Technical University of Denmark

Design of Experiments for Food Engineering

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Publication date:
2015

Document Version
Publisher's PDF, also known as Version of record

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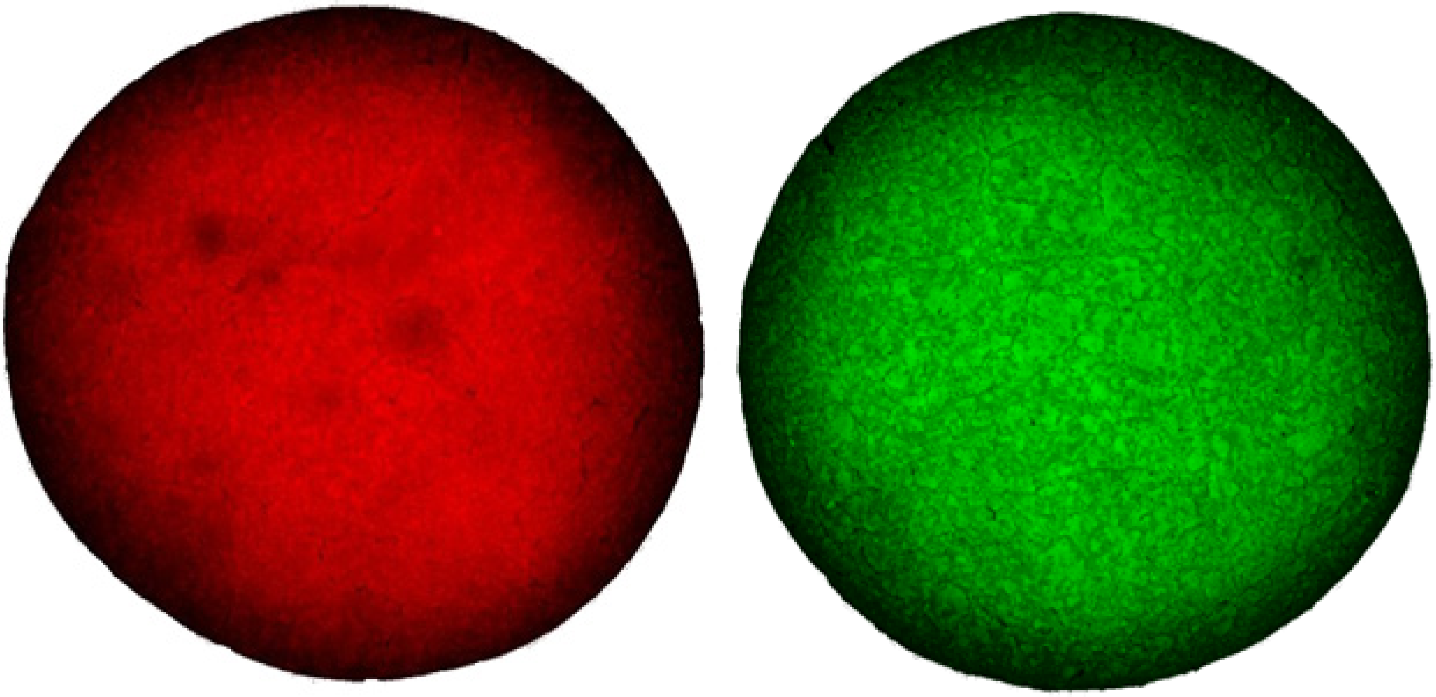

PhD Thesis
Søren Juhl Pedersen
April 2015

DTU Food
National Food Institute

Design of Experiments for Food Engineering

National Food Institute
Division of Food Technology

Design of Experiments for Food Engineering

1. edition, April 2015

Photo: Søren Juhl Pedersen
ISBN: 978-87-93109-83-4

This report is available at
www.food.dtu.dk

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Funding: This project was part of the larger research project "Integrated modelling of food production chains" under the umbrella of inSPIRe Pillar 1 funded by:

The Danish Council for Strategic Research

The Danish Council for Technology and Innovation

Preface

This PhD project was carried out at the Research Group for Food Production Engineering, National Food Institute, Technical University of Denmark (DTU). The project was a part of the research project “Integrated modelling of food production chains” under the umbrella of inSPIRe Pillar 1 funded by The Danish Council for Strategic Research and The Danish Council for Technology and Innovation which is gratefully acknowledged. The project was supervised by Stina Frosch, Murat Kulahci and G. Geoffrey Vining.

I would firstly like to declare my appreciation with all the companies and representatives which have been a part of the work. Bisca A/S, Easyfood A/S, BCH ltd., Royal Greenland, Haas-Meincke A/S and Danish Technological Institute. There are numerous people that all deserve acknowledgements and have been crucial in providing knowledge and relevance to the research in this project. I especially would like to thank Rolf Reuther-Nilsen, Allan Toft Hansen, Peer Jespersen, Lise Nersting, Jakob Søltoft-Jensen, Jacob Munck, Ken Branton and Per Jensen.

I would also like to thank all the students I have worked with and supervised. You have all been truly inspiring and have provided me with valuable contributions and knowledge. I especially would like to mention Julie Ohrt Jensen, Sissel Therese Brøkner Kavli, Camilla Pauli and Birgitte Lærke Bentsen.

My supervisors Stina, Murat and Geoff. Thank you all their invaluable work, wisdom and help in my struggles trying to tame the beast that is the scientific method. Stina, for your trust and allowing me to stray off track and then the struggles with getting me back on. Murat for your guidance and clear insight. Geoff, for sharing your hospitality, teachings, time and immense experience.

And to my dear colleagues at DTU-Food a special thanks to you all for your nice discussions and good spirits. Aberham Hailu Faissa and Martin Gram Christen, thank you both for always providing nice discussions, scientific insights, engineering aspirations and good friendships.

To my family and friends, thanks for all the support all these years. Finally my partner in life Maria for your invaluable support without which this would not have been possible.

Summary

This work looks at the application of Design of Experiments (DoE) to Food Engineering (FE) problems in relation to quality. The field of Quality Engineering (QE) is a natural partnering field for FE due to the extensive developments that QE has had in using DoE for quality improvement especially in manufacturing industries. In the thesis the concepts concerning food quality is addressed and in addition how QE proposes to define quality. There is seen a merger in how QE's definition of quality has been translated for food. At the same time within FE a divergence has been proposed in the literature emphasizing mechanistic modelling over empirical methods. This thesis proposes that this divergence is based on a misunderstanding of what empirical methods entail. The misunderstanding could stem from the issue that the majority of the literature trying to address these issues has focused on analysis procedure almost to the point of excluding the experimental principles and procedures. This thesis has tried to address this as far as possible. The focus has been on supplying practical interpretations of randomization and what blocking entails when designing experiments. These practical interpretations have then been applied in series of actual experiments which are reported and discussed again with the aim of giving practical DoE insight. The diversity of the cases is hopefully broad enough to supply inspiration for others working on their specific issues. The relevance of the fundamental principles is shown by using these directly in the following novel research studies: The promising application of ohmic heating of shrimp. The results showed a promising technology with major potential for addressing quality issues in over cooking and possibly a better control of weight loss. In a second study the application of ASCA for analyzing a designed experiment using image data as the response with the intention of investigation process design choices is presented. Great potential was seen for using image data to elucidate how and where process choices would affect appearances of product. The aim was to show that the fundamental principles of DoE have as much importance and relevance as ever for both the food industry and FE research.

Resumé

Denne afhandling har set på anvendelsen af statistisk forsøgsplanlægning indenfor fødevareingeniørkundskab med udgangspunkt i problemstillinger omhandlende fødevarekvalitet. Baseret på den seneste udvikling og brug af statistisk forsøgsplanlægning til forbedring af kvalitet og produktion i fødevareindustrien, er kvalitetsingeniørkundskab et oplagt område at udforske for fødevareingeniører. I denne afhandling diskuteres begrebet fødevarekvalitet og hvordan kvalitetsingeniører har valgt at definere kvalitetsbegrebet. Med udspring i denne diskussion fremlægges en nyfortolkning af fødevarekvalitet inspireret af netop kvalitetsingeniørkundskab. Dog demonstrerer den seneste litteratur inden for fødevareingeniørvidenskab et voksende fokus på mekanistiske modeller frem for den empiriske tilgang, som blandt andet omfatter statistisk forsøgsplanlægning og analyse. Denne splittelse er muligvis baseret på en misforståelse af hvad empiriske metoder omfatter, og kunne skyldes at hoveddelen af al videnskabelig litteratur inden for emnet fokuserer mest på de statistiske analyser til frem for forsøgsplanlægningens fundamentale principper og procedurer. Denne afhandling prøver at adressere denne manglende demonstration af forsøgsplanlægning med basis i praktiske eksempler fra fødevareindustriel forskning. Omdrejningspunktet har været at bidrage med praktiske fortolkninger af randomisering og blokning, og hvilken betydning disse elementer har, når forsøg planlægges. Dette suppleres med en række beskrivelser af udførte forsøg hvor igen den praktiske fortolkning af forsøgsplanlægning er i fokus. Den samlede række af eksempler er tiltænkt som inspiration til andre fødevareingeniører til at takle deres specifikke problemstillinger. Derudover demonstreres relevansen af statistisk planlægning direkte ved udarbejdelsen af følgende nye forskningsresultater: Forsøg indenfor ohmisk opvarmning af rejer som viste sig at være en lovende proces. Forsøgene viste at den nye opvarmningsmetode forløste nogle af de kvalitetsproblemer, der kan forekomme ved de konventionelle produktionsmetoder. Et andet studie omhandler anvendelsen af ASCA til at analysere forsøg, hvor billeder af produktet bliver brugt som ”målepunkter”. Dette er gjort med henblik på at kunne forstå, hvordan procesdesign og indstillinger påvirker produktets udseende. Brugen af billeddata til at belyse, hvordan og hvor processen kan påvirke produktets udseende viste sig at have stort potentiale. Afhandlingens formål var at vise, at den statistiske forsøgsplanlægnings grundlæggende principper har lige så stor betydning og relevans som nogensinde før for både fødevareindustrien og forskning indenfor fødevareingeniørkundskab.

List of publications

Above each paper or report is given the name for the reference used in the thesis. Copies of Papers C and D are attached in the appendix.

(Paper A)

Søren Juhl Pedersen, Aberham Hailu Feyissa, Sissel Therese Brøkner Kavli and Stina Frosch. (2015). An investigation on the application of ohmic heating of cold water shrimp and brine mixtures. *Submitted to Journal of Food Engineering*

(Paper B)

Søren Juhl Pedersen, Julie Ohrt Jensen, Murat Kulahci, Geoffrey Vining, and Stina Frosch. (2015). Analysis of designed experiment with cookie images as multivariate response using ASCA for assessing process effects on surface appearance. *Submitted to Journal of Food Engineering*

(Report I)

Søren Juhl Pedersen, Murat Kulahci, and Stina Frosch. (2014). Case presentation: Screening experimentation on a pilot scale oven for factor identification. *Abstract for the 14th European Network for Business and Industrial Statistics Conference (ENBIS-14), Johannes Kepler University, Linz, Austria*

(Report II)

Søren Juhl Pedersen, Stephanie Henriksen, and Stina Frosch. (2014). Study on the influence of resting time and dough reuse on sheeted dough rheology by response surface methodology. *Internal Report*

(Paper C)

Geoff Vining, Murat Kulahci and Søren Pedersen. (2015). Recent Advances and Future Directions for Quality Engineering. *Quality and Reliability Engineering International*

Other publications

(Paper D)

Aberham Hailu Feyissa, Martin Gram Christensen, Søren Juhl Pedersen, Minka Hickman, Jens Adler-Nissen. (2015). Studying fluid-to-particle heat transfer coefficients in vessel cooking processes using potatoes as measuring devices. *Journal of Food Engineering,* 163, pp. 71-78

Abbreviations

FE	Food Engineering
QE	Quality Engineering
DoE	Design of Experiments
RSM	Response Surface Methodology
ANOVA	Analysis of Variance
ASCA	ANOVA Simultaneous Component Analysis

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Introduction

The motivation for the work in this thesis is the goal of improving food quality. The discipline Food Engineering (FE) is the science concerning industrial food production and the quality produced. This constitutes many different types of activities. It can be in the development of technologies for processing. It can be lab experiment to increase understanding of process and product interaction or the implementation of measurement tools for monitoring the production. The different activities require a combination of different tools and techniques. These are again a combination of theory (mechanistic) and experiments (empirical) that is encompassed by the course of learning. This thesis is influenced by the narrative that in tackling engineering problems, the experimental approach is crucial. Quality Engineering (QE) address approaches for learning and improving processes based on the concept that variation defines quality. Design of Experiment (DoE) is a statistical framework and theory focused on how to design experiments while considering experimental error, resources and the objective of the experiment. QE has adopted and evolved DoE for formalizing learning and empirical modelling of systems for improving quality. Thus, experimentation is the crucial aspect. The intentional tinkering of the systems which experiments are, gives empirical knowledge concerning systems in general and at hand. This is also one of the first steps in attaining mechanistic description or the final steps in verifying a model description. Experimentation is therefore crucial to link empiricism and mechanistic understanding. A rough description of the procedure in engineering for finding solutions to problems is shown in Fig. I.1 which will help in describing the relationship between empiricism and mechanistic understanding (Coleman & Steele 1999)

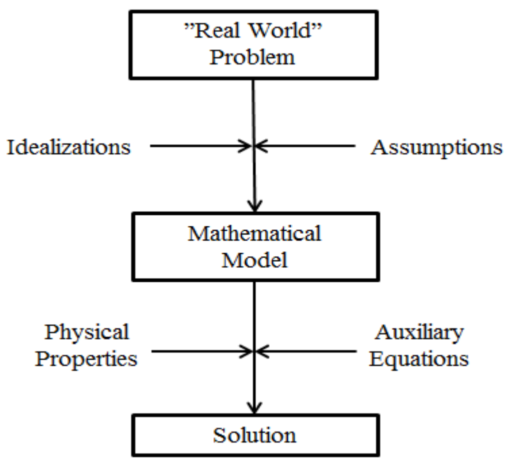

Figure I.1 The analytical approach for understand and solving problems (Coleman & Steele 1999)

The description in Fig. I.1 is what is meant by empiricism and mechanistic understanding confided to the work of engineering. By engineering is meant the application of the scientific method for providing solutions to design problems. Depending on the level of knowledge concerning the problem, different methodologies can be applied, but typically they follow the same arch as Fig. I.1. The common approach is to use idealization and assumptions to formulate mathematical models or modular descriptions of the system under study. The models can directly or indirectly be used to propose solutions. The use of experiments is an inevitable part of the process of finding or verifying solutions. This means the entire process which Fig. I.1 depicts could also represent an experiment. The original presentation of the diagram (Fig. I.1) was to represent the formulation of a physical phenomenon (Coleman & Steele 1999). This process would typically require experimentation in one or more of the steps. Typically measurements of some physical properties are needed or experimentation to verify certain idealizations before formulating a mathematical model. The belief is that the diagram in Fig. I.1 is also a depiction of the learning process when developing solutions of which experimentation is an important part.

Much research activity is focused on the development of the analytical approach focusing on numerical simulations and theoretical descriptions of the “real world” in food manufacturing (Bruin & Jongen 2003; Saguy 2015). This thesis addresses the use of DoE with the connections, applications and possibilities when applied in FE. Like the motivation driving the development of analytical tools (Bruin & Jongen 2003), the development of statistical techniques are important when addressing problems of food quality (van Boekel 1996). In the application of either empirical or mechanistic models to real life problems variation has to be recognized and accounted for especially as the problem becomes more complex without an infinite source of time, money and resources. Therefore, investigating this variation through the use of DoE and statistical modelling is important.

Research Premise and Objectives

The Premise: The work compromised in this thesis is part of a larger research project "Integrated modelling of food production chains" under the umbrella of inSPIRe Pillar 1. The aim of the research project was the development of robust engineering equations for use in design, planning and control of food productions. The placement of this thesis in the context of the research project is the recognition of the importance of sound designed and planned experimental designs in laboratory, pilot or industrial scale. QE researches and develops DoE for application areas within industrial settings and where the overall goal is to improve quality understood in the broadest possible sense. Therefore, these fields e.g. FE and QE converge in the aim of improving quality of food. The objectives of the study are as follows:

Objectives

How has DoE been applied in FE? Are there any specific requirements?

What can QE give to the understanding of food quality?

How can DoE and mechanistic understanding be combined for improving food quality?

Scope

The research is within the field of applying DoE to FE problems. The approach is the description and discussion of DoE through presentation of cases. The cases represent a small segment of possible quality improvement activities. But it is hoped that the diversity and novelty of the cases can be relatable to other food engineers. The motivation for the work is the combination of the complexity in tackling food quality problems and the recognition of applying statistical tools to structure experiments on such problems. Therefore the focus has been on the fundamental principles of DoE which in fact seldom is used. The examples used in thesis are all based on original FE research on both new technological applications and fundamental issues in production understanding. The research contributions that the experimental designs have been used for in this study:

Ohmic heating of shrimp: A promising new application the technology that could alleviate some current quality issues

Evaluation of pilot scale impingement oven: Experimental platform important for further research into upscaling modelling of industrial baking operations (Andresen et al. 2013)

Study on the implications of reintroducing scrap dough: A very common process step which has never been researched before specifically the impact scrap dough has on overall material properties

Using images for evaluating process design choices: An application of a multivariate analysis technique for process design

It is the belief that the presentations of this novel food engineering research can provide appreciation of the always relevant principles of replication, randomization and blocking even at the frontiers of food engineering.

