela, à taux de protéines constant, à cause de la qualité de ces protéines, comme le reflète la valeur du SRC-acide lactique, associé avec la force du gluten qui est de 22% inférieur pour la farine basse. Cette augmentation de la teneur en protéines du gluten réduit la longueur des biscuits, favorise une élévation de la densité et fermeté des biscuits secs. Cependant, lorsqu'une certaine teneur en protéines provenant du gluten est atteinte, toute addition supplémentaire du gluten ne s'accompagne pas nécessairement d'une augmentation additionnelle de la densité et fermeté des produits, alors que les attributs rhéologiques continuent à subir des changements plus notables. Les biscuits produits à partir des farines fortifiées avec du gluten apparaissent lisses, sans craques apparentes et de plus, la machinabilité des pâtes s'est améliorée résultant en des pâtes non collantes.Les faibles niveaux d'addition des fractions hydrosolubles dans la farine commerciale (< 5.0%), à cause de la contribution majeure de cette fraction au collant de la pâte ont seulement diminués légèrement les paramètres de viscoélasticité des pâtes et densité des biscuits, sans modifier leurs dimensions. Comme anticipé, cette fraction a exercé un effet important sur la fermeté des biscuits à cause de la teneur en pentosanes. A un niveau de 9% d'incorporation, l'amidon principal a démontré qu'il ne modifiait ni les attributs rhéologiques des pâtes ni les caractéristiques des biscuits qui restent très fragiles tout comme la farine commerciale. Par contre, toute augmentation de l'amidon de refus, résulte en une augmentation excessive de la dureté et consistance de la pâte et de la fermeté et résistance aux bris des biscuits, et cela indépendamment de la source utilisée, probablement à cause de la forte capacité d'absorption de l'eau des pentosanes, leur caractère fortement hydrophile immobilise l'eau libre dans la pâte.

Perspectives de recherche

Ces travaux ouvrent la voie à plusieurs avenues de nouvelles recherches. Il serait, en effet intéressant :

a) d'utiliser une procédure de fractionnement et de reconstitution pour étudier l'impact des fractions gliadines et gluténines et leurs proportions sur les caractéristiques rhéologiques des pâtes et biscuits secs, selon l'approche préconisée dans ce travail. Plus spécifiquement, sur la viscoélasticité des pâtes par la relaxation bi-axiale qui expliquent leurs dimensions et aussi certaines caractéristiques texturales des biscuits secs. Cette approche n'a jamais été confrontée dans la biscuiterie.

b) de mieux saisir l'effet de la teneur en pentosans (totaux et solubles), leur structure (i.e taux de branchement arabinose/xylose) sur les propriétés mécaniques des produits biscuitiers, car ces constituants jouent un rôle crucial dans la biscuiterie.

c) de bâtir des modèles prédictifs expliquant, à partir des propriétés thermomécaniques des pâtes, les changements de dimensions qui se produisent lors des différentes phases de production avec les biscuits secs, surtout en faisant varier les proportions gluten, amidon et fraction soluble. Cette approche a déjà été utilisée avec des modèles cookies.

d) d'étudier l'effet physique, c'est-à-dire absorption de l'eau par les farines versus leurs compositions biochimiques afin de valider les mérites de la méthode SRC pour mieux appréhender l'impact des constituants sur leur aptitude technologique, et surtout de déterminer l'impact de l'absorption de l'eau sur les caractéristiques des cookies

e) d'établir les relations entre analyses mécaniques et sensorielles des produits finis afin de mieux saisir l'effet du Sm et Fs et No

f) de développer des biscuits sans gluten, en exploitant le potentiel des constituants autres que le gluten, pour obtenir des biscuits aux propriétés mécaniques adéquates.

g) de comprendre les des variations qui prévalent dans les proportions des farines patente, de coupure et basse dans une farine commerciale selon les saisons et minoterie, à cause de la variabilité génétique et environnementale, car elles expliquent la valeur technologique d'une farine pour une recette biscuitière donnée. Plus spécifiquement, étudier les variations de la teneur en pentosans et protéines selon les saisons et aussi la concentration de farine basse dans chaque farine commerciale et surtout leur concentration en amidon de refus.

Références bibliographiques

.**Manohar, R.S., and H.P.Rao., 1999a**. Effect of water on the rheological characteristics of biscuits dough and quality of biscuits. Eur.Food. Res. Technol. 209:281-285.
19

AACC. International 2000. Approved methods of the American Association of Cereal Chemists, 10th Ed. Methods 08-01, 44-15A ,54-30, 56-11, 76-30A and 76-31. American Association of Cereal Chemists, St Paul,MN

Abboud, A. M., Rubenthaler, G.l., and Hoseney, R. C. 1985a. Effect of fat and sugar in sugar-snap cookies and evaluation of tests to measure cookie flour quality. Cereal chem.62: 124-129.

Abboud, A.M and R.C. Hoseney, 1985. Differencial scanning calorimetry of sugar cookies dough. Cereal Chem. 61: 34- 37

Abboud, A.M., Hoseney, R.C., and G.L. Rubenthaler, 1985b. Factors affecting cookie flour quality. Cereal Chem. **62**:130-132

Amend, T. and H.D Belitz, 1991. Microstructural studies of gluten and hypothesis on dough formation. Food Structure. 10: 277-288

AOAC. 1970a. Official methods of analysis 31.005, Cookie Moisture,11th Edit.

AOAC.1970b. Official methods of analysis. 922.06. Fat in Flour, Method 32.1.14 In: Cereal Foods, Chap 32, 11th Ed.

Atwell, W., 2001a. Composition of commercial Flour. In Wheat flour. Eagon Press Handbook Series. Chap 3: 27-45

Atwell,W.A, 2001b. Wheat Flour, Eagan Press Handbook Series, St-Paul,MN, chap 2, 15-25

Bache, I.C., and A.M. Donald., 1998. The structure of the gluten network in dough: a study using scanning electron microscopy. J Cereal Sci. 28: 127-133

Badei, A.Z.M., Raouf M.S., and A.S. Wasfy., 1992. Sieve fractionnated wheat flour.II Improved physical and baking properties for biscuit making. Egypt J Food Sci. 20: 85- 97

Baker, J.C., Parker H.K., and M.D. Mize., 1946. Supercentrifugate from dough. Cereal Chem. 23: 16 -30

Baldo B.A., and C.W. Wrigley., 1978. Ig E antibodies to wheat flour components. Clim.Allergy. 8: 109 - 124

Baltsavias, A., Jurgens,A., and T. Von Vliet., 1999. Properties of short dough biscuits in relation to structure, J.Cereal Sc. 29:245-255.

Barr D.J., 1989. The engineering of a modern starch process. In: Wheat is unique by Pomeranz Y ed, American Association of Cereal Chemist, St Paul,MN. Chap 29:502-519

Bartolucci, J.C., and B.Launay, 2000. Stress relaxation of wheat flour doughs following bubble inflation or lubricated squeezing flow and its relation to wheat quality In: Wheat Structure, Biochemistry and Functionality (J.D Schofield,ed.). The Royal Society.Chemistry., Cambridge, 323-331

Beldem SA, Puratos group. Rue Bourrier 12, 5300 Andenne, Belgium. Fax 32 (0) 858 23260.

Belton, P.S., 1999. On the elasticity of wheat gluten. J. Cereal Sci. 29: 103-107

Berger M., 1997. Le cahier de charge des farines.Ind Céréales. 103:5-13

Bernardin, J.E., and D.D. Kasarda., 1973. Hydrated protein fibrils from wheat endosperm. Cereal Chem. **50**: 529-536

Berton, B., Scher, J., and J. Hardy., 2001. Evolution des propriétés physico-chimiques d'une farine de blé au cours de son procédé technologique de fabrication. J.Recent Prog en Génie Procédés. 15 : 539-544.

Berton, B., Scher, J., Viliertas, F., and J. Hardy., 2002. Measurement of hydration capacity of wheat flour:influence of composition and physical characteristics. Powder Technology. 128:326-331

Bettge A.D., Morris, C.F., DeMacon, U.L., and K.K. Kidwell., 2002. Adaptation of AACC method 56- 11 Solvent Retention Capacity for use as an early generation selection tool for cultivar development. Cereal Chem .79: 670-674

Bettge, A.D., Rubenthaler, G.L., and Y. Pomeranz., 1989. Alveograph algorithms to predict functional properties of wheat in bread and cookies baking. Cereal Chem. 66: 81-86

Bettge, A.D., and C.F. Morri., 2000. Relationship among grain hardness pentosan fractions and end use quality of wheat. Cereal Chem. 77 : 241-247

Biarnais, J.P., 1987. Critères de choix des farines en biscuiterie industrielle. Ind Céréales.117: 35-37

Billington,E.W., and A. Tate., 1981. The physics of deformation of flow. Mc Graw-Hill:NY

Bloksma, A.H., and W. Bushuk., 1988. Rheology and chemistry of dough in Pomeranz Y (ed). Wheat chemistry and technology 3 rd ed. AACC, Minnesota 131-217

Bloskma, A.H., and W. Bushuk., 1971. Rheology and Chemistry of dough. In: Pomeranz, Y. (Ed.), Wheat Chemistry and Technology, Chapter 4, American Association of Cereal Chemistry, St Paul, MN, 54-87

Bourne, M.C., 1978. Texture profile analysis. Food Technol. 32: 62-66

Bradbury, D., MacMasters, L., and I.M.Cull., 1956. Structure of mature wheat kernel III , Microscopy structure endosperm of hard red winter wheat. Cereal Chem. 33: 361-373

Branlard, G., Benoualid, K., Rousset, M., Grébault, J., and Y.Seince, 1985. Analysis of a biscuit test: Preliminary study of significant criteria and prediction breeding. Sci. Aliments. 5: 155-172

Brun, J., and B. Mahaut, 1988. Dureté des blés français : Premier blanc d'essai. Ind. Céreales. 54 :13-16

Buré , J., 1975. Techniques meunières du blé à la farine. Econ Med Anim 16: 27-42

Buré, J. 1980. La pâte de farine de blé: la chimie du blé. Collection Promo-ENSIA.Edit S.E.P.A.I.C

Burt, P.J., and T.A. Fearn., 1984. A further quantitative study of biscuits microstructure. FMBRA Bulletin No.3, 99-104.

Burt,D.J., and T.A. Fearn., 1983. Quantitative study of biscuit microstructure. Die starke 35: 351-354

Bushuk, W., 1966. Distribution of water in dough and bread. The Baker's Digest. 40:38-40

Camire, M.E., Camire, A., and K.Krumbar., 1990. Chemical and Nutritional changes in foods during extrusion. Cr. R. Food. Sci Nutrition. 29 : 35-37.

CDAQ., 1992. Les biscuiteries : Guide du Conseil des Denrées Alimentaires du Québec. Comité exécutif Grégoire Y, Constantineau R, Morin G and Aubé C

Cheltel, J.C., and H.Cheftel., 1992. L'eau. Dans : Cheftel JC et Cheftel H (ed), Introduction à la biochimie et à la technologie des aliments 3-32, Lavoisier, Paris (1992).