Structure of the thesis

The thesis constitutes of 3 parts. The first part frames the research of the thesis within the union of food and quality engineering. The concepts of quality and food quality are discussed. The second part is presentation of some basic concepts in DoE with a practical interpretation relating it to the context of the thesis. The third part presents examples of FE research problems where the DoE concepts have been applied. For each of the cases the design process is introduced and discussed with the intention of showing the many options applicable. The cases fall under the categories of; new application of novel process unit for seafood (Paper A), testing pilot scale platforms (Report I), new research to uninvestigated product properties (Report II) and novel investigation of product process interactions with new analysis method (Paper B). The thesis ends with an overall discussion. The discussion will try to frame the thesis and the learning outcome of the work. The examples will be discussed as a whole against the backdrop of the ideas and thoughts presented on quality of food. To conclude the thesis the perspectives on future work and opportunities will be given. In the appendix is attached Paper C and Paper D.

Part 1 Food Engineering and Quality

The aim of this chapter is to provide a conceptual overview of Food Engineering (FE), quality of food and link it to statistical thinking of food quality. The description of FE is provided to give a scope of the field of application. This is followed by an explanation of the concept “quality of food” in particular and Quality Engineering and quality in general. The intention is to provide within the description of food quality, contexts from both the producer’s and the consumer’s points of view and their conceptual relationships. Different food quality concepts discussed in this Chapter primarily focus on empirical methods. The particular emphasis is on the empirical methodology used to quantify and tackle the complexity of food quality at an operational level (van Boekel 2008a; van Boekel 1996; van Boekel 2008b). This leads into an introduction to the field of Quality Engineering (QE) and retackling the issue of defining quality. The aim is to establish a broader common ground with DoE for both the FE and QE. The idea is to link FE and QE through DoE which will allow for exchange of ideas and methodologies as illustrated in Fig. 1.1. The connection is formed by the introduction of the basic principles in DoE through a series of case studies with varying degrees of elaboration.

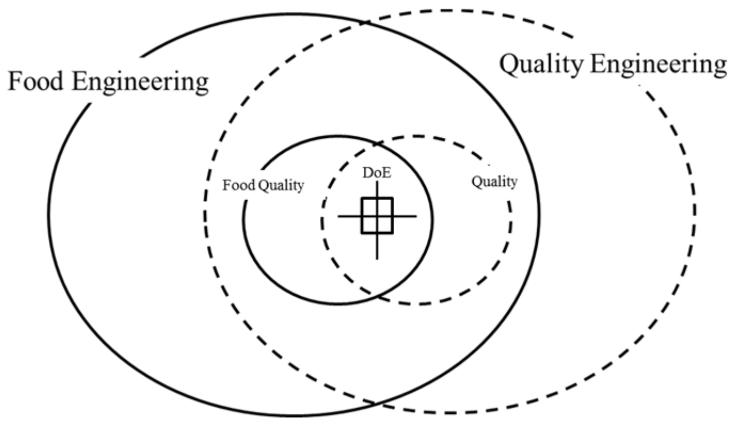

Figure 1.1 Diagram of the conceptual overlap between the fields of food engineering and quality engineering. Both fields have overlapping areas in tools and techniques used. The thesis focuses on the possible connections which DoE can bridge especially in the shared area concerning quality problems.

The technological quality (producer) and sensorial-nutritional quality (consumer) are at the heart of the majority of FE endeavors. The vastly differing food products and production situations require that the food engineer accumulate vast amounts of knowledge. The food industry often involves various production scenarios even within the same facility. They can vary from big continuous production facilities to small medium sized batch productions. It can be facilities producing the same product year round e.g. brand name cookies to small contract bakery productions that constantly discard and introduce new products. The production flow can resemble manufacturing operations like butcheries to continuous processes like fluid handling year round as in dairies. These varying situations and constraints represented by the food industry as a whole have been tackled with the development of standardization, monitoring and engineering knowhow (engineering equations (Singh & Heldman 2014)) as the food engineers main tools. FE and QE have a long shared history in these developments, especially within the areas of management, standardization and monitoring tools particularly in applications focused on food safety (Trienekens & Zuurbier 2008; Montgomery 2009b; Dora et al. 2013a; Bertolini et al. 2006). To some degree the relationship between QE and FE could be traced back to the work of William S. Gosset (student as in student's t-test) at the Guinness breweries (Lim et al. 2014). Within the recent years the field of FE has seen the starting of a split into two directions, one based on statistical thinking (van den Berg et al. 2012; Munir et al. 2015; van Boekel 2008a) (influenced by QE and chemometrics) and the other based on physical modelling (classical chemical engineering approach) (Saguy et al. 2013; Saguy 2015; Bruin & Jongen 2003). For both directions the aim is to improve quality of food. The intention here is to join the split of the two directions of FE. It is the conviction that the concepts from QE can help in this especially with the tools from DoE. QE concerns with controlling and improving quality by attempting to reduce variation in the production irrespective of the industry

The future of QE has many possible opportunities and as was found in Paper C the approach of having application driven research questions is the most promising. As it was proposed in Paper C the food industry can be a great source of many interesting problems for mutual benefit from both methodology and application perspectives. However the language barrier between statisticians and food engineers can give rise to complications (Granato et al. 2014; Box 1996). This could be the very culprit that renders food engineers reluctant in utilizing statistical tools more prevalently. This directly ties in with trying to provide insight within designed experiments for FE in order to give the engineer an expanded vocabulary i.e. bridging the gap (Fig. 1.1.). The bridge can help the communication and the many possible mutual research activities which exist in improving food and food production. It can also help both parties in the understanding of how the experimental approach is intimately linked with the physical modelling approach as mentioned previously and aid the statistician in understanding the engineers (introduction to the thesis). Arguably, the relationship between the approaches is becoming even more evident in the emerging direct coupling of DoE and physical based modelling in the DoE subfield "design for computer experiments".

1.1 Food Engineering (FE)

The definition of FE as a field is hard to pin down. It is normally perceived as a subtopic under the umbrella of food science and technology. Taking an example from a common textbook used in FE curriculum, FE is an combination of agricultural, mechanical and chemical engineering tools applied to the science of food production (Singh & Heldman 2014). This encompasses production- and unit-operations design, product development, sensor technology, and sanitation together with all the issues that are connected with these topics to various foods. There has been a historical connection between statistically founded tools and food production as mentioned earlier with regards to management, standardization and monitoring (Lim et al. 2014). Other examples are in the science of sensory analysis of food products (Pagès & Husson 2014) and multivariate statistical analysis applied to monitoring problems (van den Berg et al. 2012). However the majority of the development in FE has been focused on material characterization, physical modelling activities and sensor development (Bruin & Jongen 2003). It is only within the last decade that the use of statistically designed experiments have received growing attention (Granato & de Araújo Calado 2014) although, as has been noted elsewhere (Nunes et al. 2015), the proper attention to possible tools and analysis are still lacking. Saguy et al. (2013), addressing the development and future of FE, discuss the increased need for a broadening of the curriculum and research field. The focus is on topics such as innovation, expanding research beyond the fundamental, being forerunners in the study of connections between food, health and wellness, and increased modeling needs (Saguy 2015) whereas experimentation and statistics is only mentioned under the umbrella of response surface modelling, with a comment of its “limited” usability. Saguy (2015) presents it as two delineated approaches one for the empirical statistical models and one for the physical based models. Saguy (2015) emphasizes that the empirical method is too simplistic in its black box approach of relating product and process.

It is the author’s (of this thesis) opinion that the demarcation between physical modelling and empirical method as described by Saguy (2015) is wrong. In the scientific method and the engineering endeavor it should not be seen as an either-or choice. It is always a combination of tools at hand trying to solving the problem, as other authors also have presented (van Boekel 1996; van Boekel 2008a; Box 1996; Box 2001). With the following digression into the concept of quality in the field of FE it is hoped to document that others – the author inclusive - are still of the opinion that a further and more detailed introduction of the techniques and statistical tools from QE is still very much relevant for the future food engineer.

1.2 Definition of food quality

Providing a definition of quality for food within the context of food science and engineering is important, but also quite challenging due to its complexity (Peri 2006; Cardello 1995). The importance of such a definition should be based on the premise of what quality of food can constitute. Food has several functions and fulfills several roles. The general consensus is that the quality of food is the combination of the food itself (intrinsic, measurable, physical attributes) and the consumer experience (extrinsic, immeasurable, dynamic performance) (Cardello 1995; Peri 2006; van Boekel 2008b).

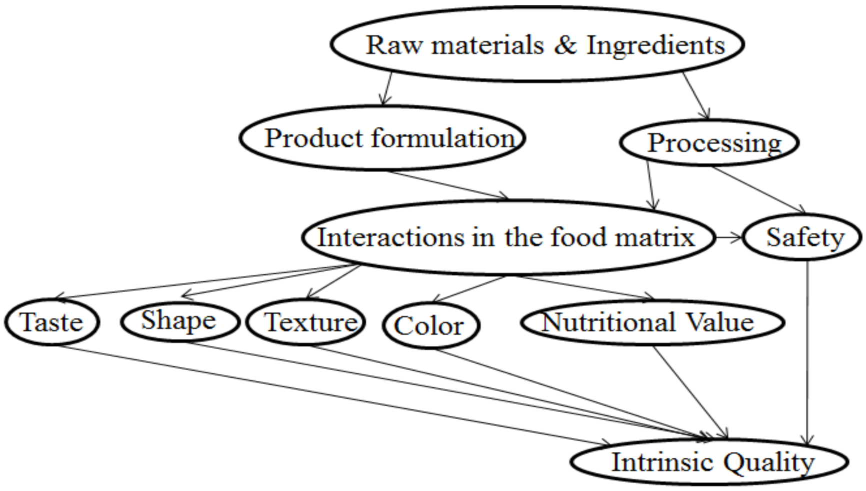

Figure 1.2.1 a rough representation of the intricate relations between the various controllable or measurable attributes and the intrinsic quality. Inspired from Fig. 1.2 (van Boekel 2008b)

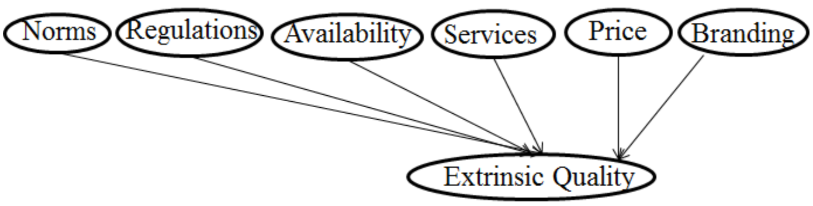

Figure 1.2.2 a rough representation of some of attributes in that the consumer perceives and the extrinsic quality. Inspired from Fig. 1.3 (van Boekel 2008b)

To connect the discussion of food quality to the definitions provided later by QE (section Definition of Quality (QE approach)) the aspect of the nominal quality has to be included as the special concept for food at least. This means that quality can be both dependent on the nominal attribute of quality and inversely proportional with the variation of the attribute. The concept of the nominal quality of the food product come into play with regards to the relationship between extrinsic and intrinsic quality attributes. The categorization of the nominal quality of the food with respect to consumer expectations can influence the producers approach to the controllable intrinsic attributes.

The idea is that for some high quality food products it is the variation in intrinsic quality attributes, determining or driving the categorization of high quality, e.g. the “homemade” appearance. This means that a certain amount of variation is as part the products quality attributes. . van Boekel (2008b) separates the concept of food quality from the food engineers view point in two categories; the intrinsic quality and the extrinsic quality. The intrinsic quality (Fig. 1.2.1) is chemical composition, nutritional value, texture, safety, shelf-life etc. The extrinsic quality (Fig. 1.3.1) is how the product relates to certain values, social norms, and price. As van Boekel (2008b) notes it is the combination of the extrinsic and intrinsic qualities that define the overall quality of the product. Due to the often unknown relation between intrinsic and extrinsic quality the use of statistical tools is highly relevant and the development of applications within FE is crucial (van Boekel 2008a; Sablani 2008; Grauwet et al. 2014). Peri (2006) discusses that food quality could be termed “fitness of consumption” and stresses the importance of the dynamic relationship between quality characteristics (intrinsic) and performance (extrinsic). He further proposes a holistic view on food quality in order to tackle the complexity of manufactured foods that satisfy consumer expectations. The proposed strategy was to define the operating space where movement within would not elicit rejection in any one of the quality attributes of the given product or products. This should be contrasted with the approach of trying to maximize preferences where the former is seen as more holistic.

The history of industrialization of food production is directly connected with the issue of supplying safe foods to the consumers. Safety is the gatekeeping quality attribute of food products and it has been a major focus area of FE and science since the early beginning. With the globalization across markets and sectors more food companies are using quality and safety management systems (Kafetzopoulos & Gotzamani 2014; Trienekens & Zuurbier 2008). The main systems are the hazards analysis and critical control points (HACCP) system, and the international organization of standard systems for quality management, ISO 9000 and 22000 series. Effectiveness in the implementation of these tools have been found to hinge upon few key parameters, mentioning especially “continuous improvement” and focus on “customer satisfaction” (Kafetzopoulos & Gotzamani 2014). It should be noted that the ISO standards for quality management are focused on the organizational structure and managerial tasks and not necessarily on the specific tools to be used for quality improvement (Trienekens & Zuurbier 2008). The issue of improving production efficiency and optimizing product quality is a multifactor and cross organizational issue. Several tools and systems have been developed with the aim of providing a structured means of addressing improvement activities in production settings, e.g. lean, six-sigma, TQM, QbD etc (Dora et al. 2013a). Although, it has been noted in several papers assessing the use of such quality management systems in food industry and other industries, the use of the statistical tools and DoE especially have in general been lacking (Tanco et al. 2010; Bergquist & Albing 2006; Sheil & Hale 2012; Timans et al. 2011). Some findings show that the implementations of quality management systems reported are mostly focused on safety and organization tools (Dora et al. 2013b). The field of QE has been a continual source for the development of the tools for these management systems and especially quantitative approaches such as experimentation, the mentioned tool that have been lacking.

1.3 Quality Engineering (QE)

The American Society for Quality (ASQ) is a global community, which authorizes the certification of quality engineer. Considering their description of QE it encompasses both the quality management activities and the quantitative tools such as statistical quality/process control and designed experiments (http://asq.org/cert/quality-engineer/bok). The aim of QE is the improvement of current processes and to drive product innovations where process and product should be understood in the broadest possible sense (Box & Woodall 2012; Bisgaard 2012). Box (1996) defines quality improvement as the use of scientific methods for the continual generation of new knowledge. Knowledge is then categorized into 3 types; Existing knowledge not yet applied, knowledge obtained by observation of processes and knowledge generated experimentally. It is the total sum of this knowledge which can provide means for quality improvement. The connection to statistics stems from the use of the scientific method. The statistician's job is the constant development and improvement of the scientific method. The understanding of the interconnectedness of work, the understanding of variation's impact on or in these connections and finally the ability to quantify and control that variation is the basis of "statistical thinking" in QE ((Hoerl & Snee 2010), Paper C). As discussed in Paper C the future of QE is a promising field with new applications and opportunities introduced by technological innovation such as "big data" and advanced sensor technology like image data. The development of the field is likely contingent on gaining insight from practical problems. Such problems and applications could be gained from the food industry.

1.4 Definition of quality (QE approach)

The premise of quality presented here is based on Gavin (1984) and Montgomery (2009) starting with an informal list of quality attributes; performance, reliability, durability, serviceability, aesthetics, features, perceived quality and conformance to standards. All these attributes have varying interpretations and applicability depending on the application being either a material product (mobile phone, cookie) or immaterial (costumer service, design of packaging, "homemade" style). The term "fitness of use" has been used as a definition of quality. The quality attributes are the requirements for the product to be of "use" (Montgomery, 2009). The issue with the term "fitness of use" was that quality improvement activities focused more on conformance with requirements than designing and improving the process and product (Montgomery, 2009). As introduced in the section about food quality Peri (2006) translated the term "fitness of use" to be "fitness of consumption" with regards to food quality. Peri's goal was to discuss the concept of quality of food based on the idea that minimizing consumer rejection instead of maximizing acceptance is a more "holistic" approach (Peri, 2006). As mentioned earlier the proposed strategy is then to define the operating space (no rejection) and optimizing the food product within this space.

Montgomery (2009) presents another approach to defining quality with the aim of changing the strategy focus from compliance to conformance to a design process. The idea is to define quality as inversely proportional to variability. Thereby an impartial measure is given. The definition of quality as such only depends on some measurability that allows for quantifying variation. Variation is then an inverse measure of quality (Montgomery, 2009). Quality improvement is then the reduction of variation in processes and products. The FDA has to some degree tried to formalize this approach with the process analytical technology (PAT) initiative and quality by design (QbD) although it could be argued that the applications have been more in monitoring than design (FDA refs (Munir et al. 2015; van den Berg et al. 2012).