Chen,W.Z., and R.C. Hoseney., 1995. Wheat flour compounds that produce sticky dough: isolation and identification. J. Food Sci. 60,434-437

Chung O, K., 1989. Functional significance of wheat lipids. Wheat is unique. Dans: American Association of Cereal Chem, St Paul, MN

Chung O.K., Pomeranz, Y., Finney, K.F., and M.D. Shgren., 1978a. Defatted and reconstituted wheat flours. III. Effects of flour moisture content and aqueous binary azeotropes on functional (breadmaking) properties. Cereal Chem. 55: 31-43

Chung, O.K., Pomeranz, Y., and K.F. Finney., 1978b. Wheat flour lipids in breadmaking. Cereal Chem. 55: 598-618

Clements, R.L., 1980. Note on the effect of removal of free lipids in the international structure of cookies by a resin embedding method. Cereal Chem. 57: 445-448

Clements,R.L. and J.R. Donelson., 1981. Functionality of specific flour lipids in cookies. Cereal Chem. 58: 204-206

Cleve,H., and F.Will., 1996. Research with the helph of the Varo Roll. Cereal Sci. Today. 11:128-130,132

Cole, E.W., Mecham, D.K and J.W. Pence., 1960. Effect of flour lipids and some lipid derivatives on cookie baking characteristics of lipid derivatives on cookie baking characteristics of lipid free flours. Cereal Chem. 37: 104-107

Cole,E.W, 1967.Isolation and chromatographic fractionation of hemicellulose from wheat flour. Cereal Chem. 44:411-416

Compoint, G., Marion, D.,Narahisoa., and T. Haertlé., 1994. Role of tryptophan side chain in the interactions of purindolines and their related synthetic tryptophan rich peptides with phospholipids in wheat kernel proteins: Molecular and functional aspects proceeding of the symposium at S. Martino et al Cimino, Uterbo (Italy) 201

Contamine, A.S., Verel, A., Abecassis, J., and B.Vergnes., 1993. Influence des conditions de pétrissage sur les caractéristiques rhéologiques de la pâte biscuitière, in TIFAN. Proceedings, le Croisic .6:16-24

Cornell, J., 1986. A comparison between two ten-point designs for studying three component mixture systems. J .Quality Technol. 18: 1-15

Cornell, J.A., 1990. Experiments with mixture: designs, models and the analysis of mixture data. Second Edition. John Wiley and Sons Inc, New York

Cornell, J.A., and J.M. Harrison., 1997. Models and Designs for experiments with mixtures. Part II-Exploring a subregion of the simplex and the inclusion of other factors in mixture experiments. Department of Statistics, Agricultural Experiment Division, Institute of Food and Agricultural Sciences, University of Florida ,Technical Bulletin 899

Cottenet, M., 1986. Les constituants biochimiques de la farine de blé tendre en relation avec la qualité biscuitière. Sci. Aliments. 6: 305-332

Czuchajowska, Z., and Y.Pomeranz., 1995. Process for fractionating wheat flours to obtain protein concentrates and prime starch, Patent 5, 439, 526

D'Appolinia, B.L and D.R. Shelton., 1984. Carbohydrate functionality in the baking process. Cereal Foods World. 29: 508-510

Daniels, N.W.R., Richmond, J.W., Russel Eggitt, P.W., and J.B.M. Coppock, 1966. Studies of the lipids of flour. III.Lipid binding in breadmaking J. Sci Food Agric.17: 20-29

De La Roche, I., Fowler, D., 1975. Wheat quality evaluation 1. Accuracy and precision of prediction tests, 11. Relationship among prediction tests. Canadian. J. Plant Sci.55:241-249

De Stefanis, V.A., and J.C Ponte., 1976. Studies on the breadmaking properties of wheat flour nonpolar lipids. Cereal Chem. 53: 636-642

Denantés, I., 1987. Incidence de la mouture sur la production d'amidon endommagé. Conséquences sur la qualité des farines, principe de dosage. Ind. Céréales. 46: 23-27

Dexter,J.E., Preston,K.R., Matsuo., R.R and K.H Tipples., 1984. Development of a high extraction flow for the GRL pilot mill to evaluate Canadian wheat potential for the Chinese market. Can Inst Food Sci Technology J. 17:253-259.

Dobraszczyk B.J., 1997. Development of a new dough inflation system to evaluate doughs. Cereal Foods World. 42: 516-519

Doescher L.C, and R.C. Hoseney, 1985. Effect of sugar type and flour moisture on surface craking of sugar snap cookies. Cereal Chem. 62**:** 263-266

Doescher L.C., 1987a A mechanism for cookie dough setting. Cereal Chem. 64: 158-163

Doesher, L., Hoseney, R.C., Milliken, G.A., and G.L. Rubenthaler, 1987. Effect of sugars and flours on cookie spread evaluated by time-lapse photography. Cereal chem. 64:163-167

Donelson, J.R., 1988. The contribution of high protein fractions from cake and cookie flours to baking performance. Cereal Chem 65:389-394

Donelson, J.R., 1990. Flour fraction interchange studies of effects of chlorination on cookie flours. Cereal Chem. 67, 99-100.

Dong, W., 1992. Measurement of certain rheolgical properties and transition in wheat flour. MS Thesis. Kansas State University;Manhattan.

Douglas, S.G., 1981. A rapid method for the determination of pentosans in wheat flour. Food Chem. 7: 139-141

Dresse, P.C., Faubion,J.M., and R.C. Hoseney., 1988. Dynamic rheological properties of flour, gluten, and gluten starch doughs.11. Effects of various processing and ingredients changes. Cereal Chem. 65:354-359

Dubé, R., Indrani, D., Leelavathi, K., and J.S. Sidhu., 1987. Flour millstreams. 1. Physico-chemical and rheological characteristics. Indian Millers. 1 : 17-34

Dubois, M., 1995. La mouture expérimentale du blé tendre. Ind. Céréales. 93:2-21

Eliasson, A.C., and K. Larsson., 1993. Cereals in breadmaking; A molecular colloidal approach. Marcel Dekker NY, chap 6, p 263

Evers, AD., 1970. Development of the endosperm of wheat. Ann Bot. 34: 547-555

Ewart, J.A.D., 1972. A modified hypothesis for the structure and rheology of glutelins. J.Sci.Fd Agric. 23:687-694

Faridi, H., and J.M. Faubion., 1986. Dough rheology: its benefits to cereal chemists. In:Fondamentals of dough rheology, American Association of cereal Chemists,MN,1-9

Faubion, J.M., and R.C. Hoseney., 1990. The viscoelastic properties of wheat flour dough. In Dough rheology and baked product texture.eds H.Faridi and J.M Faubion. Van Niostrand, Reinhold,NY.

Feather, M.S., 1994. The mechanism of non enzymatic browning reactions occuring during cookie and cracker making. In:Faridi.H (ed). The Science of Cookie and Cracker production. Chapman et Hall,N.Y 434-453

Feillet, P., 2000. Le grain de blé : composition et utilisation. INRA ,Paris .

Fellers, D.A., 1973. Fractionation of wheat into major components in industrial uses of cereal. Pomeranz Y eds. American Association of Cereal chemists MN.chap 4: 207- 228

Finney, K.F., 1943. Fractionating and reconstituting techniques as tools in wheat flour research. Cereal Chem 20: 381-396.

Finney, P. L. 1989. Soft wheat. View from the Eastern United States. Cereal Foods World. 34: 682-687.

Finney. K.F., Pomeranz. Y., and R.C. Hoseney, 1976. Effects of solvents extraction on lipid composition, mixing time and bread loaf volume. Cereal Chem. 53: 383-388

Fleckinger, J., 1935. Le grain de blé. Bulletin de l'E.F.M., 36 : 30-35

Flint, O., Moss R., and P.Wad., 1970. A comparative study of microstructure of different types of biscuits and their doughs. Food Trade Rev .40: 32-39.

Fuhr, F.R., 1962. Cookie spread: its effects on production and quality. Bakers Dig. 36:56 -58 , 71

Gaines, C. S., Kassuba, A., and Finney, P. L., 1996. Using wire-cut and sugar-snap formula cookie test baking methods to evaluate distinctive soft wheat flour sets. Implications for quality testing. Cereal Foods World. 41: 155-161.

Gaines, C.S and P.C.Finney, 1989. Effect of selected commercial enzymes on cookie spread and cookie dough consistency. Cereal Chem. 66: 73-77

Gaines, C.S. (2000). Collaborative study of methods for solvent retention capacity profiles. (AACC method 56-11). Cereal Foods World, 45: 303-306.

Gaines, C.S. 1991. Associations among quality attributes of red and white soft wheat cultivars across locations and crop years. Cereal Chem. 68: 56-59.

Gaines, C.S., 1988. Effects of damaged starch, chlorine, flour particle size, dough holding time and temperature on cookie handling properties. Cereal Chem. **65:** 384-387

Gaines, C.S., 1990. Influence of chemical and physical modifications of soft wheat protein on sugar-snap cookie dough consistency, cookie size and hardness. Cereal Chem. 67: 73-77

Gaines, C.S., 1993. Collaborative studies on the baking quality of cookie flour by wire cut type formulations AACC method 10-53 and 10-54. Cereal Foods World 38: 28-30

Gaines, C.S., and J.R. Donelson., 1982. Contribution of chlorinated flour fractions to cake crumb stickiness. Cereal Chem. 59: 378-381

Gaines, C.S., Kasenba A., and P.L. Finney., 1994. Influence of eight flours on the hardness of commercial cookies and crackers. Cereal Foods World. 39: 160, 162,164, 166-167.

Godon, B., 1991. Composition biochimique des céréales A grain d'avoine, blé, mais, orge, triticale dans : Les industries de première transformation des céréales, eds Godon B et Willm C. Tech. doc. Lavoisier eds., Paris 77 -94

Goullieux, A., Allab, K., and J.M. Bouvier., 1995. Application of cone penetrometry to measure texture in biscuits. Sci. Aliments. 15 : 3-18

Grandvoinnet, P., 1991. La valeur meunière des blés, les industries de première transformation des céréales, Godon B et Willim C, eds (tec α Doc, Lavoisier, Paris) P 221-234 Gras P.W and MacRitchie F. (1973) An improved method of volume measurement for small loaves. Cereal Sci Today. 18: 135-137

Graßberger, A., Schieberle P., and P.Köehler., 2003. Fractionation and reconstitution of wheat flour-effect on dough rheology and baking. Eur .Food Res. 216:204-211.

Graveland, A., Bosveld, P., Lichtendonk, W.J., Moonen, H., and A. Schepstra., 1982. Extraction and Fractionation of wheat flour proteins. J. Sci Food Agric. 33: 1117-1128

Graveland, A., Bosfeld, P., Lichtendonk, W.J., Marseille, J.P., and J.H.E Moonen., 1985. A model for the molecular structure of the glutenin from wheat flour. J Cereal Sci. 3: 1-16 .