Design of experiments is part of the statistical tools which can be used to facilitate learning of the connections between processing and products, and facilitate improvement activities (Kulahci & Box 2003). Quality improvement is then connected both with a deeper understanding of the product itself but also to the factors defining the process. Box (1996) stresses the role of designed experiments in quality improvement by the use of sequential experimentation. One of the ideas behind sequential experimentation is that the benefits of controlling the conditions and the direction of inquiry outweigh the costs when compared to the strategy of using historical data (happenstance data as Box calls it). The importance is further emphasized by the idea that designed experiments can validate the informed extrapolation of subject matter knowledge when applied to improving actual process (Box 1996). It is the interpretation of Box's concept which is the central motivation and influencing the narrative of this thesis.

1.5 Perspectives

The important distinction between a scientist or statistician and the food / quality engineer is distinctly summarized in Jowitt (1982): "The engineer's use of mathematical models or models in general should not be dependent on the elegance of the mathematics but on the proper description of the physical phenomena under concern. If an elegant method is applied but the answer is wrong the engineer will not get half points." The importance lies in the ability to learn from the situation at hand and to extract as much information as possible. This is exemplified by the experiences of Bisgaard (1989) documenting the difficulties in addressing process improvements from historical data and the importance of experimentation as a learning facilitation. The history of FE is closely connected to the quality concept and the constant needed discussion of quality. The major driver has been the combination of industrialization, standardization and the need for mass production of safe foods. Within the recent decade a growth in the application of designed experiments for product and process improvement has been seen in FE (Granato & de Araújo Calado 2014). However, a division is unfortunately also seen between the statistical empirical approach and the physics based modelling endeavors (Saguy 2015). It is hoped that the issues stem from the misconceptions that have been reported on the use of statistical tools (Granato et al. 2014; Nunes et al. 2015) and that by showing and discussing the process of designing experiments the gap between the approaches can be narrowed. This also ties into a more successful approach to quality management systems with the findings that continuous improvement and customer satisfaction as the key success parameters (Kafetzopoulos & Gotzamani 2014). Both continuous improvement and customer satisfaction are central in the new interpretation of quality by Montgomery urging designing the processes (Montgomery 2009a)and evolutionary operation (EVOP) (Box 1957) which is method of using DoE principles in a continues process improvement strategy.

Within the thesis some of the basic concepts of DoE will be reintroduced and also applied to examples from FE. It is hoped that this can reinvigorate and shed a more nuanced light on the use of empirical models as an important central tool in the food engineers' toolbox. It is believed that the relationship between the different aspects (extrinsic and intrinsic) driving the understanding of food quality needs the combination of both mechanistic modelling and empirical methods. The two approaches are linked in the process of extrapolating from the general into the particular and vice versa, a process typically depicted in the design and implementation of new processes also referred to as process analysis and process synthesis (King et al. 1972; Hadiyanto 2007; Box 1996). The focus on DoE could possibly also broaden the collaborative work between FE and QE beyond monitoring and management systems.

Part 2 Basic Principles of DoE

In Part 1 it was mentioned that the reported use of statistically designed experiments (DoE) has increased in the literature of food science and engineering for the last decade. Table 2.1 shows an overview of some reported cases applying DoE in the Journal of Food Engineering which is one of the most noted food engineering journals. The findings show that advanced tools from the theory of DoE are in use. The designs most used which fit under the same narrative as the thesis is the responses surface methodology (RSM) designs. The RSM designs are typically built on a strategy using small sequential experimentation. The small designs used first are typically 2-level (low and high settings) full- or fractional factorial designs. These designs can then be followed up with further experimentation that builds directly on the used design or by moving the experimental area. The last step normally use central composite designs (CCD). The aim of the last step is to capture response curvature if existing. The idea is to fit a higher order model in the area of interest so as to capture possible minimum or maximum response and corresponding factor settings. The work by Yann et al. (2005) is an example of an iterative learning process supported by RSM. The goal in Yann et al. (2005) is to lower brown color formation inside cheeses. The experiment began with the use of a 2 level fractional factorial design for screening 5 proposed process factors of interest. The experiment was then followed up by a central composite design on the 3 significant factors identified in the screening experiment. The central composite design was used for modelling the response surface and thereby gaining a more detailed picture of which process settings to use. However, the example by Yann et al. (2005) is one of few examples found using the iterative process using sequential experimentation. The general findings which, are also exemplified by Table 2.1, show that the majority of the experiments have a minimum of two responses of interest. This is typical for food engineering cases due to the multifaceted issues in defining food quality (Part 1).

The examples highlighted in Table 2.1 show some general trends. The actual protocol of the experiment is often lacking in the description with respect to changing settings and randomization. The descriptions of the experimental units are often vague. Furthermore, lack of blocking against external nuisance factors is common. Especially the lack of blocking and the unclear description of randomization is a common occurrence, which extends further to articles not mentioned in Table 2.1. This is also corroborated by other findings in the related literature. In Granato & de Araújo Calado (2014) for example, there is no mentioning of how to randomize or what to do in instances where restriction on randomization is required. Blocking is only sparingly mentioned without showing any examples or references to the literature. Granato et al. (2014) gives general recommendations to analysis of a variety of experimental cases such as microbiology and sensory science, focusing on the use of univariate statistical tests. Nunes et al. (2015) extended the findings concerning tests for food science with an assessment of software tools and multivariate techniques. Both papers have a gap by sparingly mentioning design of experiments or blocking, randomization and restrictions to randomization. Even though the field of food science and engineering has seen a growth in the use of statistical tools, the applications often are misused or misrepresented (Nunes et al. 2015; Granato et al. 2014). This has been ascribed to a general difficulty in interpreting the theory of statistics when relating it to food engineering issues. The aim of this part of the thesis is to fill in the gap with a reintroduction to some of these basic principles for design of experiments.

Table 2.1 Examples of papers using designed experiments and statistical techniques within the field of food engineering. The findings have been summarized in order to give indications to the aim of the experiment, the size and the reported execution of it.

Application	# factors	Design	Exp runs	Clear description of protocol	Mentioning randomization	Distinction between experimental unit and repeated observation	# number of responses	Ref:
Process Optimization	3	CCD(nearly)	23	No	No	Yes	2	(Iwe et al. 2004)
Product/Process modeling	3	CCD(possibly)	25	No	No	Yes	2	(Iwe et al. 1998)
Product/Process modelling	3	CCD without center runs	14	No	Table presentation looks random	Yes	2	(Elbert et al. 2001)
Process optimization	3	OFAT	?	No	No	No	6	(Zhang et al. 2013)
Process optimization	4	CCD	31	No	No	Yes	1	(Wang et al. 2008)
Kinetic model	2	Kinetic (OFAT)	5	No	Yes	Yes	3	(He et al. 2014)
product modelling	3	Constrained Mixture	11	Yes	No	Yes	5	(Pelissari et al. 2012)
Product optimization	3	Mixture	21	Somewhat	No	Yes	8	(Liu et al. 2010)
Computer experiment	4	FCD	17	Yes	Not applicable	Not applicable	3	(Rodrigues et al. 2006)
Computer Experiment	3(not clear)	D-optimal	6(not clear)	No	Not applicable	No	uncertain	(Mannarswamy et al. 2010)
Model estimation	1	D-optimal	2	No	No	No	1	(Paquet-Durand et al. 2015)
Screening + optimization+ validation	5	Fractional and central composite designs	38	Somewhat	No	No	4	(Yann et al. 2005)
OFAT (one factor at a time), CCD (central composite design), FCD (face centered design).								

2.1 Design of Experiments

A short introduction to some basic concepts and notations are needed before discussing the concepts of randomization and blocking. There exits several text books that cover the topic in-depth so the aim is only to give enough information to make the discussion easier to follow (Box et al. 2005). The focus here is on the 2 level factorial design structure noted as 2^k factorial design where k is the number of factors or variables in the experiment. The idea is to fit a linear model that describes the response over the experimental region. The assumption is that we can meaningfully describe the "system" with a mathematical function or model. The unknown function can be approximated with a polynomial model of first or second order. There are several benefits of using the 2-level factorial design for engineering problems. The first is that 2-level factorial experiments are an economical way of testing out several factors or variables in the same experiment. This has a simple argument which is that for each factor included the experiment grows by a factor 2. It should be contrasted with cases as seen in Paper B where one factor has 3 levels and another has 2. In order to be factorial all the combinations of factors has to be performed. This gives 3*2 = 6 runs if another 2 level factor is added to this experiment its 12 runs. If all three factors were at 2 levels it gives 8 different combinations also called treatments. The 2^k design is an economical design in situations where all the factors are continuous; often the case for experimentation on production processes. There are other benefits to the 2^k design which will be mentioned throughout the description and the sub sections concerning randomization and blocking. However it should not be understood as 2^k design is the perfect tool for all situations. In Part 3 where the cases are presented other designs have also been used. The design type is chosen due to its simple structure which helps in presenting the other concepts, randomization and blocking etc.

Using an example which will form the basis for the following discussion we consider an experiment of cake mixtures to be sold at the supermarket inspired from (Box & Jones 1992). The focus of the experiment is to find the most robust recipe of ingredients. The robustness of the recipe is with respect to the time and temperature of baking. The motivation is that even though temperature and time may be specified on the packing no certainty can be given that the consumer can or would follow these instructions exactly. The manufacturer is therefore interested in having a recipe that gives a "good" product within sensible limits of how the consumer would follow the recipe. In the respective example this is achieved by testing out several temperature and time combinations and evaluating the cakes taste.

We will first look at the experiment only with respect to time and temperature, so as a 2^2 design. The response or measure under investigation is the taste of the cake. The taste of the cake is assessed in this example as an average of the hedonic score (a relative number of how good it tastes) given by a tasting panel. The next step is to choose reasonable levels of temperature and time for the experiment. This should be based on the subject matter expertise (which should be part of all the steps in designing the experiments). The levels of the factors for testing could be based on an assessment of what consumers actually do when given a recipe with stated time and temperature. In this example it could be the temperature and time specified on the box plus and minus 10^oC and 5 min respectively which would yield the temperature settings of 190 ^oC and 210 ^oC and baking times of 20 min and 30 min. The model assumed to describe the taste of the cake contains main effects of temperature (T) and time (t), and their interaction:

$$y= \beta_0 + \beta_1 T + \beta_2 t + \beta_3 T \times t + \varepsilon \qquad (2.1)$$

where y is the response (hedonic index), the β's are the coefficients relating the factors changes with the taste, and ε is the experimental error. The β_0 is the base level of taste experienced which is the overall mean or intercept. The experiment is to be performed at all combinations of the levels of factors. So using coded levels of -1 for the lower level and +1 for high level the combinations of the experiment can be written as in table 2.1.1.

Table 2.1.1 of the experimental conditions

Treatment	Temperature (T) Coded units	Time (t) Coded units	Temperature in ^oC Natural units	Time in min. Natural units
1	-1	-1	190	20
2	+1	-1	210	20
3	-1	+1	190	30
4	+1	+1	210	30

Table 2.1.2 shows the inputs to the model in coded units for equation (2.1). Note that a column of +1's is added for the intercept. The interaction term in eq. 2.1 is shown by the coded levels in its corresponding column. This can be achieved by the row wise product of the coded settings for temperature and time.

Table 2.1.2 experimental conditions interpreted into the model

Treatment	Intercept	Temperature (T)	Time (t)	T x t
1	+1	-1	-1	+1
2	+1	+1	-1	-1
3	+1	-1	+1	-1
4	+1	+1	+1	+1

Table 2.1.2 if formulated into a matrix can be used directly for estimating the factors effects with least squares regression and ANOVA for the analysis of the experiments but aswas discussed previously there are already several resources available for the analysis of the experiments (Granato et al. 2014; Nunes et al. 2015; Granato & de Araújo Calado 2014; Montgomery 2009a; Box et al. 2005). The following is focused on the practical implications of the design principles e.g. are how to perform randomization and what blocking constitutes.

2.1.1 Randomization

Randomization is the basic guard against extraneous unknown factors influencing the experiment undesirably. It is a protection against all the factors which cannot be deliberately controlled that might have an influence. For food experiments these uncontrolled factors are typically the environment (temperature and humidity of lab or production site) and gradients in raw material (e.g. the particle distribution in sacks of flour). By randomizing the material and or the treatments as in table 2.1.1 the effects from the nuisances can be averaged out. How and when to randomize in the experiment is case dependent (Greenberg 1951; Box 1990) and again not all relevant situations can be captured in the following presentation. Here we focus on examples which are deemed most relevant for food engineering applications like experiments involving the use of one oven (Box & Jones 1992). To be more precise this means experiments where all treatments combinations are performed in a time sequential manner and not at once. (Ganju & Lucas 1997) provided the definition that extends the notion of what a complete randomized experiment constitutes. The definition is that a complete randomized design is when treatments are randomly allocated. This means a randomization of the run order if the design is a factorial experiment and that all factors are reset between subsequent experimental runs. The typical procedure is the random allocation of predefined treatments to the experimental run order or experimental material. The aspect of run order is the special concept, which mostly pertains to industrial cases where the experiment is made on a specific unit operation. The experimental runs (treatments) are then performed sequentially. The plan for running the different settings are randomly chosen in order not to confound any of the investigated factors with unknown nuisances. A typical nuisance in food engineering is the environmental changes occurring throughout the duration of the day which could influence the product as mentioned with the baking experiment. Using the baking experiment as the example let the case be that only one oven is available. This means that each of the possible combinations of factor levels (i.e. a treatment which could be low time setting and high temperature) has been performed in sequence of each other. So the run order of the treatments are randomized (the allocation of the treatments). Let us consider two situations. Firstly, the example already used with two factors namely time and temperature, and the design is replicated. This would result in an 8 run experiment performed during a single day of experimentation. The second situation is where a third factor, using baking parchment (P), is included and the experiment is unreplicated which also results in 8 run (2^3). The treatments numbered from 1 to 8 are randomized using some software, which then will give a random permutation of the numbers which will be the run order of the experiment. An example could be 8, 7, 3, 1, 4, 2, 6, 5 resulting in the treatment numbered 8 is run first according to the protocol. If the experiment includes replication of treatments then all the treatments including the replications are randomized. In table 2.1.1.1 and table 2.1.1.2 both examples are shown.

Table 2.1.1.1 Complete randomization of a replicated experiment of the example presented in table 2.1.1

Basis design

Treatment #	Recoding #	T	t
1	1	+	+
2	2	-	+
3	3	+	-
4	4	-	-
1r	5	+	+
2r	6	-	+
3r	7	+	-
4r	8	-	-

Resulting design after randomization of treatment allocation

Run order	Randomized Treatment #	Original Treatment #	T	t
1	8	4r	-	-
2	4	4	-	-
3	2	2	-	+
4	7	3r	+	-
5	5	1r	+	+
6	1	1	+	+
7	3	3	+	-
8	6	2r	-	+

Table 2.1.1.2 Complete randomization of a unreplicated experiment testing temperature (T), time(t) and the use of baking parchment (P)

Basis design

	Treatment #	T	t	P
	1	+	+	+
	2	-	+	+
	3	+	-	+
	4	-	-	+
	5	+	+	-
	6	-	+	-
	7	+	-	-
	8	-	-	-

Resulting design after randomization of treatment allocation

Run order	Randomized treatment #	T	t	P
1	4	-	-	+
2	2	-	+	+
3	6	-	+	-
4	5	+	+	-
5	3	+	-	+
6	7	+	-	-
7	1	+	+	+
8	8	-	-	-

An under-emphasized aspect is what could be thought of as "the secret runs", which is the experimental procedures performed up to the execution of the first experiment and between each run. Improper factor resting can induce unwanted split plotting (Derek F. Webb et al. 2004). This essentially means a run over effect between runs which as example could be due to mounting of equipment. For the baking experiment this would be a relevant concern with respect to the temperature factor or the use of parchment. For it to be properly executed the oven should be either shut off and allowed to cool or have some intermediate setting between each experimental run even if the setting in the subsequent run is the same. Otherwise it would be the same as if using the same piece of parchment paper for the subsequent run when baking a new cake.

Another concern with regards to randomization is if treatments are performed on specific experimental material. The considerations are the same as in the case of the allocation of treatments. An example could be in the baking experiment with the case that one bag of flour is used for all the cake mixtures. The flour could have a gradient of particle distribution or humidity content from top down. The first portion of flour sampled might then have bigger particles or higher humidity content than the proceeding portions. The concern is then whether the material should be randomized too. The first option is to implement a homogenization of the material if possibly. This would constitute a physical version of randomization of the material (Petersen et al. 2004). However it could be deemed unnecessary due to the fact that the treatments allocation is randomized. The second option is to portion out the flour and randomize which portion goes to which experimental run. The choice between different randomization procedures in such situations is case dependent and up to assessment of the food engineer.