Greenwood, C.T., 1976. Starch in advances in cereal science and technology ,vol 1, Pomeranz Y eds, Americain Association Cereal Chem, ST Paul, MN. 115-157

Greer,E.N., and J.J.C Hinton.,1950. The two types of wheat endosperm. Nature. 165: 756-748

Gutierrez, A.R., Abecassis, J and B.Cup., 1999. Facteurs physico-chimiques des propriétés d'hydratation des farines. Ind. Céréales. 114: 11-16

Guttieri, M.J., Mclean R., Lanning, S.P., Talbert L.E., and E. Souza., 2002. Assessing environnemental influences on solvent retention capabilities of two soft white spring wheat cultivars. Cereal Chem. 79: 880-884

Hang, W.N., and R.C. Hoseney., 1999. Isolation and identification of a wheat flour compound causing sticky dough. Cereal Chem.76: 276-281

Hareland, G.A.,1994. Evaluation of flours particle size distribution by laser diffraction, sieve analysis and near infrared reflectance spectroscopy. J Cereal Sci . 21: 183-190

Hibberd,G.E., 1970. Dynamic viscoelastic behaviour of wheat flour doughs. Part 11. Effect of water content in the linear region. Rheol.Acta 9:497-500.

Holas, J., and K.H. Tipples., 1978. Factors affecting farinograph and baking absorption. I. Quality characteristics of flour streams. Cereal Chem. 55: 637-652.

Hong, B.H.,Rubenthale, G.L and R.E. Allen., 1989. Wheat Pentosans. 1. Cultivar variation and relationship to kernel hardness. Cereal Chem.66:369-373

Hoseney, R. C., 1994. Principles of Cereal Science and Technology, 2nd ed. American Association of Cereal Chemists, St- Paul, MN. Chapter 7.

Hoseney, R.C, Wade, P, Finley, J.W., 1988. Soft wheat products. In: Pomeranz, Y. (Ed.), Wheat Chemistry and Technology, Vol 2, 3rd ed., American Association of Cereal Chemistry, St Paul, MN, pp. 407-456.

Hoseney, R.C., and D.E. Rogers., 1994 . Mechanism of sugar, functionality in cookies, p203-226. In The Science of cookie and cracker production, Faridi H.et. N.Y

Hoseney, R.C., and L. Krishnarau., 1994. Enzymes increase loaf volume of bread supplemented with starch tailings and insoluble pentosans. J Food Sci. 59:1251-1254

Hoseney, R.C., Atwell W.A., and R. Miller., 1997. Gluten Proteins In: Understanding wheat flour quality. AACC Short Course San Diego CA 10-11

Hou G., Yamamoto H., and P.K.W. Ng., 1996. Relationship of quantity of gliadin subgroups of selected US soft wheat flours to rheological and baking properties. Cereal Chem 73: 352 -357 and 73: 358-363

Ishida, K.I., and M. Nagasaki., 1989. Use of protease in biscuits processing. Nippon Shokuhin Kogyo Gakkaishi. 36: 964-967

Izydorczyk, M., and C.G Biliaderis., 1995. Cereal arabinoxylans : advances in structure and physicochemical properties. Carb. Polymers. 28: 33-48

Izydorczyk,M., and C.G Biliaderis., 1992. Effect of molecular size on physical properties of the arabino xylans . J. Agric. Food Chem. 40: 561-568

Jelaca, S.L., and I. Hlynka., 1971. Water binding capacity of wheat flour crude pentosans and their relation to mixing characteristics of dough. Cereal Chem. 48: 211-222

Jeltema, M.A., Zabik ,M.E., and L.J. Thiel., 1983. Prediction of cookie quality from dietary fibres components. Cereal Chem. 60: 227-230

Jurgens A., MaarSchalkerweerd, T.V.P and J.F.C. Rottier., 2000. Gluten microstructure and changes in hard biscuit doughs as determined by light microscopy and rheology. In Wheat structure biochemistry and functionality. Journal David Schofield Royal Society of Chemistry Greet Britain ,332-339

Kahn, K., and W. Bushuk., 1979. Studies of glutenin XII. Comparison by SDS-PAGE of unreduced glutenin from various isolation and purification procedures. Cereal Chem 56: 63-68

Kaldy, M.S., and G.L. Rubenthaler, 1987. Milling,baking and physical chemical properties of selected soft white and spring wheats. Cereal Chem. 64: 302-307

Kaldy, M.S., Kereliuk, G.R., and G.C. Kozub, 1993. Influence of gluten components and flour lipids on soft white wheat quality. Cereal Chem. 70: 77-80.

Kaldy, M.S., Rubenthaler, G, I., Kereliuk, G.R., Berhow,M.A.,Vandercook, C.E.,1991. Relationship of selected flour constituents to baking quality in soft white wheat. Cereal Chemistry 68, 508-512.

Kent, N. L., and A.D. Evers., 1969. Variation in protein within the endosperm of hard wheat. Cereal Chem . 46: 293-300.

Kiger, J.L and J.G Kiger., 1967. Techniques modernes de la biscuiterie-pâtisserie, boulangerie industrielle et artisanale et des produits de régime.Tomes I et II, (Dunod, Paris), 595p

Kim, S.S., 1990. Fractionation of polydextrose and hemicellulose by gel chromatography and the effects of fractions on starch thermal transitions. PhD. Dissertation, Kansas state Univ Manhattan KS

Kissel, R.I., Pomeranz, Y., and W.T.Yamazaki., 1971. Effects of flour lipids on cookie quality. Cereal Chem .48: 655-658

Kőehler, P., 2003. Fractionation and reconstitution of wheat flour effect on dough rheology and baking. Eur. Food Technol. 216: 204-211

Köehler, P., Graßberger, A. and Schieberle P.,1999. Fraktiotioierung und recombination von weiszenmehl einfluss auf teigrheologie und backverhalten. Getreide Mehl und Brot.53, 16-19

Krishnarau, L., and R.C. Hoseney., 1994a. Enzymes increase loaf volume of bread supplemented with starch tailings and insoluble pentosans. J.Food Sci. 59:1251-1254

Krishnarau, L., and R.C. Hoseney., 1994b. Acid-hydrolyzed starch tailings effects on cookie spread. J.Food.Sci 59:1255-1257 and 1270

Kruger, J.E., 1981. Severity of sprouting as a factor influencing the distribution of α-amylase in pilot mill streams. Can J Pl Sci. 61:817-828.

Krull,L.H., and G.E. Inglett.1971. Industrial use of gluten. Cereal Sci. Today.16:232-236,261

Kuhn, M.C., and W.Grosch., 1985. fraktionierung und rekonstituierung von roggenmehl eine tecknik zur untersuchung von NSP hydrolasen bei der roggenbrotherstellung. Getreide Mehl Brot 39: 340-345 et 1270

Kulp, H., 1994. Cookie Chemistry and Technology. American Institute of Baking, Manhattan, Kansas, pp118-132

Labuschagne, M.T., Brooks Coetzee, M.C., and C.S.Van Deventer., 1996. Biscuit making quality prediction using heritability estimates. J.Sci. Food Agric. 70: 25-28

Larkin, R.A., Mac Masters, M.M., and C.E. Ris., 1952. Relation of endosperm cell wall thickness to the milling quality of seven pacific northwest wheats. Cereal Chem. 29: 407 - 413

Larsson, H., and A.C. Eliasson., 1996a. Phase separation of wheat flour dough studied by ultracentrifugation and stress relaxation I. Influence of water content. Cereal Chem. 73: 18-24

Larsson, H., and A.C. Eliasson., 1996b. Phase separation of wheat flour dough studied by ultracentrifugation and stress relaxation. II. Influence of mixing time, ascorbic acid and lipids. Cereal chem. 73:25-31

Laszitity,R., 1984. The chemistry of cereal proteins. CRC Press:Boca Raton,FL

Launay, B., and Bartolucci, J.C., 1997. Caractérisation du comportement viscoélastique des pâtes de farine en extension biaxiale. Cahiers de rhéologie XV., 594-607 (ENSIA, Massy)

Le Roux, C., 1987. Contribution à l'étude des interactions lipides-protéines dans les produits céréaliers de caisson: propriétés physico-chimiques des lipides endogènes et exogènes dans les pâtes et le gluten de blé tendre. Thèse de Doctorat, Université de Nantes

Levine, H., and L. Slade., 1990. Influence of glassy and rubbery states on the thermal mechanical and structural properties of dough and baked products. In:Faridi H, Faubion JM (ed), Dough rheology and baked product texture, Van Nostrand NY 157-330

Levine, L., and B.A. Drew., 1994. Sheeting of cookie and cracker dough. In: Faridi, H. (Ed.), The Science of Cookie and Cracker Production, Chapman and Hall, New York, pp353-363.

Lineback D.R., and E.Wongsrikasen., 1980. Gelatinization of starch in baked products. J.Food Sci. 45: 71-75

Lorenz, K., 1986. The importance of flour particle size on the mechanical dough development bread process. N. Z. J Technol. 2: 27-31

Lukow, O.M., and S.Uthayakumaran., 2003. Functional and multiple end use characterisation of canadian wheat using a reconstituted dough system. J. Sci Food Agric. 83: 889-898

Maache Rezzoug, Z., Bouvier J.M., Allaf K and J. Tayeb., 1998a. Comportement rhéologique en compression uniaxiale et qualité technologique des pâtes biscuitières. Sci. Aliments. 18 : 393-407

Maache Rezzoug, Z., Bouvier, J.M., Allaf, K., Tayeb, J. and Patras C.,1998b. Effects of principal ingredients on rheological behaviour of biscuis dough and on quality of biscuits. J. Food Eng 35: 23-42

Maache, Rezzoug. Z., Allaf, K., Bowler, Y. M.., and Y.Tayeb., 1998c. Relations entre caractéristiques physiques et propriétés rhéologiques de la farine et pâte pâtissière. Sci. des Aliments. 18: 267-281.

Maartens, M., and H. Martens., 1986. Partial Least square regression. Ch 9 in Statistical Procedure in Food research, J.R Piggot (Ed), p 293-359. Elsevier Applied Science, London.

MacMasters, M.M., and G.E. Hilbert., 1944. The composition of the amylodextrin fraction of wheat flour. Cereal Chem. 21: 548-555

MacMasters,M.M., Hilton, J.J.C. and D. Bradbury., 1971. Microscopic structure and composition of wheat kernel. In: Wheat Chemistry and Technology (2): 51-113, St-Paul,MN,AACC

MacRichtie, F., 1985. Studies of the methodology for fractionation and reconstitution of wheat flours. J. Cereal Sci. 3:221-230.

MacRitchie, F., 1976. The liquid phase of dough and its role in baking. Cereal Chem. 53: 318-326

MacRitchie, F., 1977. .Flour lipids and their effects in baking. J. Sci Food Agric. 28: 53

MacRitchie, F., 1980. Flour lipids: Theoretical aspects and functional properties. Cereal Chem. 58: 156-158

MacRitchie, F., 1992. Physical chemical properties of wheat proteins in relation to functionality. Adv Food Nuhe Res. 36: 1-87

MacRitchie,F., 1987. Evaluation of the contribution from wheat protein fractions to dough mixing and breadmaking. J.Cereal Sc. 6:259-268

Manohar, R.S., and H.P Rao., 1997. Effect of mixing period and additives on the rheological characteristics of dough and quality of biscuits. J. Cereal Sci. 25:197-206.

Manohar, R.S., and H.P. Haridas., 1994. Interrelationship between rheological characteristics of dough and quality of biscuit: Use of elastic recovery to predict biscuit quality. Food Res Int . 9963-9969

Manohar, R.S., and H.P. Rao., 1999c. Effect of mixing method on the rheogical characteristics of biscuits dough and the quality of biscuits. Eur Food Res. Technol. 210: 43-48

Manohar, R.S., andH.P.Rao., 1999b. Effect of emulsifiers, fat level and type on the rheogical characteristics of biscuits dough and quality of biscuits. J.Sci.Food. Agric. 79:1223-1231.

Mansour, K., 1982. Quality control in soft wheat flour. Cereal Foods World. 27: 315 -318

Martin, G., 1990. Chaleur , sécheresse et qualité des blés . Ind.Céréales. 68 : 13-16

Masi, P.E., Cavella,S., and M. Sepe., 1998. Characterization of dynamic viscoelastic behaviour of wheat flour doughs at different moisture contents. Cereal Chem. 75:428-432

Mattern,P.J. and P.M. Sandstead., 1957. The influence of the water soluble constituents of wheat flour on its mixing and baking characteristics. Cereal Chem. 34, 252-267

Mauritzen, C.M., and P.R Stewart., 1965. The ultracentrifugation of dough made from wheat flour. Aust J Biol Sci. 18: 173-189

Mecham, K.H.,1968. Changes in flour during mixing. Cereal Sci. Today. 13:371-375.

Medcalf, D.G., and K.A. Gilles., 1968. Structural characterization of a pentosan from the water insoluble portion of durum wheat endosperm. Cereal Chem. 45: 550-556

Menjivar,J.A and H.Faridi., 1994. Rheological properties of Cookies and Crackers Doughs. In: The Science of Cookies and Cracker Production.Ed.H.Faridi, Chapman and Hall, 283-322

Meuser, F., and P.Suckow., 1986. Non starch polysaccharides. In «Chemistry and Physics of baking» Blanshard J.M.V., Frazier P.S.,Gaillard,T .Eds., 4:42-61.

Meuser, F.F., Althoff, F.,and H. Huster.,1989. Developments in the extraction of starch and gluten from wheat flour and wheat kernels, chap 28. In: Wheat is unique, Y Pomeranz, ed. Am.Assoc. Cereal Chem., St Paul, MN, p 479-500

Miflin, B.J., Field , J.M., and P.R. Shewry.,1983. Cereal storage proteins and their effects on rheological properties in seed proteins. Daussant Journal, Masse Journal, Vaughran Jour

Miller, K. A., Hoseney, R. C., and C.F. Morris, 1997. Effects of formula water content on the spread of sugar-snap cookie. Cereal Chem. 74: 669-671.

Miller, K.A and R.A.Hoseney., 1997a. Factors in hard wheat flour responsible for reduced cookie spread. Cereal Chem .74: 330 -336

Miller, K.A., 1985. The use of the penetrometer to measure the consistency of short dough 117-132. In: Rheology of wheat products edited by Faridi H, AACC, St Paul

Miller, K.A., and R.A. Hoseney, 1999. Dynamic rheological properties of wheat starch gluten doughs. Cereal Chem. 76: 105-109

Miller, K.A., and R.C Hoseney, 1997b. Use of elongational viscosity to estimate cookie diameter. Cereal chemistry. 74:614-616

Miller, K.A., Mattew, R., and R.C Hoseney, 1996. Use of thermomechanical analyser to study the apparent glassy transition in cookie dough. J. of Thermal Analysis. 47: 1329-1338

Moenen, H., 1998. Enzymes for the production of baked foods. Getreide Mehl und Brot. 52 : 270 -272

Morris, C.F., and S.P Rose., 1996. Wheat in cereal grain quality. Chapman and Hall, London edited by R.T Menry and P.P Kettwell 3-54

Morrison, W.R., and K.D. Hargin., 1981. Distribution of soft wheat kernel lipids into flour milling fractions. J. Sci. Food Agric. **32**: 579-587.

Morrisson, W.R., Hargin, K.D., 1981. Distribution of soft wheat kernel lipids into flour milling fractions. Journal of the Science and Food of Agriculture 32, 579-587.

Morrisson,W.R., and P.J Barnes., 1983. Distribution of wheat acyls lipids and tocols in mills treams. In: Lipids in cereal technology , pp149-164., London; Academic Press

Myers, R. H., and D.C. Montgomery., 1995. Response Surface Methodology. Process and Product Optimization Using Designed Experiments. John Wiley & sons. New York.

Nelson, P. N., and C.E McDonald.,1977. Properties of wheat flour protein in flour from selected mill streams. Cereal Chem. 54: 1182-1191.

Nemeth, L. Y., Williams, P. C., and Bushuk, W., 1994. A comparative study of the quality of soft wheat from Canada, Australia and the United States. Cereal Foods World. 39: 691-700.

Nikolaidis, A and T. Labuza., 1996. Glass transition state diagram of a baked cracker and its relationship to gluten. J Food Sci. **61**: 803-806

Nuret, H ., 1994. Soft wheat milling. Chap 13, 269-290 In: Primary cereal processing- A comprehensive review book, VCH publishers Inc.

Nuret, H., 1991. La mouture de blé tendre, In : les industries de première transformation des céréales, Godon B et Willm C, eds (Tec et Doc , Lavoisier, Paris 333-361

Olcott, H.S., and D.K. Mecham., 1947. Characterization of wheat gluten I- protein-lipid complex formation during doughing of flours, lipoprotein nature of the glutenin fraction. Cereal Chem. 24: 407-410

Olewnik,M.C and K. Kulp..1984. The effect of mixing time and ingredient variation on farinograph of cookie doughs. Cereal Chem. 61:532-537

Oliver, G., 1995. Predicting the processing performance of biscuits doughs. Food Tech. Int. Eur 107-109

Oliver, G., Thacker, D., and R.J. Whealer., 1995. Semi sweet biscuits : The influence of sodium metabisulfite on dough rheology and baking performance. J Sci Food Agric. 69: 141-150

Osborne, T.B., 1907. The proteins of wheat kernel. Publ. No 84. Carnegie Institut Washington, D.C

Payne P.E., Nighingale,M.A., Krattiger,A.S and L.M Holt., 1987. The relationship between HMW glutenin subunits composition and the bread making quality of british-grown wheat varieties. J.Sci.Food and Agric. 40:51-55

Perlin, A.S and S.Suzuki., 1965. A note on the polysaccharides of wheat flour squeegee. Cereal Chem. 42: 199- 201

Petrofsky, H.E., and R.C.Hoseney., 1995. Rheological properties of dough made with starch and gluten from several cereal sources. Cereal Chem. 72 :53-58

Pomeranz, Y., 1977. Theorie and practice of determining the disparate effects of individual wheat flour constituents on baking performance. Getreide Mehl und Brot. 31:147-152

Pomeranz, Y., 1988. Composition and functionality of wheat flour components. In : wheat chemistry and technology Pomeranz Y eds. AACC,MN, 219

Pomeranz,Y., 1980. What? How much? Where? What function? in breadmaking. Cereal Food World. 25: 656-662

Pomeranz,Y., 1980. What? How much? Where? What function? in breadmaking. Cereal Food World. **25**: 656-662

Popineau, Y., 1996. Propriétés biochimiques et physico-chimiques des protéines de céréales in : protéines végétales (Lavoisier, ADRIA, Paris) 161-211

Popineau, Y., and S.Denery-Papini., 1996. Les protéines de reserve du grain de blé. In proteins vegetales. Godon B, ed, Lavoisier, Tec et Doc,Paris 120-172

Posner, S.E, and Hibbs., A.N., 1997. Wheat flour milling. American Association of Cereal Chemists, Chap **25**, p257 -271

Potus, J., Amrani, E .F., Ameille, V., and N. Raid., 1996. Les hydrolases en panification. Ind Céréales. 96 :7-12

Prabhasankar, P., Sudha,M.l., Rao H.P., 2000. Quality characteristics of wheat flour milled streams. Food Res. Int. 33, 381-386

Pritchard, P.E., and Brock C.J., 1994. The glutenin fraction of wheat protein: the importance of genetic background on its quantity and quality. J.Sci. Food.Agric. **65:**401-406

Pritchard,P.E., and Bhandary, D.G., 1992. The glutenin fraction of wheat protein: biochemical studies and its importance in the bread making process. MAFF Commission-Project report no. F9-89-3

Ranhottra, G.S., Gelroth,J.A., Astroth,K. and Posner ,E.S., 1990. Distribution of total and soluble fiber in various mill streams of wheat. J. Food Sci. 55, 1349-1357.

Rasper, VF, Pico ML and R.G.Fulcher., 1986. Alveography in quality assessment of soft white winter wheat cultivars. Cereal Chem. 63: 395-400

Renard, C and S.Théry., 1998. Détermination des méthodes physico-chimiques pour prédire la qualité biscuitière des blés français. Ind. Céréales. 109:31-36

Robinson,I.M., Dockerty,J.F., and T. Fearn., 1984. The conditioning of wheat-The influence of conditioning temperature on flour yield, moisture content and color. J. Sci. Food Agr. 35:1340-1346.

Roels, S.P., Cleemput,G., Vandewalle, X., Nys,L., and J.A Delcour.,1993. Bread volume. Ceral chem.. 70:318-323

Rogers, D.E., Hoseney, R.C., Lookhart, G.L., Curran, S.P., Lin, W.D.A., and R.G. Sears., 1993. Milling and cookie baking quality of nearisogenic lines of wheat differing in kernel hardness. Cereal Chem. 70:183-187.

Rouau, X., 1993. Investigations into the effect of an enzyme preparation for baking on wheat flour dough pentosans. J Cereal Sci. 18: 145-157

Rouau, X., 1996. Les hemicellulases en panification. Ind.Céréales . 96 : 13-

Rouau, X., and A. Surget., 1994. A rapid semi automated method for the determination of total and water extractable pentosans in wheat flours. Carb. Polymers. 24: 123-132

Rouau, X., El Hayek, M.L., and D. Moreau., 1992. Effect of an enzyme preparation containing pentosanases on the bread making of flour in relation to changes in pentosan properties. J Cereal Sci. 19: 256-272

Rouau,X., and L. Saulnier., 1994. Les polysaccharides pariétaux des grains de céreales. Session de formation INRA

Sandsted, R.M ., 1961. The function of starch in the baking of bread. Baker's Digest. 35: 36-44

Sapirstein, H. D., and Johnson, W.J. 2000. A rapid spectrophotometric method for measuring insoluble glutenin content of flour and semolina for wheat quality screening.

Wheat gluten: Proceedings of the 7th International Workshop Gluten 2000. University of Bristol, 307-312.

SAS .,1999. Version 8.01, SAS Institute, Cary,NC

SAS Institute Inc., 2004. Getting started with the SAS 9.1 ADX Interface for Design of Experiments, SAS Institute Inc., Cary, NC.

Saulnier,L.,Pepneau N., and J.F.Thibault.,1995. Variability in grain extract viscosity and water soluble arabinoxylan content in wheat. J.Cereal Sc. 22:259-264

Shanot, M.A., 1981.Sweetners functionality in cookies and crackers. A.I.B. Res. Dept. Tech. Bull vol .3 N .4. Am. Inst. Baking, Manhattan KS

Sharp, T., 2001. The key aspects of biscuits quality: Interactions of the major Ingredients-Flour and fat. Report no.133 Campden &Chorleywood Food Research Association Group, Gloucestershire, UK

Shewry, P.R., Halford, N.G., and A.S. Tacham., 1992. High molecular weight subunits of wheat glutenin. J Cereal Sci .15: 105-120

Simpson, F.J., 1954. Microbial pentosanes. A survey of microorganisms for the production of enzymes that attack the pentosans of wheat flour.Can J. Microb. 1:131-139

Sipes, K., 1993. Factors affecting protein and starch interaction. MSc thesis ,Kansas City state University, Manhattan

Slade, L., and H. Levin., 2000. AACC course. Food Polymers Science. Approach to ingredients functionality in cookie and cracker baking technology.