2.1.2 Replication

A short comment is needed with regards to replication. The need for replication with regards to experimentation and statistics is well known. The comment pertains to what entails proper replication. In table 2.1.1.1 is shown the complete randomization of the experimental design with the replication included. As can be seen the treatment combination #4 with level settings T at low (-), t at low (-) is replicated in the following experimental run. In between the runs some sort of factor resetting has been performed. One common misconception is that replication could be gained by baking two cakes at the same time i.e. having several observations from one experimental run. This would constitute a repeated measure and not a replication. The observations are not independent observations of the result of the experiment with respect the temperature setting and time. Some definitions are relevant for this subject used to help against the misconception; the experimental unit and the observational unit (Hinkelmann & Kempthorne 2012; Vining 2013). The experimental unit is the smallest unit to which a treatment is applied and the observation unit is the unit measured. These units can be the same entity as in baking one cake at certain oven temperature and for a given time. If baking two like cakes at the same time then there are two observational units but only one experimental unit because they (the cakes) are in the same oven at the same time and the experiment looks at the effect of different oven settings.

2.1.3 Blocking

Blocking is a means of accounting for known sources of variability affecting the experiment which cannot be directly manipulated. This could be several similar process units where the same 2 temperature settings are tested and the resulting product from each process unit at each temperature setting is analyzed. The blocking could then be with respect to the different process units. The resulting experiment is focused on the effect of the temperature on the product and the difference between the process units is removed from that analyses i.e. blocked. A more precise description blocks are where experimental units are more homogenous within the blocks than between the blocks. This statement implies that we expect that products produced with one process unit to be more alike than compared to other similar process units. The assignable variability is included in the experimental design by randomization of the treatments within each block. This would mean randomizing the protocol of the temperature settings independently for each process unit. There are some classic sources of variability that are typically blocked in engineering cases. These would be days, production lines, material from different manufactures or from different batches etc. Blocking is to improve the precision of the experiment by accounting for extraneous sources of variation, which can be partitioned from the experimental error. The block assignments themselves are not randomized otherwise the nuisance- factor or sources in question should be readdressed. Returning to the baking example from table 2.1.1 .1 with replication of the 2^2 experiment. A possible constraint on the experiment could be that only 4 experimental runs can be performed per day. This means that the 8 experiments should be allocated across 2 days. The experimenter's assumption could be that the environmental conditions are more similar within a day than between days. The first 2^2 design is performed on the day 1 and replicated on day 2. Within each day the experimental run order is randomized. Table 2.1.3.1 shows an example of this type of blocking.

Table 2.1.3.1 Randomized block design of a replicated experiment

Basis design

Treatment #	Block (day)	T	t
1	1	+	+
2		-	+
3		+	-
4		-	-
1r	2	+	+
2r		-	+
3r		+	-
4r		-	-

Resulting design after randomization of treatment allocation

Run order	Randomized Treatment #	Block	T	t
1	3	1	+	-
2	2		-	+
3	1		+	+
4	4		-	-
1	2r	2	-	+
2	1r		+	+
3	4r		-	-
4	3r		+	-

As mentioned, blocking could be performed with respect to batches of material. The situation could be that there is not enough material from one batch to perform all treatment combinations. This would need a second batch of material in order to perform all the experimental treatments. If the assumption is that there is bigger difference between batches than within it would be meaningful to block for the batches. A resembling situation could be case that not all experiments can be performed during a day of experimentation as with the blocked baking example where the replications had to be performed on another day. The time constraint could be more severe. A situation could be that only 2 runs could be performed per day or stated more generally not all treatment combinations or replication can be executed within a block. For both cases (not enough material in one batch or not enough time) the experimental design is fractionated or partitioned out between the blocks. This has the consequence that the effect of one of the factors is confound with the effect of the block. The positive thing is that it can be controlled which factors effect is confound by ensuring that the factor has unchanged signs (all + or -) within each block. Using again the baking example where 4 experimental runs can be performed within a day, the use of baking parchment is now included as a factor in the investigation. This results in a 2^3 design where the experimental runs are fractioned out between the days. A 2^3 design requires 8 experimental runs and only 4 runs are allowed per day so the experiment has to be fractioned out. The following described method is also applicable if the experiment was to be replicated which would require 2 more days of experimentation (8*2 = 16 runs with 4 runs per day gives 4 days). The preliminary design and the resulting design with blocking are shown in table 2.1.3.2. As mentioned the experimenter can control which factor's effect is confounded with the block effect. The sensible choice is to take the highest order interaction which is the T x t x P interaction based on assumptions like the Pareto principle. The Pareto principle implies in this context that the active factor space is frequently smaller than the design space (Box et al. 2005). Then organizing the design so that all the treatments with the T x t x P interaction at low level is allocated to 1 block and randomized. This would mean that the treatments numbered 2, 3, 5 and 8 in the basic design are performed on day 1. These numbers are then randomized to get the run order of that day of experimentation. The procedure is repeated for the second day experimentation where the signs for T x t x P interaction would be +. The resulting design is shown in table 2.1.3.2. If the experiment was replicated the procedure would be the same allocating treatments with the same signs for T x t x P interaction to be within a day of experimentation.

Table 2.1.3.2 Randomized block unreplicated design with higher order interaction confounded

Basis design

Treatment #	T	t	P	TtP
1	+	+	+	+
2	-	+	+	-
3	+	-	+	-
4	-	-	+	+
5	+	+	-	-
6	-	+	-	+
7	+	-	-	+
8	-	-	-	-

Resulting design after randomization of treatment allocation

Run order	Randomized Treatment #	Block	T	t	P	TtP
1	3	1	+	-	+	-
2	5		+	+	-	-
3	8		-	-	-	-
4	2		-	+	+	-
1	6	2	-	+	-	+
2	7		+	-	-	+
3	4		-	-	+	+
4	1		+	+	+	+

2.1.4 Restrictions to randomization

In the preceding example blocking is used in order to accommodate a restriction to completely randomizing an experiment across days (table 2.1.3.2). However, other situations could lead to restrictions on how freely the level settings can be changed due to economical or physical limitations. These limitations could pertain to one of the factors under investigation. It could be that resetting the experiment with respect to one of the factors would make the experiment too expensive or the duration too long. As in the baking example of Box & Jones (1992) it could be sensible to bake all the possible cake mixtures at the same time for each of the possible temperature and time settings. This is what constitutes a split plot experiment which Kowalski et al. (2007) supplies a succinct tutorial on. The example often used to illustrate the situation is that one of the factors under investigation is difficult to change between experimental runs. This might be due to mounting of special equipment or that the resetting of a factor would cost too much in respect to the overall time or be to expensive. The idea is then to reduce the number of times the factor is changed in an experiment. The other factors in the experiment are then varied independently (sub plot) for each level of the hard to change factor (whole plot). Thus the normal wording for split plot when used in this context is the easy-to-change factors, which are the subplots and hard-to-change factors e.g. the whole plots. The caveat is that the restriction gives separate variance components for the hard-to-change and easy-to-change factors respectively. (Ganju & Lucas 2005) discussed the importance of recognizing the resetting of factors between subsequent experimental runs. If this is not done then the experiment will result in unintended split-plotting which was alluded to in section 2.1.1. Let us look at the baking experiment again, now in a form resembling the original presentation of the problem (Box & Jones 1992). Two examples can be constructed for the split plot case. The one example might be that the oven is of industrial size and therefore changing and resetting the temperature between each experimental run would take too much time. The industrial baking case could then have two other variables (sp1,sp2) which are still easily changed. The randomization process would be first to randomize the oven setting and then to randomize the settings of the other two factors within the oven designated oven setting. Another example is that a normal oven is used and the experimenter has two ingredients which are to be varied in the cake mixture. The oven has the possibility of baking four cakes at the same time. Thus the oven could be set at a temperature and the four possible cake mixtures combinations of the ingredients are baked at that temperature. Here the randomization is with respect to the temperature setting only. Both situations are equivalent with respect to the experimental design as shown in table 2.1.4.1.

Table 2.1.4.1 Split plot design with T(h-t-c) and sp1, sp2 (e-t-c)

Basis design					
	whole plot	T	Subplot	es1	es2
	1	-	1	+	+
		-	2	-	+
		-	3	+	-
		-	4	-	-
	2	+	1	+	+
		+	2	-	+
		+	3	+	-
		+	4	-	-
Resulting design after randomization of treatment allocation					
Run order	Randomized	T	Subplot	sp1	sp2
1	2	+	1	+	+
2		+	2	-	+
3		+	4	-	-
4		+	3	+	-
5	1	-	4	-	-
6		-	1	+	+
7		-	3	+	-
8		-	2	-	+

2.2 Discussion

The preceding presentation is short and merely for introductory purpose of some of the principles of DoE found to be lacking in food engineering literature. There exists several resources, specifically Box et al. (2005) and Montgomery (2009) can be recommend for in-depth introductions on the topics and also more extensive presentations of actively using sequential learning and experimental designs. With regards to the findings in table 2.1 and in the papers (Nunes et al. 2015; Granato & de Araújo Calado 2014; Granato et al. 2014) the presentation of the principles should convey some of the relevant considerations to be made when designing an experiment. The focus has been on the practical interpretations of concepts. Such as how to perform randomization and how the result experimental protocol should be executed etc. This is important in order to qualify the necessity and the proper application of randomization and blocking to the problem under investigation.

General considerations concerning whether to randomize or not, are still vague. This pertains to all aspects at least in regards to the implications of performing; complete randomization, no randomization but proper resetting of the factors and randomizing a design but not resetting a factor. Regarding the first two stated scenarios performing complete randomization and no randomization nonetheless proper resetting of factors should essentially be equivalent if the assumption of stationarity of the system holds (Box 1990). At least this will be the case for smaller sized experiments run in one campaign or in blocks where the possible nuisance factors have a small impact or are dissipated due to the experimental resetting. If the design is fractional or saturated then the probability of the factors effects being confounded with error increases however this is irrespective of whether the design is randomized or not. So should the design be randomized? The answer still falls back to the same arguments as George Box presented (Box 1990). Randomize the design if it does not complicate anything. (Greenberg 1951) showed a small practical laboratory example of a comparison test where the principles of systematic design, randomization and blocking were discussed. The discussion provided by Greenberg (1951) showed that it was the practical choices made in the execution of the experiment which had a profound impact. The example is of two groups of mice a control and an immune group where both groups are inoculated with parasites and the immunity is tested. The discussion concerning different statistical procedures quickly develops into a discussion corning different manual procedures such as changing syringes or cleansing the needle between inoculations. The paper is a good presentation of the difficulty in interpreting and implementing the basic experimental design protocols. The major takeaway point from Greenberg (1951) is the importance of using blocking. As has been mentioned previously (section 2.1.3) blocking is an active method of accounting for external or unwanted sources of error.

Randomization can be seen as a passive way of protecting against extraneous sources of noise that is unknown to the experimenter. The discussion concerning whether to randomize or not can be dated back to Fisher and Student (William S. Gosset) (Student 1938; Barbacki & Fisher 1936; Neyman & Pearson 1938). Cox (2009) supplied a review on the role of randomization in experimental design which provides an historical overview of the theoretical developments from the most influential papers. Freeman et al. (2013) mentioned that much work only repeats the intentions of the randomization process and rarely provides enough contexts for non-statistician or even for statisticians to understand the implications of randomization in experiments. The issue repeatedly shown in papers that there arguably is no all-encompassing way of describing and implementing randomization that will pertain to all experiments. There is a nonobvious benefit of a randomized experimental design. The generated protocol is not a straightforward and systematic execution, but a protocol that demands the attention of the experimenter. This could be positive influence on the attention to the experiment. This also has a direct connection to the important case mentioned concerning the situation of having randomized the design but not resetting the factors. This issue has been addressed by Ganju & Lucas (1999) and Ganju & Lucas (2005) showing and discussing examples from the literature. For clarification the point concerning the benefits of the randomized design in connection with the actual execution is rephrased and reiterated. The normal operating conditions of a laboratory, pilot plant or production line is a high degree of systematic and repetitive procedures. The problem is that this can also dull the attention of the person performing the procedures. With a randomized design attention is demanded that forces the experimenter to confirm each time between experimental runs that levels are set at correct settings for the run. The issue is then whether a proper resetting has been performed. The unintended split plotting or run over effect that results from not resetting a factor properly can inflate experimental errors or confound factors effects with unknown sources. Ganju and Lucas (1999) argue that the unintended split plotting is the "worst" possibly situation. Clear communication is required between the experimental designer and the person's executing the experiment in order to remove such possible complications. If such complications is present and the analyst knows this, then Box (1990) recommends to analyze the experiment anyway. The danger is when the complications exist and the analyst does not know about it which could skew conclusions. In the following Part 3 presenting the cases several of the discussion points from this Part 2 will be revisited. In all of the experiments presented in Part 3 a randomization procedure has been used. An important difference in the examples presented compared to the findings in the literature (table 2.1) is that blocking has been used more extensively. As the preceding discussion points towards a general rule for randomization cannot be given, only case specific considerations and the advice to do it if it does not complicate execution. An essential point concerning this is that no experiments have been shown where direct comparison has been made between a randomized and a structured design relevant for engineering purpose making the comparison possible.

Part 3 Case presentations

This part presents the application of designed experiments to food engineering problems. Four examples are presented all based on engineering problems with particular focus on process and product interactions or process characterization. The presented cases are based on work in direct collaboration with companies focusing on quality issues or improving processes (Paper A and Report I), or inspired from work with companies (Paper B and Report II). The companies are 2 process equipment developers and 3 manufacturers. One of the process equipment companies is specialized in novel technology (BCH Ltd) such as vacuum cooling and ohmic heating and the other company (Haas-Meincke A/S) is specialized in processing equipment for bakery and confectionery unit operations. The 3 manufacturing companies are Royal Greenland A/S producing fish products, and Easy Food A/S and Bisca A/S producing bakery goods. This means that the applications have an appreciably broad scope and touches upon industry sectors other than dairy and ingredients technology which have been the sectors with most applications reported connected to DoE and statistical thinking in the literature (Munir et al. 2015; van den Berg et al. 2012; Granato & de Araújo Calado 2014).

The examples are presented in short format excluding some of the subject specific literature review which is described in the related papers. Instead the discussion will focus on describing the thoughts behind the experimental design process together with some considerations or adjustments which could have been made following the remark by R.A. Fisher “the best time to design an experiment is after you have done it” (Box 2001). The intention is to give some inspiration on how to tackle the experimental design process, by describing and discussing the choices made for the design structures. The common thread for all the examples is the considerations made concerning the experimental units, blocking and randomization. As was presented in Part 2 presentations and clear descriptions of the experimental design are often lacking in these crucial details. It cannot be claimed that the presented cases have solved this issue. However, the hope is that the combination with the discussions provided along with the cases could instigate a discussion on the greater use of experimental designs and experimental unit structure in food industry and serve as inspiration. The issues when presenting experiments could possibly be due to no clear standardization of the method sections in the different papers/journals within FE. A possible solution could be to adopt and develop graphical tools such as the factor relationship diagram (Bergerud 1996) advocated by Sanders & Coleman (2003) whom had the same concerns with presentation stile of the actual experimental units. Such tools could help in more clear description of the DoE performed.

The first example concerns the application of ohmic heating for cooking shrimps (Paper A). The example constitutes several small experiments such as paired comparisons, classic comparative, experiments (one way ANOVA) and a 2 level factorial design. The experiments were conducted in a bench top version of the unit operation and the aim of the experiments were to clarify several questions about ohmic heating of shrimps. Hopefully, the outcome would provide both a proof of concept and more detail learning of the physics in relation to ohmic heating. The motivation was a company looking at alternative means of heating shrimp that could theoretically address some of the current quality issues. The application of ohmic heating to seafood was also a novel research question.

The second example illustrates the use of a fractional factorial design (Report I). The situation concerns a screening experiment on a pilot scale impingement oven focusing on identifying how and if the variables influence the process conditions. Firstly the variables/factors were the possible factors that could be changed on the equipment such as temperature, air speed, fresh air intake etc. Secondly, by process conditions is meant the measured convective heat transfer, which relates to how fast the air will heat the products. The experiment was motivated by the collaborative work with a company designing and producing impingement ovens. The pilot scale oven was also designed in collaboration with said company (Andresen et al. 2013). There were two main questions which the experiment had to address; firstly was the oven applicable for the kind of experimentation it was intended for and secondly, if so then which factors impacted the process conditions. Included in the discussion of this example is also a discussion on designing experiments on the fly during execution but relying on some the DoE principles.

The third example involves a central composite design (Report II). The focus of the study was to evaluate the effect of the amount of reuse dough and resting time on the resulting material properties of the dough sheets. The definition of reuse dough or scrap dough is given later. The example highlights the intricacies in clear problem formulation and definition of the material under investigation. The motivation for the research question was based on observations made in working with the industrial bakeries. The tradition of using reuse dough is common but the implications and the reasons behind, other than as an attempt to lower waste, are unknown and unquantified. This was also a research problem that has not been investigated when previously reviewing the scientific literature.

The last example is a mixed level factorial experiment with a multivariate response which demands novel methodologies with respect to the analysis of the experiment (Paper B). The focus of the experiment was on evaluating different types of baking sheet design. The baking sheets were small versions of industrial type baking sheets. The evaluation of the sheets was made by baking cookies in impingement air convection oven. The types of design used meant that the air speed was included as a factor. Images of the cookies were taken in order to get quantitative measures on their appearances. Each image is a multivariate response of 3 dimensions; two spatial and one spectral. The paper shows one approach on how to preprocess the image data and then how to combine all the images and analysis with respect to the experimental investigation.

Example 1: Ohmic heating of shrimp (Paper A)

Ohmic heating uses electric current for resistance heating a material. The normal application in food processing is through two electrodes either in direct contact with the food material or submerged in a conducting liquid containing the food, and then to apply a current across the system. If the food is conductive it will then start to heat evenly throughout the material (Varghese et al. 2012). The aim of this study was to test out the efficacy of using the unit operation for heating shrimps. The work was a sub project in collaboration with Royal Greenland A/S trying to address quality issues with the currently used steam cooking process. As described in Paper A shrimps have varying sizes, which due to the process conditions, resulting in some shrimps being over-processed resulting in decreased quality and yield. The manner in which ohmic heating works could theoretically solve some of these issues. The strategy was then through a series of experiments to address certain key questions; was the ohmic heating of shrimp size independent, what was the effect of brine concentration and electric field strength (voltage applied to the system) on the cooking time, and did the pretreatment of the shrimps influence the ohmic heating? The order of the questions is presented in the same order as they were posed during the experimentation. The first question was a preliminary qualifier for whether the technology could be used on shrimp or not. This was followed up by screening the ohmic process conditions and evaluating several product quality attributes of the cooked shrimps. The final question was to investigate whether the results observed was dependent on the pretreatment used in connection with the ohmic heating. The questions and the resulting experiments were formed also with the intent of clarifying some of the physics happening during the process. The intent of observing and supplying data of the physical process was also a part research initiative for formulating physically based models. The questions posed gave central insights into the complexity level needed of such models.

An investigation on the application of ohmic heating of cold water shrimp and brine mixtures

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Abstract

Cooking is an important unit-operation in the production of cooked and peeled shrimps. The present study explores the feasibility of using ohmic heating for cooking of shrimps. The focus is on investigating the effects of different process parameters on heating time and quality of ohmic cooked shrimps (*Pandalus Borelias*). The shrimps were heated to a core temperature of 72°C in a brine solution using a small batch ohmic heater. Three experiments were performed: 1) a comparative analyses of the temperature development between different sizes of shrimps and thickness (head and tail region of shrimp) over varying salt concentrations (10 kg m^{-3} to 20 kg m^{-3}) and electric field strengths (1150 V m^{-1} to 1725 V m^{-1}) with the heating time as the response; 2) a 2 level factorial experiment for screening the impact of processing conditions using electric field strengths of 1250 V m^{-1} and 1580 V m^{-1}and salt concentrations of 13.75 kg m^{-3} and 25.75 kg m^{-3} and 3) evaluating the effect of pretreatment (maturation) of the shrimps before ohmic processing. The maturation experiment was performed with the following maturation pre-treatments: normal tap water, a 21.25 kg m^{-3} brine solution and without maturation. The measured responses for experiments 2 and 3 were: the heating time until the set temperature of the shrimps was reached, weight loss, press juice and texture profile. It was possible to fit the main effects model relation between electric field strength and salt solution applied and the heating time, weight loss and press juice measurements. Furthermore, the results showed that the shrimp achieved a comparable quality compared to the conventional heating processes reported in the literature and that over the tested process work space no significant changes were seen in the texture measurements. The findings show a promising utilization of ohmic heating as a unit operation for the shrimp processing industries.

Keywords:

Ohmic heating; Voltage ; Field Strength; Salt Concentration ; Shrimp ; TPA

1. Introduction

Optimal heating of solid foods using the conventional technologies can be challenging due to the fact that heat transfer is limited by internal conduction. Heating of the shrimps can be problematic due to the size variation within a batch of shrimps. The size variation within a batch induces over-processing of especially the smallest prawns in the batch. This means that in order to process according to safety criteria and to meet product specification there is a certain risk for deteriorated product quality and yield loss.

Ohmic heating is a technology, which potentially heats the product volumetrically by passing an alternating electrical current through a conductive food material (Sastry, 2008). The volumetric heating can reduce or eliminate temperature gradients within the shrimps and thereby alleviate overcooking issues. However, this is conditional on the electric conductivity of the product - this means that- in the case of products with spatial variation in the electric conductivity due to heterogeneous spatial composition spatial differences in the heating profiles can be induce. Ohmic heating is an old application that has been under development for a long time and in recent decades seen a rise in research and development. The application of ohmic heating has been studied for various applications and food stuffs, and the research results have been extensively reviewed (Kaur & Singh, 2015; Knirsch et al. 2010; Sastry 2008; Varghese et al. 2012). Salengke & Sastry (2007) investigated the temperature distribution when using ohmic heating of solid-liquid mixture in both static and mixed (agitated) conditions for cases with a difference in electric conductivity between the inclusion particle and the surrounding medium. The authors observed that for the cases where the particle was more conductive than the medium the cold spot was within the medium in zones parallel to the particle (also referred to as shadow regions). Additionally, it was found that the process conditions (static and agitation) and particle size had an impact on the temperature development. This occurrence in solid-liquid mixtures has been observed and studied by several other authors (Davies et al. 1999; de Alwis et al. 1989; Fryer et al. 1993). Such phenomenon is highly important to understand and to be able to determine the slowest heating location (cold spot) within the system to secure food safety.

Some possible benefits of ohmic heating include reduced burning of the surface, better nutrient and vitamin retention and increased energy efficiency and environmentally friendly systems (Sensoy, 2012). The study on ohmic heating of meat and meat products is a growing field (McKenna et al. 2006; Sarang et al. 2008) although the application of ohmic heating on portions of fish and crustaceans reported in the literature is sparse. The use of ohmic heating for thawing of shrimps has been reported (Roberts et al. 1996; Roberts et al. 1998; Roberts et al. 2002). The results were compared against water immersion thawing and showed comparable findings in microbial safety, quality and weight loss.

Several studies have reported on the influence of traditional thermal processing of shrimps with heated or with boiling water, and the resulting impact on product quality (Erdogdu et al. 2004; Erdoğdu & Balaban, 2000; Mizuta et al. 1999; Niamnuy et al. 2007; Niamnuy et al. 2008; Murakami, 1994). In these studies the quality assessments were on textural properties either with instrumental measurements or sensory evaluation, water loss and safety assessment of microbial inactivation. Erdoğdu & Balaban (2000) reported on the change in texture of thermally processed shrimps and correlated the findings with sensory assessment showing a general higher acceptability of minimally processed shrimp. Niamnuy et al. (2007) studied the impact of boiling shrimps in a brine solution and showed that time was the significant factor for the observed changes in texture and shrinkage.

The scope of this study is to provide knowledge on ohmic heating of shrimps which could be either used as a pretreatment or the main cooking operation. The experiments performed address factors such as influence of shrimp size and ion (salt) concentration in the brine, which in the literature has been identified as important variables in relation to ohmic heating (Sastry, 2008). The experiments were planned in a stepwise manner. *The first step*; is to assess the effect of size variation and the effect of spatial variation within the shrimp (head and tail) with respect to the temperature profile. *The second step*; is to evaluate the effect of the ohmic heating process variables (electric field strength and salt concentration) on the process time and quality of shrimps (weight loss and texture). *Finally, the third step*; is to evaluate the effect of preprocessing (maturation step) on the process time and quality of shrimps (weight loss, press juice and texture) when processing with the ohmic heating. The maturation step is a common unit operation used both as a buffer and pretreatment before heating of shrimps in the industry to promote the peeling in the later stages. The overall intention was to identify the possible process conditions for industrial implementation of ohmic heating and verification of the feasibility of the unit operation. The responses chosen for the experiment were: the time until a core temperature of 72^{o}C was reached (standard processing conditions), press juice, weight loss and texture of the cooked shrimp.

2. Materials and Methods

2.1 Ohmic heater

The ohmic heater used in this study was built by BCH ltd. (Lancashire, UK). The unit consists of a holding cell made of W500 grade polyethylene with variable size adjustment and mountings for temperature loggers (K-type). The ohmic heater unit can maximally supply a 230 voltage using alternating current (60Hz, sinusoidal). Titanium electrode was used which has high corrosion resistance in chloride environments (Samaranayake & Sastry, 2005). The distance between the electrodes was set at 12cm apart; the width of the chamber is 9.5cm and the liquid height including shrimp approximately 4.5 - 5cm.

2.2 Raw Materials

Raw frozen shrimps (*Pandalus Borelias)* were supplied by Royal Greenland A/S (DK). The shrimps were kept in cold storage (-18^{o}C) until testing at the Technical University of Denmark. The individual shrimp weight varied from approximately 8-13g.

2.3 Experimental Procedure

The shrimps were matured in accordance with the following procedure: first the shrimps were defrosted in tap water and then placed in a specified brine solution for 24 hours at refrigeration temperature of 0-5^{o}C. The concentration of the brine solution was prepared as weight of salt to the total volume of water and salt ($m_{salt}/v_{water+salt}$) with the specific brine concentration shown for each of the experimental conditions in the reported results tables. The water was normal tap water at approximately 15-20^{o}C. 20 shrimps were heated in each experimental run which approximately corresponds to total weight 220g of shrimp. For the ohmic heating process a new brine solution corresponding to the maturation brine concentration was prepared, and together with the shrimp added to the ohmic heater. The ratio of shrimp to brine used in the ohmic heater was approximately 1:2 in the weight respectively. The placement of the shrimps was in a parallel position of the body with the electrode plates and the shrimp inserted with a thermocouple was placed perpendicular to the bottom of the cell with the head pointing up (the exact thermocouple placement within the shrimp for each experiment is described in 2.6, 2.7 and 2.8). The shrimp and brine were then heated with the ohmic heater until a measured core temperature in the shrimp of 72°C was reached. The time, temperature and electrical current were recorded during ohmic heating of the shrimps. After the ohmic heating the shrimps were cooled in excess ice water for five minutes. The weight of each batch was recorded before the heat treatment and immediately after cooling for the assessment of weight loss. The shrimps were then placed in plastic bags. The samples were allowed to thermoset in the water at room temperature (20-25°C) for an hour before texture profile analysis (TPA) and press juice measurements (PJ) were performed.

2.4 Texture profile analysis and press juice measurements

Two shrimps from each experimental run were peeled before TPA and PJ measurements respectively. The measurement protocols are the same as used by Erdoğdu & Balaban (2000) and Niamnuy et al. (2007). For both the TPA and the press juice measurements a Texture Analyzer XT.Plus (Stable Micro Systems Ltd. UK) was used with a cylindrical probe with Ø of 4cm on plane surfaces. The compression was done at constant speed 1mm/s to 60% deformation with 0.1 sec pause between the compression readings. The PJ test was made with filter paper on top and underneath the shrimp, which was then sustained a 30 sec compression at 1mm/s constant speed and a 122.5N load cell. The shrimp and filter paper were weighed before and after compression and the response was percentage of press juice of shrimp mass.

2.5 Data analysis

The statistical analysis was performed using both Microsoft Excel 2010 (USA) and R (R development core team, 2014).

2.6 Assessment of heat distribution in shrimp and effect of size differences on the heating rate

Paired temperature measurements were performed. One thermocouple was placed in the head (fig. 1, zone A) and one in the tail part of the shrimp (fig. 1 zone C), and the temperatures were recorded to evaluate the difference in the heating rate according to position. In the experiment for testing the effect of differences in shrimp size paired temperature measurements were performed where thermocouples were placed in the smallest and biggest of the shrimps for each batch. The thermocouple was placed in the thickest part of the shrimp between the 2nd and 3rd segment (fig. 1, zone B). The shrimps were then heated and the time to reach a core temperature at 72°C was recorded for each shrimp. For robustness considerations of the results both set of experiments were replicated over varying voltages and salt concentrations. The statistical tests performed were paired t-test against an alpha value of 5%.

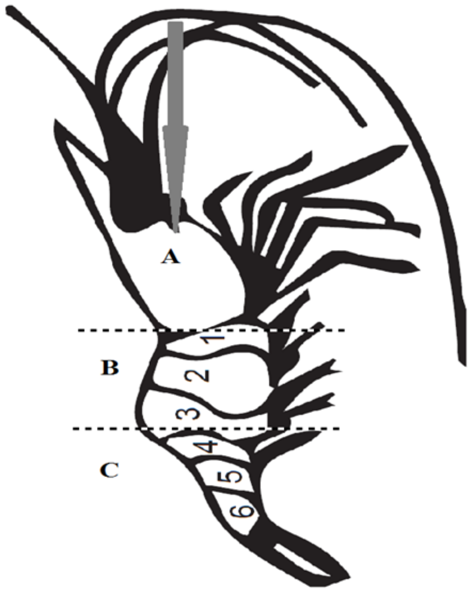

Figure 1 Diagram of shrimp with numbering of segments. The letters mark zones for the temperature measurements. The temperature measurement was made in the head (A), tail meat thickest part (B) and in tail meat thinnest part (C). The arrow indicates the direction of the thermocouple placement.

2.7 Effect of ohmic process conditions

The experimental design used was a replicated 2 level factorial design fully randomized. The salt concentration and the electric field strength were varied at 2 levels, respectively; salt concentrations of 13.75 kg m^{-3} and 25.75 kg m^{-3} and electric field strength of 1250 V m^{-1} and 1580 V m^{-1}. The amount of water and shrimp were held constant. Maturation of the shrimps before ohmic heating was performed with a brine solution similar in the salt content to the subsequent processing brine concentration. Each of the experimental runs constituted an individual brining and processing step thus the replication refers both to maturation and heating. The temperature was measured in one shrimp using a thermocouple placed in zone B (Fig. 1). ANOVA and regression analysis was made on the model containing main effects and interaction. The model equation is given by Eq.1.

$$Y = a_o + a_1 x_1 + a_2 x_2 + a_3 x_1 x_2 \qquad (1)$$

where the Y is the response, a_o is the intercept, x_1 is the electric field strength, x_2 is the salt concentration, and x_1x_2 is the interaction term.

2.8 Effect of maturation

The effect of the maturation process before heating was addressed by preparing samples of shrimps that were matured in three different ways and subsequently heated under the same settings in the ohmic heater at 1417 V m^{-1} and 21.25 kg m^{-3} brine solution. The settings for the process conditions in the ohmic heater are roughly at the center point of the 2 level factorial experiments (section 2.7). The first maturation preparation method was to thaw the shrimps as described in the standard procedure and mature in normal tap water for 24 hours at refrigeration temperature. The second maturation method was maturation in 21.25 kg m^{-3} brine solution for 24 hours at refrigeration temperature. The third method was to simply thaw the shrimps according to the standard procedure as described in section 2.3 and immediately apply ohmic heating. Each maturation method and the thawed shrimp preparation were replicated twice. Core temperature profile of one shrimp was recorded for each experimental run with the thermocouple placed in zone B (fig. 1). A one-way ANOVA model was used for the statistical test.

3. Results and discussion

3.1. Assessment of size variation and head and tail heating times

Table 1 shows the results from the paired t-test performed on the obtained heating times of the shrimps classified as the biggest and smallest in each batch. The goal of the experiment was to identify if volumetric heating is taken place in the shrimps. The results showed no significant differences between the different sizes of shrimps according to heating time. The results further verify that this finding is irrespective of the voltage and salt concentration used. For the time difference between the head and the tail part to reach 72°C we see a significant difference in heating time over the varying process conditions depending on the position of measurement (Table 2). The results show that the tail part of shrimp is heated slower compared to the head part of shrimp. The results also indicate that this difference disappears as salt concentration and voltage is increased which at the same time reduced the heating time. These findings implied that the size of the shrimps was not of concern for the following experiments, screening the process conditions and assessing maturation (section 3.2 and 3.3). The evaluation concluded that the temperature measurements should be made in the tail meat part which would give the heating profile of the possible cold spot.

The results from the experiments on size variation and spatial differences indicate a volumetric heating of the shrimps shown by the small (0-2 sec.) to no differences in heating time either between big and small shrimps as well as between head and tail part at higher salt concentrations (20 kg m^{-3}) and electric field strengths (1533-1725 V m^{-1}) (Table 1 & 2). This was possibly due to the shrimps and brine being at comparable electric conductivity at the higher salt concentrations. Temperature zones have been observed with ohmic heating of solid-liquid mixture with differing electric conductivities between the solid and the liquid (Salengke & Sastry. 2007). This could be part of the explanation for the spatial differences (head and tail meat) and the differences between sizes observed at lower salt concentrations.

Table 1. The effect of the size of shrimp on the heating time – measured data and results from paired test

Salt concertation (kg m^{-3})	Electric field Strength (V m^{-1})	Time to reach 72 in seconds		Difference
		Big	Small	
10	1150	54	49	5
	1342	47	43	4
	1533	37	37	0
	1725	27	27	0
15	1150	47	51	-4
	1342	38	33	5
	1533	26	26	0
	1725	23	23	0
20	1150	36	36	0
	1342	28	38	-4
	1533	25	23	2
	1725	17	16	1
	T-test (two-sided paired with H0: Mean diff = 0)		P-value	0.8372

Table 2. Measured data and results from paired test on the head and the tail part of shrimp differences in heating time

Salt (kg m^{-3})	Electric Field Strength (V m^{-1})	Time to reach 72 in seconds		Difference
		Tail	Head	
10	1150	58	52	6
	1342	47	42	5
	1533	33	33	0
	1725	28	28	0
15	1150	45	39	6
	1342	35	32	3
	1533	28	25	3
	1725	24	24	0
20	1150	36	36	0
	1342	32	32	0
	1533	24	24	0
	1725	22	22	0
	T-test (two-sided paired with H0: Mean diff = 0)		P-value	0.0240

3.2. Effect of ohmic processing conditions on heating time and quality

Table 3 presents the results from the factorial experiment performed on the impact of processing conditions on product quality. The TPA and PJ measurements for the individual sampled units are shown; however, the statistical models are based on the mean of the two samples for each run. This means that the models fitted are based on the replication of the process variables (electric field strength and salt concentration) and an averaging over the shrimp to shrimp variation.