Slade, L., and H. Levine., 1994a. Aspects of soft wheat quality of current interest to a cookie/cracker baker. 1 and 11. Cereal Foods World. 39:640-643.

Slade, L., and H. Levine., 1995. Water and glass transition. Food Eng. 24: 431 – 509

Slade, L., and Levine, H. 1994b. Structure-function relationships of cookie and cracker ingredients. In The science of cookie and cracker production, Faridi, H., ed. (Chapman and Hall, London), 23-142.

Slade, L., Levine, L., and J.W Finley., 1989. Protein water interactions : water as plasticizer of gluten and others protein polymers. In: Influence of processing on food proteins. D Phillips and JW Finley eds, Marcel Dekker NY (1989).

Slade, L.,Levine, H., Craig, S., and H. Arciszewk., 1994. Reducing checking in crackers with pentosanase. US patent 5: 362-502

Sollars ,W.F., 1959. Effects of the water soluble constituents of wheat flour on cookie diameter. Cereal Chem. 36: 498-513

Sollars, W.F. 1969. Flour constituents affecting Mac Michael viscosity. Cereal chem.6: 44-55

Sollars, W.F., 1956a. A new method of fractionating wheat flour. Cereal Chem. 33:111-120

Sollars, W.F., 1956b. Evaluation of flour fractions for their importance to cookie quality. Cereal Chem. 33: 121-128

Sollars, W.F., 1958. Fractionation and reconstitution procedures for cake flours. Cereal Chem. 35: 85-99

Sollars, W.F., and S. M Bowie., 1966. Effect of the subfraction of starch tailings on cookie diameter . Cereal Chem. 43: 244-260

Souza, E., 1998. Soft wheat with quality to improve cookie and cracker texture. Technical paper presented at 73rd meeting of the Biscuit and Cracker Manufactures Association (BMCA)

Souza, E., Kruk, M., and D.W. Sunderman, 1994. Association of sugar snap cookie quality with high molecular weight glutenin alleles in soft white spring wheats. Cereal Chem. 71: 601-605

Stauffer,C., 1996. How flour functions. Baking and snack 47-48

Steele, R.G.D., and J.H.Torrie., 1980. Analysis of variance 1: the one-way classification. In: Principles and procedures of statistics. McGraw-Hill, New York.

Tanaka, K and W. Bushuk., 1973. Changes in flour proteins during dough mixing .III: Analytical results and mecanisms. Cereal Chem. 50: 605-612

Tatham, A.S., Shewry, P.R., and P.S Belton., 1990. Structural studies of cereals prolamines including wheat glutens. In Advances in Cereal Science. Pomeranz Y eds, American Association of Cereal Chemistry, St Paul 10: 1-78

Tharrault, J. F., 1994c. Caracterisation de l'écoulement d'une pâte biscuitière : l'essai d'extrusion. Bulletin de liaison du CTUC. 15: 49-55.

Tharrault, J.F., 1994a. Actualisation d'un test de cuisson biscuitier. Industries des Céréales, 87 : 36-39.

Tharrault, J.F., 1994b. Actualisation d'un test de cuisson biscuitier. Bulletin de liaison du CTUC. 15 : 23-28..

Tharrault, J.F., 1995. Qualité biscuitière des farines de blé tendre: des blés biscuitiers pour une bonne maîtrise de la texture des biscuits. Bulletin de liaison du CTUC, 19 :16-24.

Tharrault, J.F., 1997. Qualité biscuitière des farines de blé tendre : des blés biscuitiers pour une bonne maîtrise de la texture des biscuits guide pratique d'analyses dans les industries des céréales. Godon B et Loisel W, eds (Paris) 623-634

Tjomb, P., 1995. Boulangerie-biscuiterie : de nouvelles applications. RIA. 544:88-89.

Tolstogusov, V., 1997. Thermodynamic aspects of dough formation and functionality. Food hydrocolloids 11, 1181-1193.

Tolstoguzov,V., 2002. Thermodynamic aspects of biopolymer functionality in biological systems, foods and beverages. Crit Rev Biotechnol .**22**: 89-174

Tsen, C.C., 1967. Changes in flour proteins during dough mixing. Cereal Chem. 44: 308-317

Upton,E.M ., and E.E. Hester., 1966. Non-starchy polysaccharides and proteins of soft wheat flour tailings. Cereal Chem. 43: 156-168

Uthayakumaran, S., and O.Lukow., 2003. Functional and multiple end-use characterisation of Canadian wheat using a reconstitute dough system. J. Sci. Food Agr. 83:889-898.

Van Der Borght, A.,Goesaert,H.,Veraverbeke,W.S., Delcour, J.A., 2005. Fractionation of wheat and wheat flour into starch and gluten: overview of the main processes and the factors involved. J. Cereal Sci. 41: 221-237.

Varriano-Marston, E., KE., V., Huang, G., and J.R. Ponté., 1980. Comparison of methods to determine starch gelatinization Cereal Chem. 57**:** 242-248.

Verel, A., 1996. Quel blé tendre pour la biscuiterie? Process. 1116:54-56.

Wade ,P.,1972a. Action of sodium metabisulphite on the properties of hard sweet biscuit doughs. J. Sci. Food Agric. 22: 333-338

Wade, P., 1972b. Flour properties and the manufacture of semi-sweet biscuit. J Sci Food Agric. 23:737-744

Wade, P., 1988. Principle of the craft. Biscuit cookie and crackers. Elsevier Applied Science, London, vol 1, 2, 3,176p

Wade, P., 1995. Biscuits, Cookies and Crackers, volume 1. Blackie Academic and Professional, 2nd Ed., Glasgow,118-135.

Wainwright, A.R., Cowley, K.M., and P. Wade., 1985. Biscuit making properties of flours from hard and soft milling single variety wheats. J Sci Food Agric. 36: 661-668

Warthesen, J.J., 1995. Cereals : The source of ingredients for baking. Cereal Foods World. 40: 14-15

Weegels, P.L., Hamer, R.J., and J.D Schofield., 1996a. Critical review: Functional properties of wheat glutenin. J Cereal Sci. 23: 1-18

Weegels, P.L.,Van de Pijpekamp, A.M., Graveland, A., Hamer, R.J., and J.D Schofield.,1996b. Depoplymérisation and repolymerisation of wheat glutenin during dough processing. I-Relationship between glutenin macropolymer content and quality parameters. J Cereal Sci . 23: 103-111

Wieser, H., Seilmeier, W.,and H.D. Belitz.,1989. Extractability of glutenins with water and alcohol mixtures under reducing conditions. Z.lebensm Unters Forch. 189: 223-228

Willm, C., 1995. Comportement en mouture de 9 variétés de blé tendre I. Influence de la dureté et de l'apport azoté. Ind.Céréales. 92: 18-29

Willm, C., and S. Joliet., 1994. Préparation du blé à la mouture. Ind .Céréales. 86:12-26

Willm, C.,1992. New process for new products. In: Cereal chemistry and technology : A long past and a bright future, 9th International Cereal and Bread congress, Paris 1-5,95-110

Wu, J.Y., and R.C. Hoseney., 1990. Rheological changes in cracker sponges during the first five hours of fermentation. J Food Process Preserv .14: 17-26

Yamamoto, H.,Worthington, S.T., Hou ,G ., and P.K.W. Ng., 1996. Rheological properties and baking quality of selected wheats grown in the United States. Cereal Chem. 73: 215-221

Yamazaki, W.T. and Donelson, J.R., 1976. Effects of interactions among flour lipids, others flour fractions and water on cookie quality. Cereal Chem. 53: 998-1002.

Yamazaki, W.T., 1953 An alkaline water retention capacity for the evaluation of cookie baking potentialities of soft winter wheat flours. Cereal Chem. 30: 242 -246

Yamazaki, W.T., 1954. Interrelations among bread dough absorption, cookie diameter and AWRC of soft wheat flours. Cereal Chem. 31:135-142

Yamazaki, W.T., 1955. The concentration of a factor in soft wheat flour affecting cookie quality. Cereal Chemistry, 32: 26-37.

Yamazaki, W.T., 1959a. Flour granularity and cookie quality.I .Effect of wheat varieties on sieve fractions properties. Cereal chem. 36: 42 – 51

Yamazaki, W.T., 1959b. Flour granularity and cookie quality.II. Effect of changes in granularity on cookie characteristics. Cereal chem. 36: 52-59

Yamazaki, W.T., 1969. Soft wheat flour evaluation. Bakers Digest. 43: 30 - 33 and 63

Yamazaki, W.T., and J.R. Donelson., 1983. Kernel hardness of some US wheat. Cereal Chem. 60: 344-350

Yamazaki, W.T., Donelson, J.R., Knowlek, W.F., 1977. Effects of flour fraction composition on cookie diameter. Cereal Chem..54:352-356.

Ziegler,E., and E.N.Greer.,1978. In: Y Pomeranz, Wheat chemistry and technology, St-Paul, MN: American Association of Cereal Chemists. 115-199.

Zoulias, E., Oreopoulou, V., and E.Kounalaki., 2002.Effect of fat and sugar replacement on cookie properties. J Sci Food Agric. 82:1637-1644.

ANNEXE

Matériels et Méthodes

1. Fractionnement des farines en leurs constituants : gluten, amidon et fraction hydrosoluble

La procédure d'extraction utilisée a été adaptée d'après les travaux précédents de MacRitchie (1985). La première étape consiste à former le réseau de gluten en préparant une pâte avec 210 g de farine et 100 g d'eau à l'aide d'un malaxeur (National Mfg Co. Lincoln, Nebraska) pendant 60 s. Cette pâte est ensuite placée dans un seau de 3 l dans lequel sont ajoutés 500 ml d'eau distillée. La pâte est alors malaxée lentement à la main afin de maintenir le réseau de gluten le plus compact possible pendant une période variant entre 3 et 8 minutes selon les manipulateurs. Six lavages successifs sont ainsi effectués. Les eaux de rinçage sont récupérées dans un contenant après passage dans un tamis de 355 μm afin de récupérer les éventuels résidus de gluten. Ceux-ci sont alors réincorporés au sein de la boule de gluten formée. L'ensemble du gluten récupéré est découpé en morceaux (1 cm^3) puis étalé sur un plateau de lyophilisation et mis à congeler à –40 °C.

Les eaux de rinçage sont alors placées à 4° C pendant 2 h pour permettre une première séparation de l'amidon de la fraction soluble, par simple décantation, puis centrifugées à 5000g for 10min (rotor JA-10, model 12-21. Beckman, Spinco Division, Palo Alto, California) Le surnageant (fraction soluble) est alors récupéré et directement mis sur des plateaux de lyophilisation. Le résidu aqueux trouble est centrifugé à 7, 500 rpm pendant 10 min (rotor JLA-10; Model J2-21, Beckman, Spinco division, PaloAlto, California) pour séparer la fraction soluble de l'amidon (phase solide). L'ensemble amidon et fraction soluble est mis à congeler à –40° C avant de procéder à une lyophilisation à 18° C pendant 48 h.