Table 3 Experimental settings and results from replicated 2^2 factorial design in the structured order.

								Texture measurements					
Run order	Exp nr	Field strength (V m-1)	Salt concentration (kg m^{-3})	Time to 72 °C	Weight loss (%)	Sample Unit	Press juice (%)	Adhesiveness (N*mm)	Hardness (N)	Springiness (mm)	Cohesiveness	Gumminess (N)	Chewiness (N*mm)
1	1	1250	13.75	53	12.77	1	13.57	-0.03	0.75	3.20	0,70	0.48	1.54
						2	16.91	-0.02	3.37	3.89	0.76	2.41	9.38
6	2	1580	13.75	40	8.35	1	11.92	0.00	3.45	3.52	0.88	2.94	10.33
						2	16.57	0.00	2.74	5.78	0.86	2.28	13.20
8	3	1250	28.75	34	7.71	1	17.53	-0.01	4.79	4.96	0.82	3.72	18.44
						2	13.37	-0.01	1.67	4.33	0.86	1.40	6.04
4	4	1580	28.75	22	2.63	1	13.00	-0.01	4.57	4.84	0.85	3.71	17.95
						2	12.36	-0.01	2.35	3.89	0.84	1.90	7.39
5	5	1250	13.75	64	10.95	1	16.77	-0.01	3.64	4.46	0.80	2.73	12.18
						2	14.02	-0.01	3.25	3.96	0.86	2.69	10.63
7	6	1580	13.75	37	7.92	1	12.56	-0.01	1.17	5.34	0.85	0.97	5.16
						2	20.53	-0.01	10.85	4.52	0.81	8.34	37.70
3	7	1250	28.75	37	3.54	1	17.66	-0.01	5.71	4.27	0.86	4.73	20.18
						2	16.57	0.00	2.67	3.20	0.77	1.93	6.18
2	8	1580	28.75	25	3.35	1	13.20	-0.01	7.66	4.90	0.75	5.40	26.44
						2	19.42	-0.01	4.85	5.02	0.83	3.81	19.12

The time is measured in seconds (column 4)

Table 4 presents the results from the ANOVA and regression models built on the results from the factorial experiment (see Eq.1 and Table 3). The results from the analysis are based on the full model fitted – this means that both factors and their interaction term are fitted. Each model is tested against an alpha value of 5%. The results show some interesting aspects as no significant changes were seen for the texture profile between the assessed factor levels whereas the weight loss and press juice showed significant effects. For the press juice, the magnitudes of the effects are all under or close to a 1% effect change between low and high levels (Table 4). The low magnitude of effect change compared to the intercept indicates only marginal improvement attainable by raising the electric field strength and the salt concentration in regards to lowering press juice. The weight loss is lowered when raising salt concentration and electric field strength. The findings show that the direction of lowering the amount of press juice and weight loss is in the same direction as the faster heating. Comparing the results of the press juice, weight loss and the TPA, the changes in water holding capacity of the shrimps seem not to affect the texture. It could be postulated that the shrimp to shrimp variation is bigger, than the possible effect of changing the parameters of the ohmic heating, with respect to TPA. This seems plausible considering the short time needed to reach 72°C which is the factor found by others (Niamnuy et al. 2007) as significantly impacting texture and that the findings reported in here are for shrimps heated for relatively short time periods for all experimental settings in comparison. The results from the present experiment show that all the shrimps have approximately the same texture profile. The results are comparable to the findings made by others for texture measurements of shrimp heated to a temperature of 75°C (Erdoğdu & Balaban, 2000).

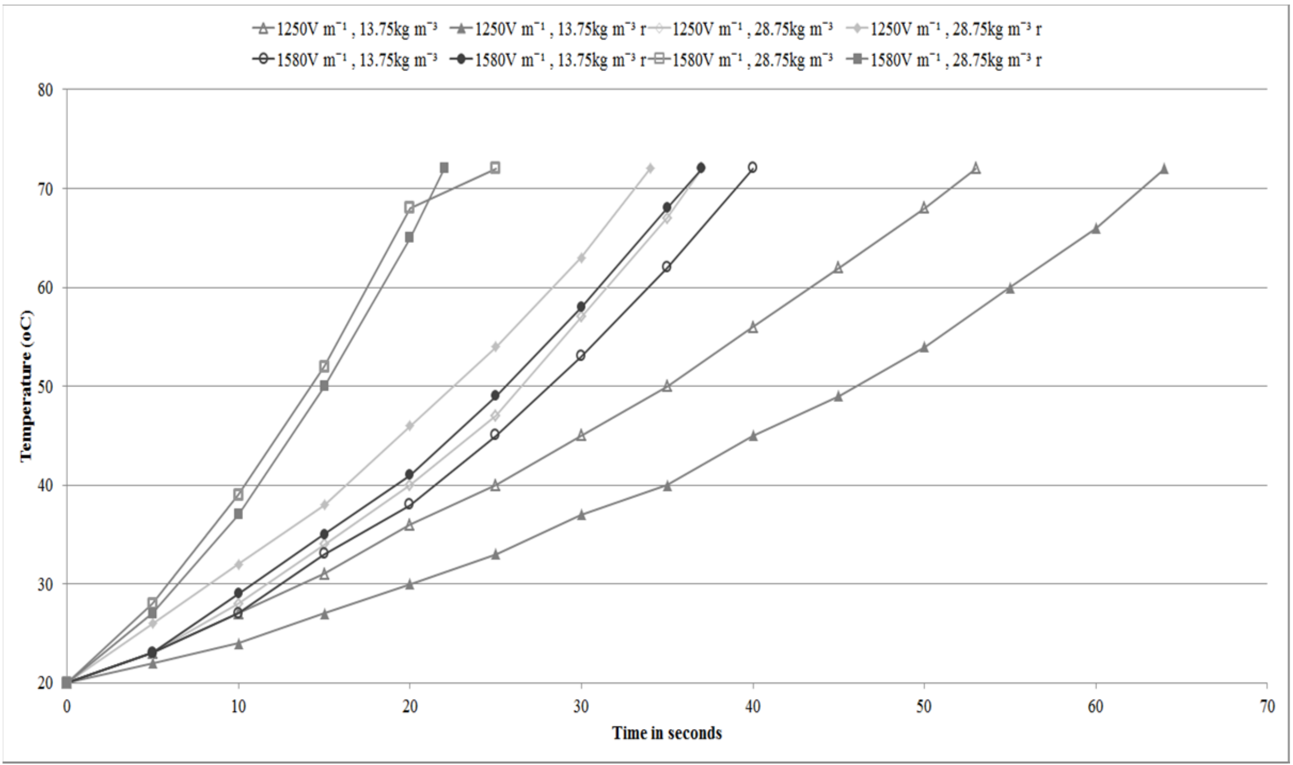

Figure 2 Measured temperature profiles of shrimps at varying salt concentration (13.75 kg m^{-3}, 28.75 kg m^{-}3) and electric field strength (1250 V m^{-1}, 1580 V m^{-1}). The experimental setup is shown in Table 3. The small r and a filled in icon indicates a replicate run.

The model tested for the heating time (Table 4) show that the salt concentration and electrical field strength effects were both significant and with no interaction term. Both factors have the effect of lowering the heating time when set at the higher level (28.75 kg m^{-3} and 1580 V m^{-1}, respectively). Figure 2 shows the measured temperature profiles from the experiments and Figure 3 shows the measured current.

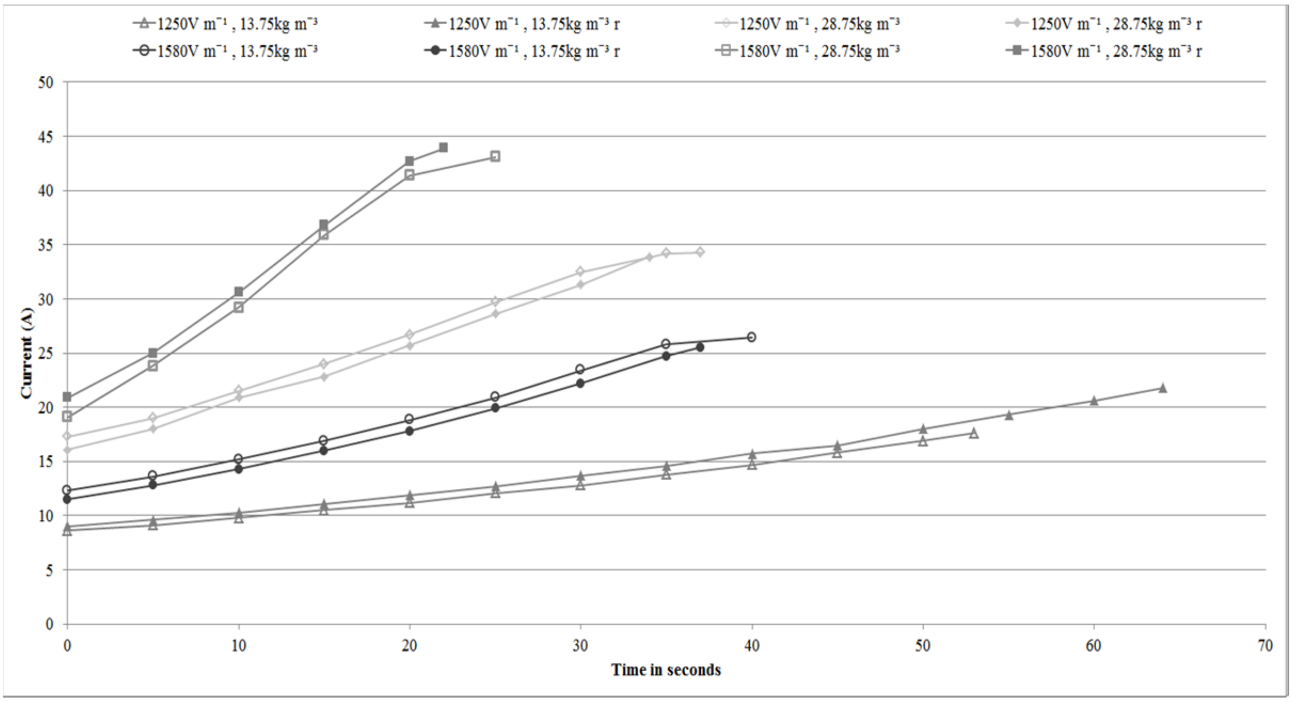

Figure 3 Measured current (A) profiles of shrimps and brine mixture at varying salt concentration (13.75 kg m^{-3}, 28.75 kg $m^{-}3$) and electric field strength (1250 V m^{-1}, 1580 V m^{-1}). The experimental setup is shown in Table 3. The small r and a filled in icon indicates a replicate run.

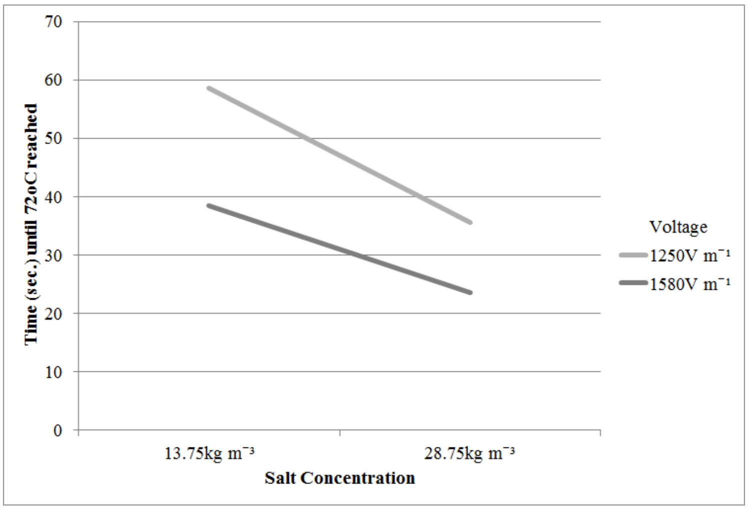

Figure 4 Interaction plot of the results from the factorial experiment of the heating times.

The Figure 4 shows an interaction plot which confirms the statistical result that no interaction effects are present for the time temperature relation tested. In Figure 2 this can be seen by the overlapping temperature profiles for experiments at 13.75 kg m^{-3} salt and 1580 V m^{-1} and 28.75 kg m^{-3} salt and 1250 V m^{-1}. The temperature measurements also point towards that raising electrical field strength and salt concentration lower differences between replicated runs. This can be seen in the difference between replicate temperature curves for the low setting of electric field strength and the salt concentration compared to the high settings (fig. 2). This was also observed in the study on the effect of shrimp size differences and spatial variation of the shrimp on the heating time (3.1). A simple main effects linear model seemed to explain the relation over the chosen design space on heating time which is shown in eq. 2 (with coded factors) and in eq. 3 (with transformed factors). All coefficients of the model are shown with the appropriate standard error in the parenthesis as shown below.

$$\begin{aligned} t_{sec.} = {} & \underset{(1.6)}{39} - \underset{(1.6)}{8}\ x_1 - \underset{(1.6)}{9.5}\ x_2 \end{aligned} \tag{2}$$

$$\begin{aligned} t_{sec.} = {} & \underset{(14.79)}{134.52} - \underset{(0.01)}{0.05} * E - \underset{(0.22)}{1.27} * C \end{aligned} \tag{3}$$

Table 4 results from ANOVA and regression on 2^2 designed experiment showing the effect estimates, model correlation and p value of the full model.

				Texture measurements					
Statistical results	Time to 72ºC	Weight loss (%)	Press juice (%)	Adhesiveness (N*mm)	Hardness (N)	Springiness (mm)	Cohesiveness	Gumminess (N)	Chewiness (N*mm)
Intercept (a_0)	39	7.154	15.38	-0.009	3.969	4.379	0.820	3.089	13.867
Salt (a_1)	-9.5(**)	-2.85(**)	-0.70(*)	0.002	0.316	0.046	0.004	0.234	1.350
Electric field strength (a_2)	-8(**)	-1.59(*)	-0.22	0.003	0.737	0.347	0.015	0.579	3.296
Interaction (a_3)	2	0.27	-0.98(*)	-0.003	-0.165	-0.111	-0.019	-0.198	-0.788
R2	0.94	0.88	0.87	0.44	0.36	0.56	0.38	0.42	0.48
P-value	0.0056	0.0223	0.0296	0.4572	0.5785	0.3041	0.5446	0.4952	0.4098

** indicates significant effect term, with * being 5% and ** 1%.*

3.3. Maturation effect

Table 5 shows the results from the impact of the maturation step on the heating time, and quality (press juice, mass loss and TPA). The results from the three treatments chosen were tested in a one-way ANOVA and no significant differences were found on the outcome except for the weight loss (Table 6). The later (weight loss) was expected because of the inclusion of the experimental setting using tap water where some osmosis would be expected to occur over the 24 hour time span. The results showed that no differences were found between the samples that were thawed immediately before ohmic heating and the samples that matured in brine. In Figure 5 the temperature measurements of the shrimps are shown. The heating times recorded until a temperature of 72^{o}C is reached are within the range observed for the other experiments. Figure 6 shows the measured current as a function of time. The shrimp matured in water and without maturation indicate different trends towards the end of the cooking compared to the shrimps matured in the salt solution. The shrimps matured in a brine show the same trend in rising current for the shrimp brine mixture as seen in Figure 3. However, for the water matured shrimps and those without maturation, the measured current increase at the beginning of OH heating and towards the end it reaches the peak thereafter decreases slightly. No difference was observed for the press juice test or the TPA results. That there is a difference in weight loss and not in press juice between the different maturation methods could be that there is osmosis into the layer between shell and meat e.g. the sub cuticle layer especially for the shrimp matured in water. This could maybe also explain the tapering of the measured current for water matured shrimp in Figure 6.

Table 5 results from maturation experiments

						Texture measurements					
Experiment #	Maturation	Time to 72^{o}C degrees	Weight loss (%)	Sampled unit	Press Juice (%)	Adhesiveness (N*mm)	Hardness (N)	Springiness (mm)	Cohesiveness	Gumminess (N)	Chewiness (N*mm)
1	non	45	-0.27	1	16.56	-0.01	6.15	5.27	0.89	5.32	28.07
				2	15.92	0.00	1.81	3.60	0.92	1.64	5.92
2	non	37	-1.69	1	19.79	-0.01	1.76	4.09	0.81	1.34	5.48
				2	22.94	-0.01	5.00	4.95	0.85	4.10	20.32
3	water	52	8.98	1	14.44	0.00	2.73	0.00	0.88	2.32	--
				2	18.67	-0.02	3.03	4.31	0.82	2.33	10.06
4	water	45	5.27	1	16.76	-0.01	1.53	3.19	0.90	1.35	4.31
				2	19.57	0.00	2.70	4.55	0.85	2.22	10.08
5	21.25 kg m-3 brine	41	2.26	1	10.24	-0.01	2.48	2.49	1.15	2.96	7.35
				2	18.99	-0.01	3.59	4.33	0.81	2.75	11.92
6	21.25 kg m-3 brine	32	0.46	1	16.58	-0.01	3.94	4.10	0.79	2.92	11.98
				2	18.18	-0.03	3.23	4.13	0.77	2.33	9.64

Table 6 Results from ANOVA analysis on maturation experiment.

		P-value
	Time to 72 degrees	0.25
	Press Juice	0.58
	Weight	0.038(*)
Texture Measurements	Adhesiveness	0.35
	Hardness	0.16
	Springiness	0.29
	Cohesiveness	0.97
	Gumminess	0.15
	Chewiness	0.46

** indicates significant effect term, with * being 5% and ** 1%.*

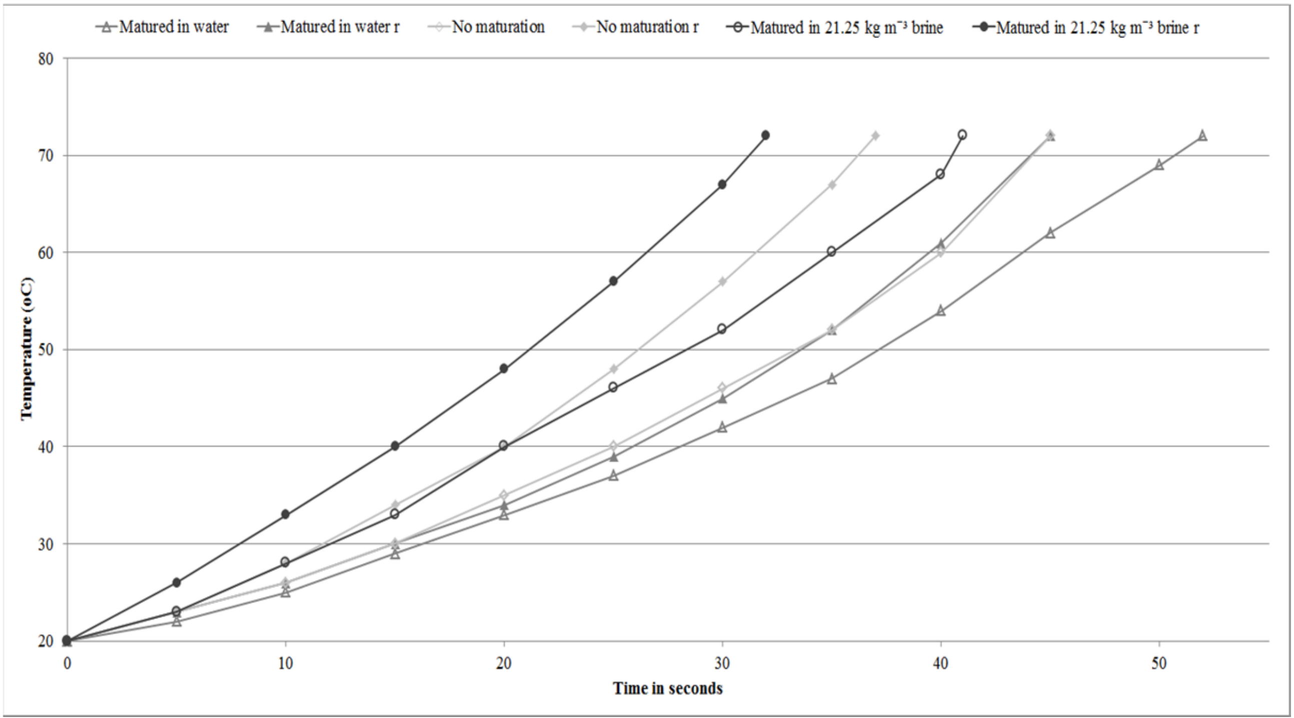

Figure 5 The measured temperature profiles of shrimps during the ohmic heating (electric field strength of 1417 V m^{-1} and salt concentration of 21.25 kg m^{-3}) with three pretreatment methods (maturation): maturation in water, maturation in 21.25 kg m^{-3} salt brine and with no maturation just thawing. The small r and filled-in icon indicates a replicate run.

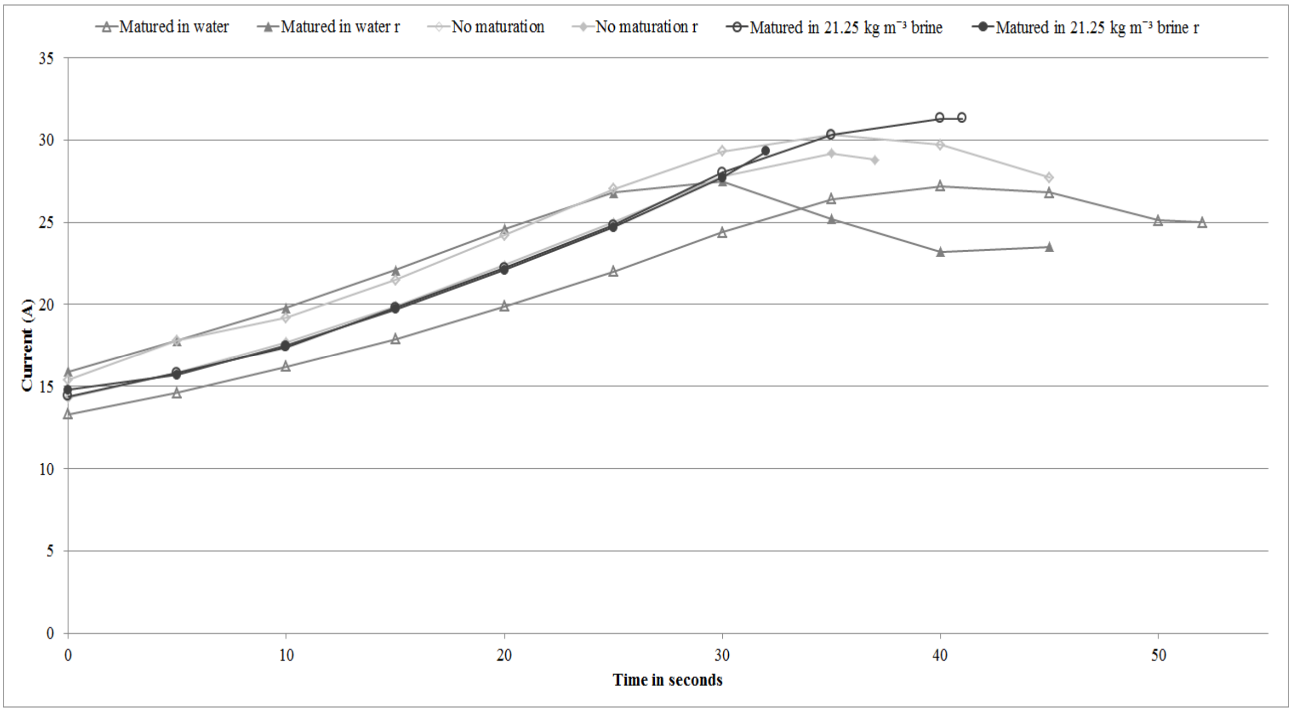

Figure 6 The measured current (A) of shrimp and brine mixture which have been applied a 1417 V m^{-1} electric field strength after one of three pretreatment methods (maturation): maturation in water, maturation in 21.25 kg m^{-3} salt brine and with no maturation just thawing. The small r and filled-in icon indicates a replicate run.

3.4. General discussion

The results from this study are the first to show ohmic heating for thermal processing of shrimps. The results seem to confirm instances of volumetric heating, and indicated a main effects linear relation between the process variables (electric field strength and salt concentration) and the heating time with no interaction between variables. The heating time decreases with increasing salt concentration and electric field strength level. Additionally, the results show that over the chosen experimental settings no significant difference occurred to product quality defined by the TPA. This has major implications for the design and implementation of ohmic heating industrially, which could imply a faster implementation of the technology if the relations persist when up-scaling the process. Further studies could be made on if or how the processing with ohmic heating influences the naturally batch-to-batch variation raw material quality.

The results show a rise in difference between replicate cooking times when electric field strength and salt concentration is lowered as seen in the paired t-test (section 3.1) and the factorial experiment (section 3.2). This would be relevant investigation point screening whether it was due to differences in electrical conductivity inducing shadows or whether it is a result from shrimp variations. The results are although opposite of the findings by Salengke & Sastry (2007), where smaller temperature differences within the ohmic heater was shown when using lower electric field strength. This was reported for the situation when the solid had lower electric conductivity than the liquid medium. The difference seen in the results from those reported for shrimp was that higher electric field strength gave lower differences between replicated temperature profiles, are hard to discern. An important distinction between the experiments performed by Salengke & Sastry (2007) is that the temperature differences is within the ohmic heater and the experiments reported here the temperature differences are between experimental runs. This could cause a confounding by the mentioned shrimp to shrimp biological variation. It could also point towards that in between the range of tested electric field strengths and salt concentrations reported in this paper and those used by Salengke & Sastry (2007) an unknown transitional phenomenon takes place for the physics concerning electric conduction.

For the results of the weight loss and press juice measurements, when assessing both the 2 level factorial experiments and the maturation experiment together, show interesting relations. Over the span of salt concentrations, the combined results seem to indicate an optimum i.e. lower weight loss seen for the brine matured shrimp in the maturation experiment. The reason could be the relationship between the ionic strength, voltage and the solubility of the proteins in shrimp over the tested experimental range has been at levels approximately at when both salting-in effects (at lowest salt concentration of 13.75 kg m^{-3}) and salting-out (at lowest salt concentration of 28.75 kg m^{-3}) effects are predominant (tables 3 and 5). The electric field strength and salt concentration used in the maturation experiment is an approximate center point setting when compared to the 2 level factorial experiments. Similar findings are also seen when boiling shrimps (Niamnuy et al. 2007; Niamnuy et al. 2008).

It should be noted that a tentative assessment of the ease of peeling the shrimps was performed for the experimental trails. The results are not given here since the scale used was not calibrated to validate the results. However, the tentative findings did point towards an easier peeling of shrimps when higher electric field strength and salt concentration were used and also that the maturation, be it either in brine or water, had a positive effect compared to no maturation on the ease of peeling.

The results showed that using a set temperature gave a robust operating criterion with respect to impact on the quality which was invariant over the chosen process settings. Do to limitations in the experimental setup the application of a holding cell was not possible. This would have allowed for a more extensive study into thermal inactivation and a proper safety assessment of the technology. The set point temperature used is an industrial specified for existing conventional processing.

Future studies are ongoing concerning the possible utilization of ohmic heating in improving the efficiency in de-shelling (peeling) operations. It would have been pertinent also to address the question of the impact of water to shrimp ratio. Some preliminary work has been done but the experimental design is not straight forward to resolve it. It would be interesting to try other species of commercially sold shrimps in order to assess whether the findings are similar.

4. Conclusion

The findings exhibited a linear relationship between the heating time (the time to reach 72°C) and process variables (electric field strength and the salt concentration) over the experimental range tested. The press juice was also influenced by the process variables for the ohmic heating step but not the maturation method. The variation in shrimp size had no impact on the heating time. For the temperature history within the shrimp a difference was observed at lower levels of salt and electric field strength. The difference was that the head was heated faster than the tail part of shrimp. The results showed a comparable texture of the shrimp to that of conventionally cooked shrimp reported in other studies. No impact was observed on the quality measures from the texture profile due to the processing conditions. The findings show a promising application and future possibilities in utilizing ohmic heating for thermal processing of shrimp.

Acknowledgement

This work was performed within the research platform inSPIRe and within the research project - innovation consortium project *Optimized heating and cooling of meat, seafood and cheese products*. The financing of the work by The Danish Agency for Science, Technology and Innovation and all collaborators in this project are gratefully acknowledged. This work was performed in collaboration with Royal Greenland A/S and BCH ltd.

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Example 1: Discussion

The structure of the experiments offers various discussion points in the application of designed experiments. The first experiments with the paired temperature measurements of small vs large shrimps and tail vs head part clarified cold spot considerations and size dependence. The cold spot considerations are important for the safety aspect of the product quality. The size dependence would verify if the specific application was volumetrically heated. The way in which it was tested was by using the example of a boy's shoe experiment originally given in Box et al. (2005). A company wanted to test a new sole material against the older version by making pairs of shoes of both materials, and letting several boys wear down the shoes (Box et al. pp. 81, 2005). Instead of having two types of material for shoes it was small vs large shrimp and tail vs head that was tested in the shrimp case. As discussed by Box et al. (pp. 81, 2005) in the boys shoe example one way of conducting the experiment could have been to have one boy test out several pairs. The boy would generate a series of observations from which to estimate uncertainties and test against. The caveat is the results from the "one-boy–experiment" would be a weaker inference with respect to extrapolating the results to other users. By using several boys the results of the paired testing has a firmer basis from which the company could trust to represent the user segment and their usage. The same argument holds for the shrimp experiment, where the instead of having a population of boys to sample from, the experiment has a population of process setting combinations to sample from. Using several levels of the salt concentration and field strength, the basis for extrapolating the conclusions drawn from the results have a wider applicability or formulated differently the experiment has as Fisher called a "wider inductive base" Box et al. (pp.82, 2005).

In designing the screening experiment for the effect of process settings using the 2 level factorial design simplicity in execution was the aim. Two factors salt concentration and electric field strength were chosen, although other factors were also discussed for inclusion in the experiment. One factor which was discussed was the ratio of brine to shrimp within the container of the ohmic heating unit. The setup used had a fixed ratio of brine to shrimp but when upscaling the application for pilot scale or industrial scale this ratio may need to be revised. The implications of trying to include the ratio as an experimental factor quickly raised many concerns in the ease of executing the experiment. The main reason for the concern was the issue of how to achieve the different ratios of brine to shrimp. The shrimps are individuals with a certain weight. This could mean that half or a quarter of a shrimp was needed in order to achieve the required ratio. This would result in a different type of shrimp meat exposure and unwanted diffusion from the meat into the brine. The aim of this experiment was to screen the effect of different process settings on several quality attributes of the cooked shell on shrimp. The choice was to have two process variables reasonably asserted to be the most important and to include more responses relating to quality. There were no formulated expectations concerning the findings of the experiment. The fact that it was a novel application and that it was unknown whether there would be time and material for follow up experimentation meant that a simple replicated design was most appealing.

The final experiment concerning the pretreatment was formulated after analyzing the results from the screening experiment. The results from the screening experiment indicated that the technology seemed promising. It also raised other questions pertaining to the actual implementation of the unit operation in an industrial production line. The pretreatment step (maturation) used in the experimental procedure mimicked the company's standard procedure. The questions were then how did the step influence the ohmic processing and if the step was needed at all. This meant that an extra experiment testing shrimps with and without a pretreatment was conducted. The tap water pretreatment step was made in order to contrast the salt influence in the pretreatment on shrimp. This could also indicate whether it was salt in the pretreatment or in the brine for ohmic heating which was important for the ohmic heating process. The choice of the level settings of the ohmic heating process was to center them with respect to the previous factorial experiment. This would give a second set of observations executed using the same procedure as for the screening experiment but with level settings in the middle of the experimental region. The choice was based on a "common sense" approach. The argument was that since the focus was on the pretreatment step then centering the settings of the ohmic process settings was a reasonable choice. The results of the experiment then gave valuable findings in and of themselves with respect to understanding the pretreatment step. The other interesting finding was from the observations of the center point treated shrimps with respect to the weight loss of the shrimps compared with the results from the screening experiment. In Fig. 3.1 weight loss is plotted against the salt concentration. The interesting findings indicate that over the experimental region, the phenomena of salting-in and salting-out could possibly occur at the respective concentrations.

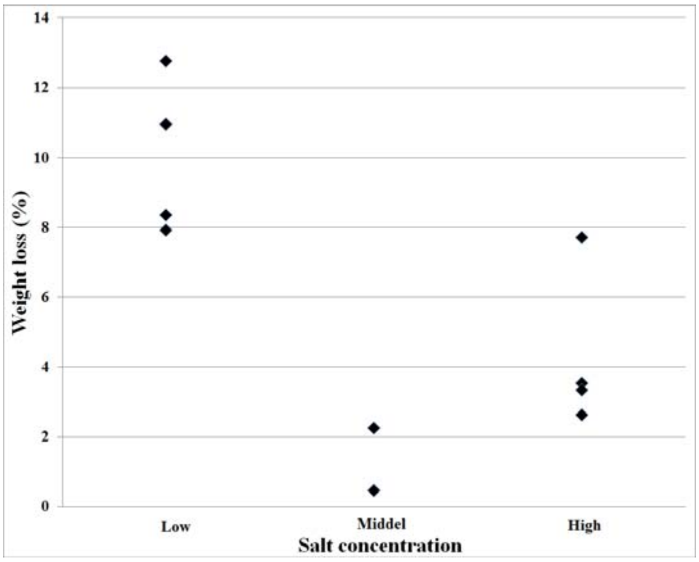

Figure 3.1 weight loss measurements of shrimp cooked with ohmic heating.