Chaque fraction récupérée (exceptée la fraction soluble) est soumise à un moulin de mouture centrifuge (Brinkman Retsch, Model LB 49) en s'assurant que la température de la poudre ne dépasse pas 30° C afin d'éviter toute dénaturation des protéines. Par la suite, les échantillons de poudre passent à travers un tamis de 250 μm. Chaque fraction est pesée afin de déterminer le rendement de l'opération d'extraction ainsi que leur pourcentage respectif dans la farine de départ. Ces valeurs sont alors corrigées par le taux d'humidité déterminé au préalable. Le pourcentage de chacune des fractions exprimé en ms est calculé par la relation suivante :

$$\text{Pourcentage de fraction} = \left[\frac{M_e * (1 - T_e)}{M_f}\right] * 100$$

M_e : masse de la fraction considérée obtenue après lyophilisation

T_e : taux d'humidité de la fraction considérée

M_f : masse sèche totale des échantillons obtenue après lyophilisation provenant de la même farine de départ

Les fractions sont ensuite scellées hermétiquement dans des sacs d'aluminium et entreposés à 4° C avant utilisation. Lorsque l'ensemble des fractions a été broyé, tous les lots de même provenance sont combinés au moyen d'un mélangeur Hobart, Modèle N50 (Hobart Corporation) durant 5 min afin de réduire les variabilités éventuelles de traitement.

2. Détermination des pentosanes hydrosolubles et totaux

Les pentosanes totaux sont solubilisés dans une solution d'acide dilué pour moins de 10 min; les pentosanes solubles dans une solution aqueuse, pour 15 min. Les étapes de solubilisation et extraction sont décrites dans l'article de Rouau X et Surget A, 1994. Les extraits sont analysés pour contenu en pentose selon une adaptation de la méthode de phloglucinol de Douglas, S. G. (1981, Food Chemistry 7; 139-145) en utilisant une méthode semi-automatique décrite par Rouau X et Surget

Pour les pentosanes totaux : peser 0,1 g de farine dans un tube de 10 ml en verre, et bien mélanger, avec 5 ml d'acide sulfurique 1 M. Le tube est ensuite porté à ébullition pour 10 min dans un bain-marie, refroidi et finalement centrifugé à 5000 g pour 5 min. Un aliquot du surnageant est dilué 10 fois et soumis à l'analyse de l'auto-analyseur. Pour les pentosanes solubles : 1 g de farine est déposé dans un tube centrifuge de 10 ml. Quatre ml d'eau distillé sont ajoutés dans le tube et le contenu homogénéisé par une agitation au vortex. Le tube est par la suite agité à 40 révolutions/min^{-1} et agité pour 5 min à 5000 g. Le surnageant est dilué 50 fois et soumis à l'analyse. Nos échantillons ont été soumis au laboratoire del'INRA à Montpellier en France, à l'Unité de Technologie des Céréales et Agropolymères.

3. Protéines solubles et protéines insolubles (gluténines insolubles)

La quantification de la teneur en protéine de la farine qui ne peut être extraite par une solution de 50 % de 1–propanol, mais extraite dans ce solvant en présence d'un agent réducteur, permet de mesurer les macro-polymères de la gluténine (ie. les gluténines insolubles, aussi référées comme agrégées) (Sapirstein et Johnson, 1998, 2000).

Matériel – Préparation du tampon (Propanol–Tris)

0,08 M TRIS (trizma base ou hydroxymethyl aminomethane C_4H_{11} NO_3MW 121.1) c'est-à-dire 0,97 gm TRIS dans 100 ml d'eau déionisé ajusté à pH 7,5 avec HCl 1 M.
Ajouter 100 ml 1–propanol avec 100 ml de solution TRIS (0,04 M final).
Cette solution est stable et peut être préparée à l'avance.

Approximativement 50 mg de farine sont extraits, deux fois, avec 1 ml d'une solution de 50 % 1–propanol pour 30 min avec une centrifugation entre chaque extraction à 2 200 G [22 G] pour 5 min.
Les extraits (surnageants) qui contiennent les protéines monomériques (albumines, globulines, et les gliadines et gluténines solubles) sont utilisés pour la quantification des protéines solubles dans une solution de 50 % 1–propanol (Sapirstein, H. et Johnson, 2000) à 214 nm .
Le résidu libéré des protéines monomériques référé comme « protéines 50 PI » dans la méthode est incubé avec 1 ml de 1 mg/ml DTT dans le tampon TRIS (25 mg DTT ou

dithreitrol avec 25 ml du tampon TRIS) pour 1 heure après agitation avec un vortex. Après 1 heure d'incubation à 55 °C, centrifuger à 10 000 G [100 G] pour 10 min et récupérer le surnageant pour analyse (rejeter le résidu).

Une courbe de calibration peut être préparée à partir de la farine de blé ou semolina ou encore à partir de la protéine 50 PS, et la teneur en protéine peut être déterminée à l'aide de la méthode Leco ou Kjehdahl.
Note : il est nécessaire de préparer un contrôle contenant la solution de 1 mg/ml DTT dans le tampon 1–propanol TRIS et l'incuber à 55° C comme décrit pour les échantillons.

Le Beckman UV est ajusté à 214 nm et ajusté à zéro avec le contrôle.
Le contrôle contient 3 ml de 1–propanol (50 %) + 50 µl de tampon DTT (1 mg/ml) dans le tampon TRIS qui a été incubé à 55° C (les deux cellules pour la lecture à zéro).

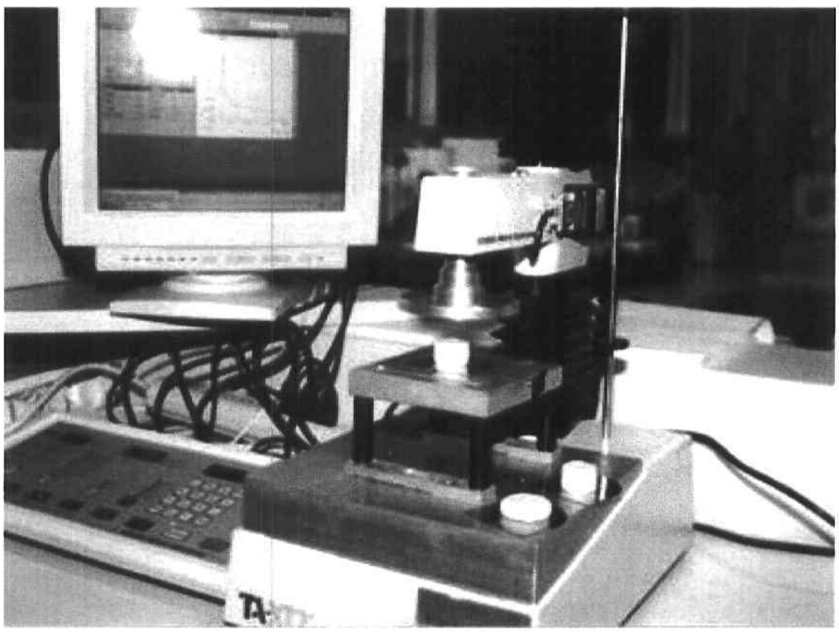
Ajouter, de façon séquentielle, dans la cuvette à échantillon 10, 20, 0 et 60 µl de la solution de gluten standard (absorbance approximative 0,098, 0,176, 0,340 et 0,454)
Pour la lecture des échantillons, on ajoute 3 ml de 1–propanol 50 % + 50 µl de l'échantillon.

4. Relaxation après compression d'un disque de pâte en conditions lubrifiées (déformation biaxiale)

Un disque mince de pâte biscuitière est comprimé entre deux plateaux. Les interfaces entre les échantillons et les plateaux sont lubrifiées avec de l'huile végétale de haute viscosité (vaseline) afin de minimiser les frictions, d'éviter de cisailler la pâte biscuitière et aussi les problèmes de déshydratation (la pâte s'assèche et durcit rapidement).

Nos essais ont été réalisés sur les pâtes biscuitières d'hydratation constante (24 % d'eau) entreposées 45 min à 27 °C dans des sacs en plastique déposés dans un bain-marie. Des disques de pâte ont été découpés à l'aide d'un emporte-pièce avec un coupe-fil contrôlant

la hauteur de 20 mm et le diamètre de 25 mm. Les surfaces du fond de la sonde, du support et de l'échantillon sont enduites d'huile de vaseline (η = 30 mPa.s).
Un appareil de texture (TA.XT2, force maximale 25 kg, Texture Technology Stable MicroSystems, Scardsale, NY) a été utilisé. La sonde est mise en contact de la pâte de manière à ce que la force contact soit inférieure à 0,1 N. La traverse descend alors à une vitesse de 0,8 mm/s, jusqu'à 40 % de compression du disque (10 s). La relaxation est suivie pendant 150 s. La fréquence d'acquisition est de 25 points/s. Six disques de pâte ont été analysés pour ce test.



Le rhéogramme de la Figure 1 comporte deux phases distinctes :
– une phase de compression, phase obtenue lorsque la cellule déforme le pâton à vitesse constante,
– une phase de relaxation (relaxation de la contrainte car la déformation est maintenue constante), qui débute lorsque l'outil de compression est immobile.

La force maximale (F_{max}) à 40 % de déformation constitue un paramètre caractéristique de la pâte, qui a été retenu dans l'étude.

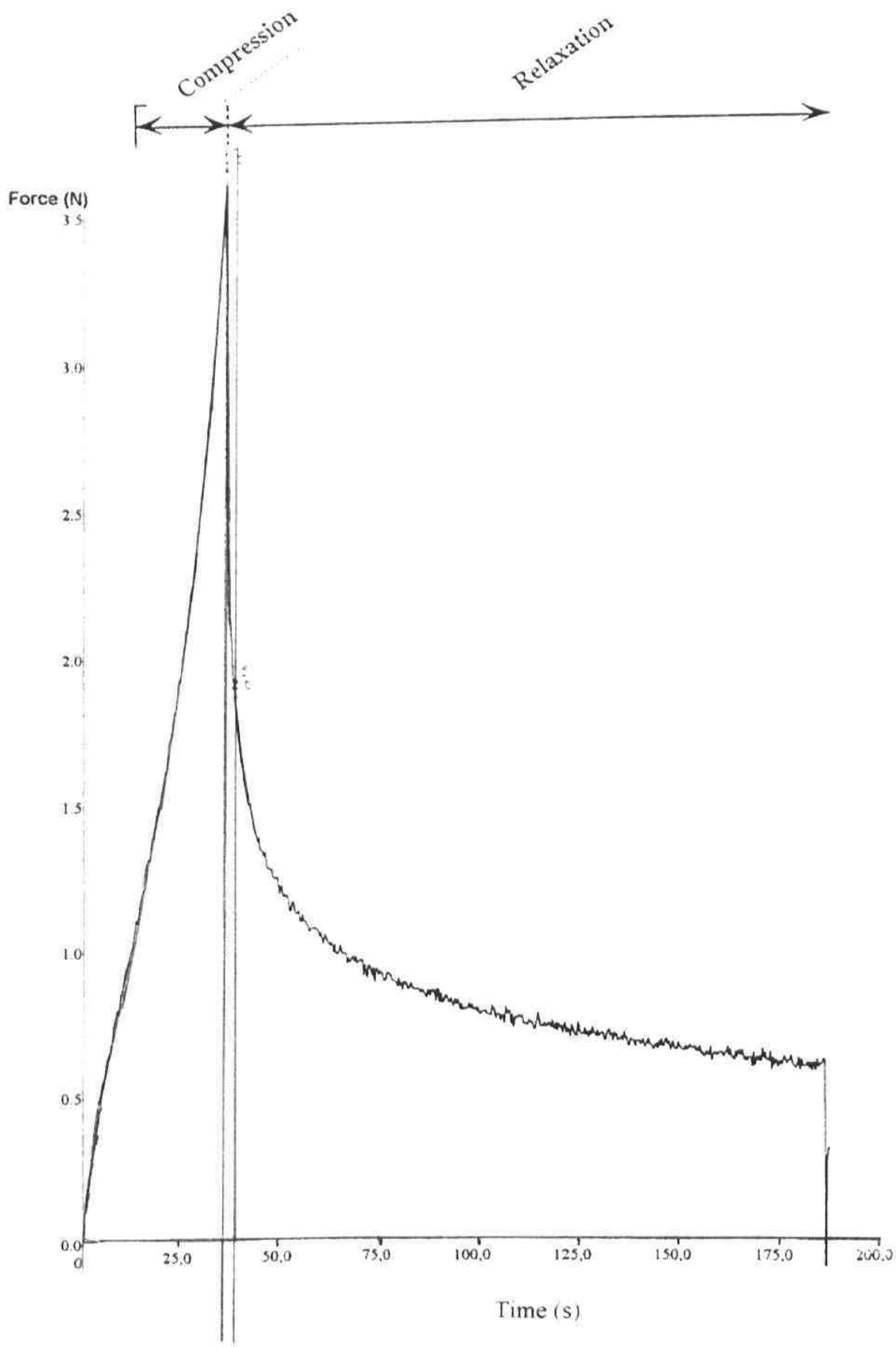


Figure 1 : Compression et relaxation des pâtons

Le modèle analogique non linéaire proposé par Launay (1990) a été retenu pour caractériser physiquement les pâtes biscuitières. Sa loi de comportement fait appel à la contrainte mesurée lors de la sollicitation du matériau, c'est-à-dire au rapport de la force mesurée sur la surface de contact « échantillon–cellule de compression ». Cette grandeur n'étant pas une donnée expérimentale directement accessible, il est nécessaire de traduire la force en contrainte.

Donc pour transformer les rhégrammes Force-Temps en rhéogrammes contrainte versus temps, nous considérons alors les surfaces de contact comme étant idéales. Ainsi le pâton est supposé garder son volume et forme cylindrique lors de la déformation.

La surface idéale pour une déformation isovolume et parfaitement cylindrique s'exprime alors, a un temps t quelconque par la relation :

$$S(t) = \frac{h(0)}{h(t)} * .So$$

où h(o) :hauteur initiale; h(t) : hauteur au temps t; S_0 : surface initiale et S(t) : surface de contact

Le modèle rhéologique utilisé correspond à un liquide de Maxwell dont la viscosité suit une loi en puissance avec la vitesse de déformation, introduisant une non-linéarité de comportement. L'équation générale peut s'écrire :

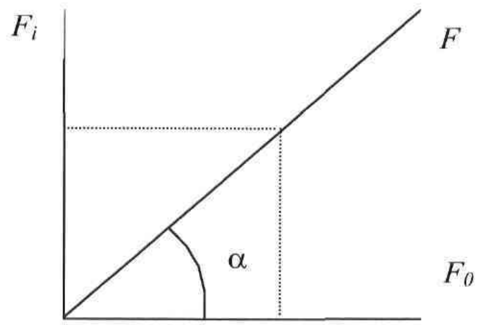
$$\sigma(t) = \sigma_i \left[1 + k\left(\frac{1}{n} - 1\right)t \right]^{\frac{n}{n-1}} \qquad \text{Équation 1}$$

où $\sigma(t)$: contrainte à chaque temps et

$\sigma(i)$: contrainte au temps initial.

Pour les deux types de relaxation, l'ajustement porte sur la contrainte normalisée :

$$\frac{\sigma(t)}{\sigma_i} = \frac{F(t)}{F_i} \qquad \text{où } \sigma = \frac{Force}{A(aire)}$$



où $F_0 = F_i \cos \alpha$

$$\frac{F_0/A}{F_i} = \cos \alpha$$

De l'équation 1, on peut déduire :

$$\left[1+k\left(\frac{1}{n}-1\right)t\right]^{\frac{n}{n-1}} = \frac{\sigma(t)}{\sigma_i}$$

où $\frac{\sigma(t)}{\sigma_i} = \frac{F(t)}{F_i}$

Les paramètres issus de l'ajustement sont :

k : la constante de vitesse de relaxation (s^{-1}) et

n : l'indice de comportement en écoulement/sans dimension variant de 0 (solide élastique, pas de relaxation) à 1 (liquide moins viscoélastique, relaxation totale).

De plus, on obtient deux autres paramètres :

F_i : force initiale de relaxation (ou maximale de compression Fmax), c'est-à-dire la force correspondant à un taux de compression de 40 % et

t_{la} : temps de demi-relaxation pour lequel la force est $F_i/2$.

Dans ce travail, nous avons utilisé l'équation 1 à l'aide du logiciel. Table curve 2D, distribué par Jandel Scientific.

5. Viscosité élongationnelle

Les disques des pâtons lubrifiés ayant subi une compression de 40 % de la hauteur du disque de pâte de 20 mm, avec une vitesse de traverse de 0,8 mm/s ont été quantifiés pour leur hauteur et rayon après compression afin d'obtenir la viscosité élongationnelle (Pa.S) qui est obtenue selon la formule (Miller, R. A. and Hoseney, R. C., 1997).

$2\ Fh/R^2V_Z$

où F : la force F_{max} pour assurer la déformation du disque de la pâte (g),
h : hauteur du disque après compression en mm,
R : rayon du disque après compression en mm et
V_Z : vitesse de traverse en mm/s.

Note : $\frac{1N}{mm^2} = 10^6$ Pascal (1 MPa) et 100 g = 0,981 N

6. Dureté, consistance, élasticité et intensité de cohésion des pâtes biscuitières

Les petits disques de 20 mm de hauteur et 25 mm de diamètre ont été utilisés. Dans ce cas, les pâtes ne sont pas lubrifiées à la surface comme dans le test précédent.

L'appareil et le système mécanique discutés antérieurement ont été utilisés sur des pâtes biscuitières d'hydratation constante (24 % d'humidité) entreposés 30 min à 27° C dans un bain-marie et découpés avec l'emporte-pièce avant l'analyse. Les paramètres utilisés pour la procédure du TPA étaient descente de traverse, 0,8 mm/s, jusqu'à 40 % de compression du disque de pâte avec un temps de récupération entre les deux compressions de 5 s. La fréquence d'acquisition est de 100 points/s. L'analyse des rhéogrammes permet de déterminer :

-La consistance de la pâte (N.s) : correspond aux surfaces des pics A1 et A2 (Figure 2)

-L'intensité de cohésion TPA (sans unité) : rapport des surfaces de compression (A_2/A_1). Représente la quantité d'énergie nécessaire pour effectuer le travail de compression. Une cohésion de 1 indique un aliment élastique idéal. Cet attribut réfère donc à l'intensité des liaisons internes à l'origine de la structure du produit.

-La fermeté ou dureté TPA (g) : force correspondant à la hauteur du premier pic. C'est la force nécessaire pour atteindre une déformation donnée. C'est donc la force nécessaire F pour compresser l'échantillon la première fois. Certains auteurs préfèrent exprimer la fermeté comme force par une unité de surface de l'échantillon (F/S en N/mm^2); ou la force par unité de déformation (F/d en N/mm).

-L'élasticité TPA (sans unité) : correspond au rapport des longueurs L2/L1. C'est la déformation récupérée (après compression) ou le pourcentage de recouvrement d'un matériel ayant subi une déformation lorsqu'il n'est plus soumis à cette contrainte.

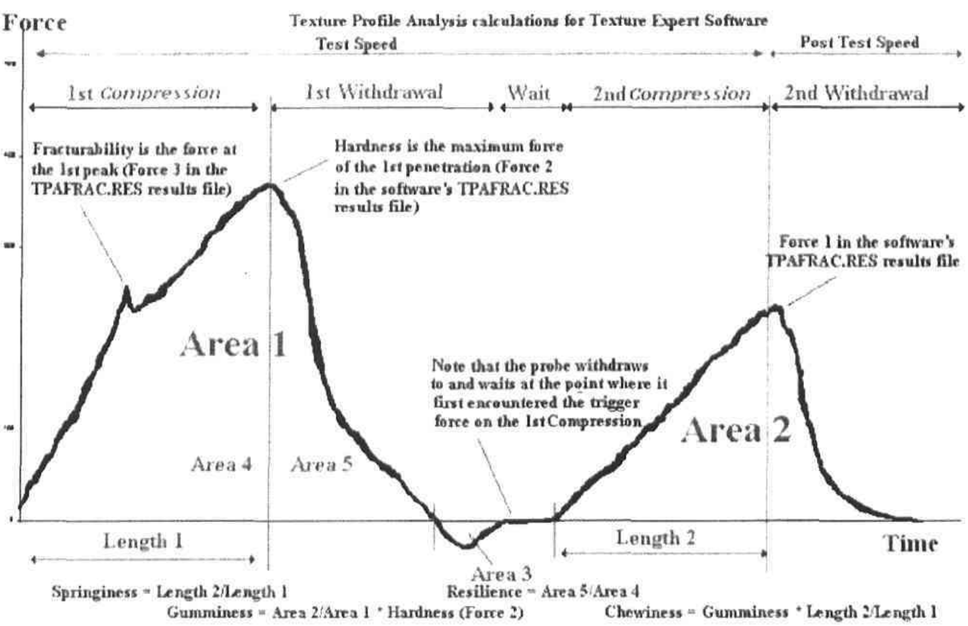


Figure 2 : Test de la double compression TPA

7. Test biscuitier du CTUC

- La formule est présentée au tableau suivant :

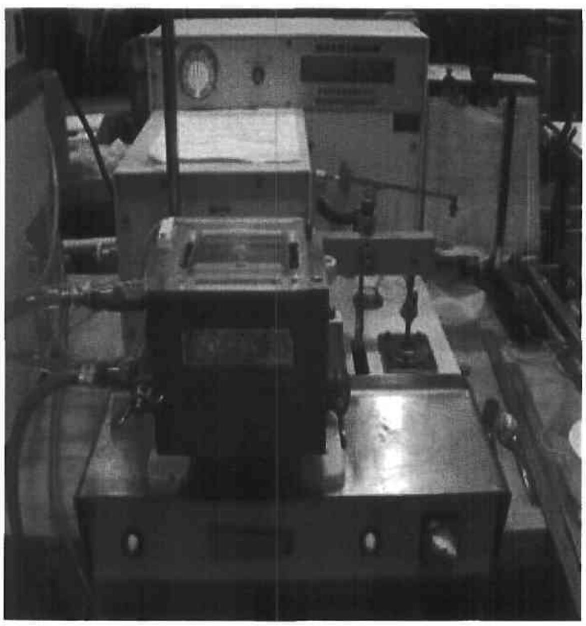
Farine	100,00 *
Sucre fin	30,00
Shortening	8,00
Bicarbonate d'ammonium	1,00
Sel	0,63
Bicarbonate de sodium	0,50
Pyrophosphate de sodium	0,50
Eau	26,00 *

- à déterminer pour 350 g de pâte de teneur en eau totale de 24 %. Pour déterminer la quantité de farine et d'eau à utiliser pour cette pâte, nous avons utilisé un système de deux equations à deux inconnus. Le logiciel Excell a été utilisé pour résoudre l'équation. Pour les recettes reconstituées, les fractions possèdant des humiditées différentes, les calculs utiliseront la masse sèche de la farine à utiliser comme facteur fixe.

Cuisson des pâtes

Tous les ingrédients de la recette doivent être préalablement tempérés à 18-20 °C. Le bicarbonate de sodium ainsi que le pyrophosphate de sodium sont pesés ensemble, puis tamisés avec la moitié de la farine. Le bicarbonate d'ammonium est, quant à lui, solubilisé dans l'eau de coulage.

Le pétrissage est effectué dans le pétrin du farinographe de 300 g de type résistographe Brabender (Mackensack, NJ 07600) couplé d'un bain thermostaté (Fisher Scientific Isotemp 3016) pour maintenir la température à 27 °C. La vitesse du fraseur est de 47, 5 tours/min. La durée du pétrissage est de 13 min. Tous les ingrédients pulvérulents sont introduits dans le pétrin. Ce dernier est ensuite mis en marche pendant 30 s. Puis il est arrêté pour déposer le shortening qui est mélangé manuellement avec la poudre afin de briser le shortening en petits morceaux. Le pétrin est ensuite remis en marche. L'eau de coulage est versée pendant 10 s environ au travers de l'orifice circulaire situé au centre du couvercle de la cuve du pétrin. La durée du pétrissage (13 min) est décomptée après avoir versé l'eau de coulage.



La température du bloc de pâte est prélevée à la fin du pétrissage. La totalité de la pâte est disposée dans des sachets en plastique hermétiques en flottaison dans un bain-marie thermostaté à 27 °C pendant 30 min pour un temps de repos.

Le laminage est effectué sur le laminoir Sinmag de Picard Equipment (Bakery Equipment Inc., Victoriaville, Qc) constitué de deux cylindres de laminage de 9 cm de diamètre tournant à une vitesse de 140 tours/min. La vitesse du tapis est de 24 m/min. Les écartements des cylindres pour le passage de la pâte sont respectivement de 15 mm (une fois), 7 mm (une fois), 4,5 mm (une fois), 2,5 mm (deux fois aller-retour et pliage de la pâte entre chaque passage) et finalement, 1,75 mm (deux fois aller-retour).

Les pâtons sont découpés à l'aide d'un emporte-pièce carré de 6 cm sur 6 cm munie de 13 aiguilles. Cette opération se fait à l'arrêt sur le tapis. Les pâtons sont par la suite déposés sur un plateau de cuisson préalablement taré.

La cuisson est réalisée à une température de 280 °C ± 5 °C. La cuisson est arrêtée après une perte à la cuisson de 20 % (perte pondérale). Les biscuits sont ensuite transférés sur une grille pour leur ressuage à température ambiante durant 30 min.
Les biscuits sont entreposés dans un emballage hermétique (film en aluminium) et conservés deux semaines à température ambiante avant d'étudier leurs caractéristiques dimensionnelles et mécaniques

8. Propriétés mécaniques des biscuits

Lors du test de pénétrométrie conique, la tête mobile du vérin est munie d'un piston conique de demi-angle au sommet connu. L'échantillon est posé sur un support présentant un petit orifice cylindrique au centre, afin de ne pas abîmer la pointe du cône lorsque le produit est transpercé. Par l'intermédiaire du clavier et d'une carte d'acquisition, l'évolution de la force subie par l'échantillon est enregistrée en fonction du temps par le micro-ordinateur.

Lorsqu'une contrainte mécanique est appliquée dans une direction, une déformation instantanée et irréversible peut être reliée à la rupture partielle ou totale de l'échantillon. La pénétrométrie conique couple une compression renouvelée et un cisaillement, et doit permettre une bonne caractérisation du comportement mécanique du produit.

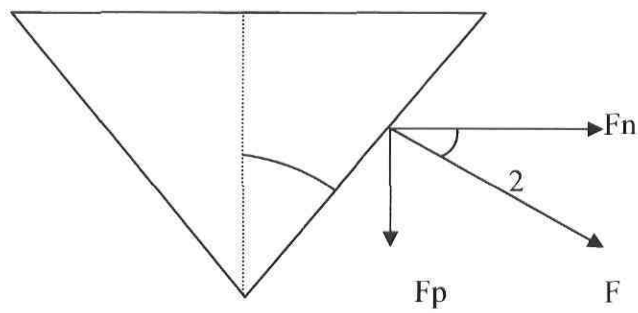


Schéma des forces appliquées au cône

En absence de cisaillement, la force de compression (F), normale à la surface du cône, est divisée en deux composantes : la force (F_p) parallèle à l'axe du cône et la force (F_n) normale à l'axe. La force mesurée, intégrant l'ensemble des forces parallèles (F_p), est liée à la force F par la relation :

$F_p(y, 2) = F(y, 2).\sin 2$

Dans l'ensemble des produits considérés ici (matériaux solides fragiles) sera seule prise en compte la force effective, F, appliquée de manière renouvelée au biscuit, détruisant sa structure par rupture des liens entre une particule ou un groupe de particules sollicitées et l'ensemble du produit.

Le signal enregistré correspond à la détection de l'évolution de la force en fonction du déplacement. Trois critères ont été retenus sur la courbe force-déplacement (Figure 3) :

–le travail (S) au cours d'un certain déplacement y, qui correspond à l'aire sous la courbe entre 0 et y (N.m);
–le nombre de pics (N) au cours du déplacement y, qui est représentatif du nombre de grains ou de groupe de grains arrachés;

-la chute de force (ΔF) pour chaque pic, représentant la résistance propre à l'arrachement des grains ou groupe de grains, c'est-à-dire la force spécifique à l'arrachement (N).

À partir de ces critères ont été déduits trois paramètres significatifs, permettant de décrire le profil mécanique des biscuits :

–la force moyenne globale de pénétration, notée Sm :

$$Sm = \frac{S}{y} \qquad \text{(N)}$$

S : aire sous la courbe entre o et y = travail (Newton.m)
y : distance de pénétration du cône (mm)

–la fréquence spatiale d'arrachement, (nombre de grains ou groupes de grains par unité de distance de pénétration) notée N_0 :

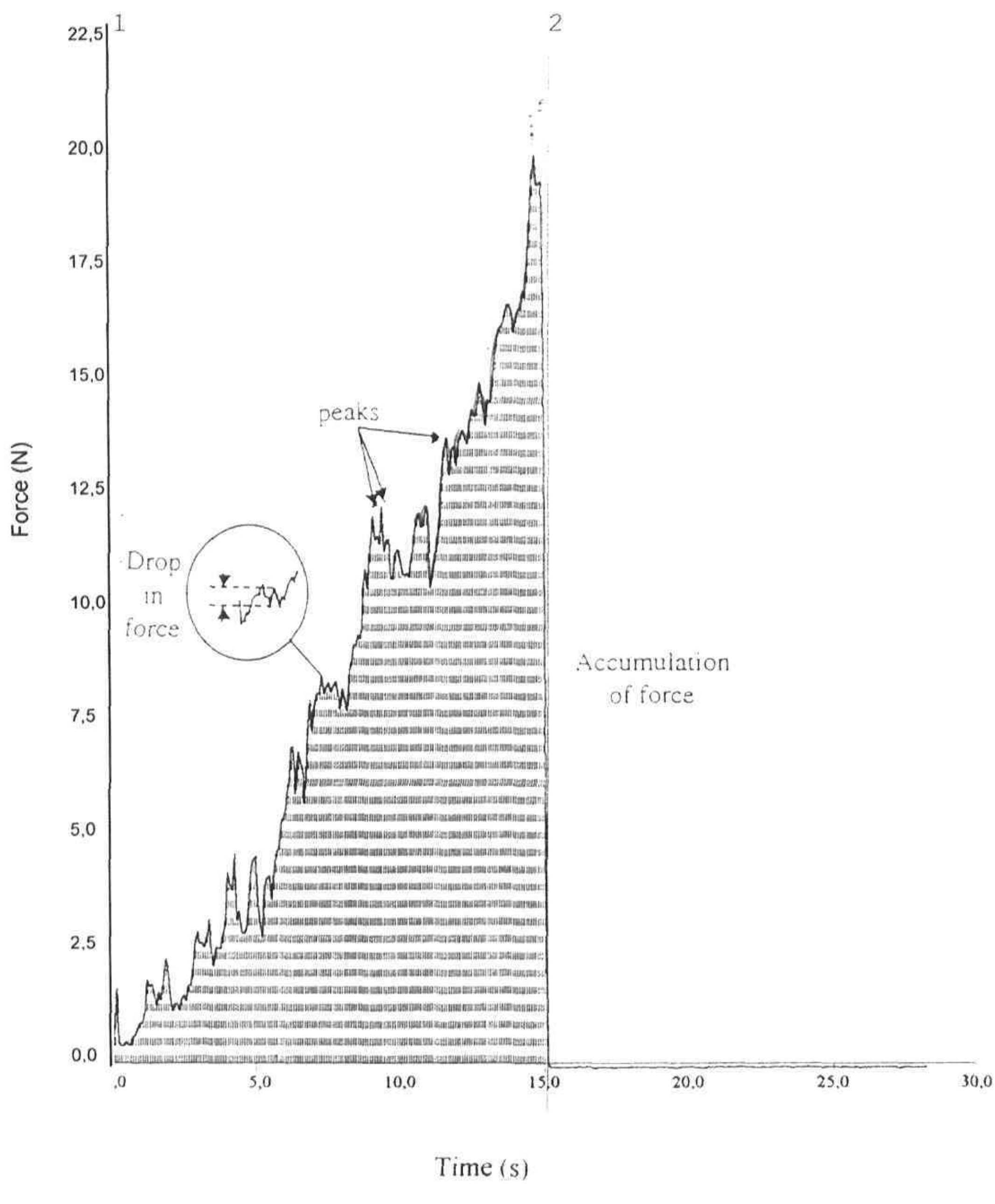


Figure 3 : Test de pénétrométrie conique

$$N_0 = \frac{N}{Y} \qquad (1/\text{mm})$$

– la force spécifique moyenne d'arrachement, notée FS :

$$FS = \frac{\sum(\Delta F)}{N} \qquad (\text{N})$$

Un quatrième paramètre peut être proposé, qui combine la force spécifique et la fréquence spatiale d'arrachement des grains :

$$FS/N_0 \qquad (\text{N.mm})$$

Un biscuit est d'autant plus friable qu'il présente lors de sa pénétration un grand nombre de grains arrachés (N_0 élevé) pour une faible force d'arrachement (FS faible). Donc, le rapport (FS/N_0) serait d'autant plus faible que le biscuit est friable. Ce paramètre traduit donc « l'effort de friabilité » ou référé comme le croquant des biscuits.