The findings although speculative are very interesting, and could have major potential implications for the use of ohmic technology. These findings warrant continued experimentation on the relationship between process settings and weight loss. The shrimp examples show how to combine small consecutive experiments in order to frame a greater understanding.

Example 2: Factor screening on pilot scale impingement oven (Report I)

This example is of a fractional factorial design used for screening several factors simultaneously. The fractional factorial design uses as the name implies a smaller fraction of the complementary full factorial version (Box & Hunter 1961). The reason for using fractional designs is to screen a large number of factors specifically the main effects and in some instances second order interactions in as few experimental runs as possible. The cost payed by having fewer experimental runs means having fewer observations which also means a loss in degrees of freedom and confounding effects with high order interactions. The situation in question concerns a screening experiment performed on a pilot scale impingement oven with the process conditions measured by heat flux and heat transfer coefficient as the responses. As mentioned in the general introduction the motivation for the experiment was driven by the collaborative work with the company that supplied the oven. Two major questions were posed, firstly the already mentioned screening of factors influencing the heat transport responses. Secondly, an overall assessment of the ability of the oven for use as a pilot scale experimental platform. The final design of the experiments was made almost during the actual execution of the experiments. This meant little time for in depth considerations on which designs to use. This was mitigated by the thought process on different experimental situations that could occur up until the day of experimentation. The choices were focused on simplicity and immediacy. The reason for emphasizing these points was that one of the factors, height of inlet air ducts, required dismounting of the oven door, which was both laborious and posed a safety concern. This situation with a hard-to-change factor would have posed a natural situation for the application of a split-plot experiment (Kowalski et al. 2007).The considerations on these mentioned points; immediacy, simplicity and restrictions, are further discussed in the examples main text and discussion.

Case presentation: Screening experimentation on a pilot scale oven for factor identification
Short title: Screening experimentation on a pilot scale oven

Søren Juhl Pedersen, DTU Food, Murat Kulahci, DTU Compute, Stina Frosch, DTU Food

Abstract

We present an experimental study performed on a new pilot scale oven specifically built for elucidating the mechanisms in industrial baking of food by convection heating. The work was initiated by the manufacturer of the oven with which we were involved in a collaborative project concerning optimization of baking processes and equipment. The experiment in this study was motivated by two questions; one concerning a specific design variable's influence on baking conditions and the second question concerning the evaluation of the efficacy of the current setup's ability for experimentation. The case depicts an intricate relationship between the subject matter knowledge and statistical thinking in engineering research. The specifics concerning the design of the experiments, the constraints involved and how the experiments were performed are presented. The results unexpectedly revealed a significant interaction effect and raised important questions for further experimentation for the optimization of convective baking processes. One of the key learning experiences from the experiment was the valuable insight on process improvement gained by introducing statistical thinking and also using engineering knowledge. For example, the systematic data collection scheme through designed experiments generated the insight of equipment related issues that would not have been detected otherwise.

Keywords

Statistical thinking, Process Knowledge, Fractional factorial design, Foldover Design

1 Introduction

This case study is an example of the combination of subject matter knowledge and basic design of experiments principles applied to a problem of process development and improvement in food engineering. The focus is on introducing the proper statistical thinking in the choices that have to be made during experimentation. The concerns were how to reset the experimental conditions between runs and to comply with the constrained timeframe. Should the experiment be performed using a split-plot structure or complete randomization? What is the most feasible way of resetting the experimental conditions after each run? These concerns are central for experimentation of industrial processes especially in continuous processes (Vanhatalo et al. 2010).

It is our observation that the research in the field of design of experiments has lately been overwhelmingly about the mathematical aspects of designing experiments based on some objective functions and the subsequent analysis of the derived designs. While we can see some merit in those efforts, the reality often requires us to think beyond the textbook examples depicting an "ideal" setup. This usually takes shape during the execution of the experiments. We believe that the duty of the statisticians is not limited to designing and analyzing experiments. In fact statisticians should face the reality about the difficulties of actually running the experiments and make the utmost effort to be more involved in the execution. Would you like to learn how to drive from somebody who knows all the traffic rules and the operating manuals of any car by heart and yet has never driven a motor vehicle in their life? Admittedly reality is challenging which can be a deterrent to some. However in an applied field such as design of experiments, this is a fact of life and there is no escaping it. The case we present in this paper is about an experimental study plagued with various challenges particularly during the execution of the experiments, the ad hoc decisions that needed to be taken due to resource and time limitations, and making utmost use of engineering knowledge in the analysis of the experiments in order to make the most sensible conclusions.

There have been some scientific papers that documented the actual progression of experimental work and intermediate analysis. As a great example, (Bisgaard 1989) discusses the difficulties in a real case study and the iterative process of reaching meaningful conclusions. Bisgaard's case study contains interesting details typical to many industrial problems today. One example worth mentioning is the starting description of how the company tried to identify and solve their problem with the use of excessive monitoring, ending up with a large collection of data. Bisgaard reports briefly looking over these data, and recommending rethinking the issue by cutting down the number of responses and performing designed experiments. The description of the case shows how the back and forth iterative process evolved, changing parameters without getting further clues and the fast assessments that had to be made with a tight deadline. It shows how valuable indications can be even though there were no statistically significant results. In this paper we discuss the pre-determined protocol of running experiments compared to what actually happens due to unforeseen restrictions materialized during the execution of the experiments. The paper is structured by first giving background for the study (in section 2), also in order to describe some of the theoretical considerations from an engineering point of view. In section 3 the execution of the experiments is described and the preliminary analysis is provided. Section 4 describes some of the concerns and corrections made on the measurements. Section 5 and 6 contain the final results and a short summary.

1.1 Background

The specific problem was part of an industrial research project which concerns the tools used in food engineering for process optimization, prediction and scheduling. It is structured around several parallel small projects each working on different aspects of food engineering in cooperation with private companies from food producers to process equipment suppliers. The company in this case is a supplier of continuous ovens and supporting equipment for baking industry, and heavily involved in the product development. The company was interested in two things; firstly a further evaluation of a pilot scale version of an industrial convection oven and secondly a specific design question concerning the impact of height between air ducts and baking sheet on the heat flux. How to tackle these questions and what response (or responses) appears to be relevant is the main focus of this case study. The company's interest was centered on measured heat flux as the response. However we suggested that they consider the heat transfer coefficient, which is a measure invariant to temperature and is a commonly used measure of process characteristics used in food engineering. Both responses provide knowledge about processing conditions and are used for predictive calculations of heating profiles and production- or equipment scaling.

1.2 Oven process theory

The baking oven is one of several staple process operations in food engineering. This paper deals with convection oven design and more specifically with impingement ovens. The impingement is due to the design by which the airflow is above a certain threshold in speed and via the air feed system is forced through inlet holes creating a cone profile that impinges on the baking sheet. The full scale process is a continuous operation whereby the time and throughput of the product require higher transference of energy in order to achieve the specified baking profile of the product. The impinging of air is done with the aim of raising the transference of heat from the air to the product. In engineering this is quantified with terms such as heat flux and the heat transfer coefficient specifically for heating with a fluid which in this case is the air. The heat transfer coefficient gives a measure for how much energy can be moved between a fluid (air) and solid (product) and is the measure of proportionality in a given situation between the heat flux and the temperature difference. The main relation can be seen in a simplified version of the equation for Newton's law of cooling:

$q = h * A * \Delta T$ eq. 1

where q is the energy which when divided by the area, A gives the heat flux, h is the heat transfer coefficient and the ΔT is the temperature difference between air and product (eq. 1). An important issue to mention is that the impingement oven delivers turbulent flow of air and nearly every specific situation in regards to the flow needs specified empirical relations with experimentally determined coefficients. Approximation of flow pattern and the expected heat transfer coefficients can be obtained through computational fluid dynamics (CFD) modeling. However, this still needs to be validated experimentally. So experimentation is crucial in providing estimates of the parameters for modeling and design construction, and also for validation of the models. This was the main motivation for constructing a pilot scale version of an industrial oven whereby different design configurations could be setup, measured and used for formulating models giving an approximate description of the process (Andresen et al. 2013).

1.3 The pilot scale oven

The pilot oven used was designed to mimic an industrial continuous oven (Andresen et al. 2013). In order to achieve the resemblance of the movement inside the industrial counterpart, the pilot oven has been specially built with frames housing the air ducts whereby the whole frame moves from side to side. The air ducts are punched or drilled out from a frame, which can be replaced with new frames with different designs.

The oven was constructed to allow some design parameters to be easily changed. The baking sheets can be changed from chain link type to for example solid sheet metal baking sheets where the choice is mainly dependent on the type of product to be baked. The height between air ducts and the sheet can be changed independently for the bottom and top set hanging on the frame. The amount of air from either the top or the bottom can be independently set via butterfly valves to give the option of letting more air hit the sheet from either underneath or the top. The speed by which the frame moves from side to side can also be regulated. There is an option of regulating a fresh air intake valve which is of importance mainly in baking situations with high humidity for example in baking sponge cakes. Then lastly the temperature of the heater and the frequency of the fan can be changed in order to change baking temperature and air speed. The variables chosen for this experiment and there settings are given in Table 1. It was expected that the fresh air intake and speed of the frame movement would not impact the response in any significant way. Nonetheless they were included for the evaluation of all the possible oven configurations in order to meet the experiments objectives.

Table 1 description of variables, name, possible range and the settings used

Factors	Code	Range	Low-high
Distance /height in cm	A	5-20	11-15 cm
Temperature	B	0-250	180-220^{o}C
Air flow (frequency of the fan)	C	Turn nub from 0 to 10	5 – 10
Fresh air intake fraction (the setting of the exhaust valve)	D	0-100%	10%-40%
Fractioning of air flow between top (T) and bottom(B) inlets (setting of the valves)	E	T:100 B:0 T:0 B:100	T:50%- 80% B:50%- 20%
Speed of band (frequency of sideways motion of the air ducts - frame)	F	0-50 m/s	5 – 25 m/s

2 The execution and preliminary analysis

The amount of time allocated for experimentation was originally spanning 4 days; 2 full days followed by a ½ day and finally a full day again to resolve possible ambiguities. However there were issues in borrowing and the delivery of the special measuring equipment for the heat flux which delayed the start of the experimentation and reduced the experimentation time to 2½ days. The feasible number of experiments that could be performed during a day was also difficult to predict. A trial was performed before the actual experiment where it was found that a tentative guess of 2 hours per experimental run was feasible which also included the resetting of the system before the next setup. During the trial it was found that changing the height between air ducts and baking sheet was not without difficulties as the oven door had to be dismounted. This was a hazardous maneuver requiring a harness due to the considerable weight of the door. This sparked a discussion of putting restrictions on randomization. An idea was to run the experimentation in a split plot structure with the height variable as the whole plot factor. This was dismissed due to several concerns. The estimated maximum number of experiments in a day was between 8 to 12 runs and using a split plot structure would create difficulties in the design. Hypothetically this would entail running a 2^{5-2} on the sub plot level and combined with the whole plot would give a 16 run experiment. The whole plot variable would only change once during the day which raised the concern of possibly confounding the variable with environmental changes between noon and evening. A third consideration then fell on the time it would take to perform an experimental run and then reset the system. Dismounting the door allowed a bigger amount of fresh air to flush the system which would give a faster cooling and shorten the time between runs.

The conclusion concerning which design to use was made on the day of experimentation at the beginning of which there was a further delay in the arrival of the measurement equipment. The decision was to use a fractional design with a maximum of 8 runs. A premade design was chosen hastily, without much consideration of towards the aliasing. The screening design used was a 2^{6-3} design (e-Handbook of Statistical Methods, 2014) given in actual run order in Table 2 with the measured heat flux in the last column. The defining relation of the design is I = ABD = ACE = BCF = DEF = BCDE = ACDF = ABEF.

Table 2 The screening 2^{6-3} design for first day of experimentation.

	Factors						Average
Run	A	B	C	D	E	F	Y (q) kW/m^2
def	-1	-1	-1	+1	+1	+1	8.64
af	+1	-1	-1	-1	-1	+1	7.83
be	-1	+1	-1	-1	+1	-1	8.34
abd	+1	+1	-1	+1	-1	-1	10.44
cd	-1	-1	+1	+1	-1	-1	9.51
ace	+1	-1	+1	-1	+1	-1	10.73
bcf	-1	+1	+1	-1	-1	+1	10.08
abcdef	+1	+1	+1	+1	+1	+1	8.06

Two center runs were also performed during the first day which became possible due to faster tempo that was achieved from repeating the execution of the experimental steps. Also the choice of running fully randomized runs and thus allowing for the flushing of cool air between runs reduced the time from 2 hours to 1½ hours for each run.

The preliminary analysis based on ANOVA was performed. It was noted during the analysis that the hasty choice of the screening design led to an unintended and undesirable aliasing among interesting effects and two factor interactions. For example the height variable was confounded with the interaction between airflow and fractioning of airflow. One saving grace was that the two factor interactions BE, CD and AF were confounded and these interactions were expected not to be significant and hence to give a measure of the experimental error. The analysis was ad hoc and performed using a basic spreadsheet program. This was also done in order to supply the company representative with a data sheet wherein he could see some of the calculation steps for the ANOVA and graphical output. The results are summarized in Table 3 of measured heat flux.

Table 3 The analysis of the 2^{6-3} design in table 2 of heat flux			
Factor	Contrast	effect	SS
Intercept	9.20		
A	0.49	0.12	0.03
B	0.19	0.05	0.00
C	3.12	0.78	1.22
D	-0.32	-0.08	0.01
E	-2.10	-0.52	0.55
F	-4.41	-1.10	2.43
BE=CD=AF	-6.15	-1.54	4.73

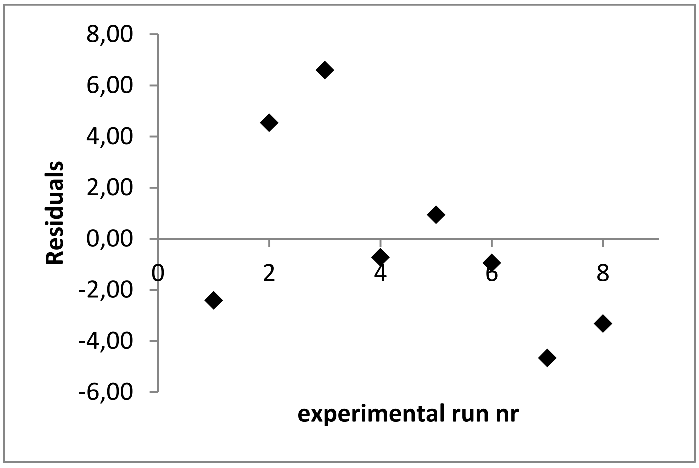

Figure 3 Residuals against run order from first day of experimentation.

From Table 3 it can be seen that no factors really showed any “signal” against the BE=CD=AF factor which was expected as a measure of noise. There was no theoretical explanations for why the BE=CD=AF factor should be seriously considered. The results show a general problem of high experimental noise. It could have been an idea to perform the experiment with the height variable as the whole plot parameter. The concern from an engineering point of view was then that the door dismounting “effect” and the within day duration would confound the effect of changing the height. It was deemed more important to resolve the confounding present and then wait for later experiments to address the experimental error. The plot of residuals against run order (Fig. 1) was troubling due to the pairwise observed tendency and what seems like a trend from run nr 2 until the end of experimentation. The trend could be the ambient temperature impacting seen by the fact that experiment nr 1 was performed around noon and the last experiment close to midnight. This also posed a problem of how to block not only for the days but also for shift from morning to evening.

The day shift was not resolved since it would demand three blocks; one for the Friday and two for the Sunday experimentations. Two factors had influence on how to proceed. The first was the time constraint which also impaired the analysis coupled with the tools available e.g. reference books and spreadsheet programs. The second consideration concerned the experience gained by working with the pilot oven. During execution several observations were made on the performance of the oven regarding leaks in the system, safety caps coming undone and the frame for moving the air ducts having trouble with the wheels going askew and wire setting mechanism that shift in their setting. All these have direct relations to BE=CD=AF effects and explains the high SS value (and hence high noise) associated with this assumingly negligible alias chain. During the day for the midway analysis there was also performed some additional experiments; two center runs were performed and additionally replication of 2 of the design points. This was not performed in random order but in a structured way with first a replication of a design point and then center etc. Replications of the design points were meant for warming up the machine and the environment to give some credibility to possibly using the center runs for computing error term or possible testing for curvature. In Table 4 can be seen the measurements for the center points measurements of day 1 and 2 which are not significantly different from each other.

Table 4 center runs performed on the first and second day of experimentation

Day	Center run #	Y (q) kW/m^2
1	1	9.67
1	2	9.61
2	3	9.68
2	4	9.34

The assessment based of the sums of squares showed high experimental error even though the center points between days show the opposite. It should also be mentioned that the preliminary analysis was based on the use of the heat flux as a response. The heat transfer coefficient estimates given from the equipment gave nonsensical values, and unfortunately could first be resolved after the experimentation was done. This will be further elaborated in the section concerning the measurements system. The decision was to fold-over the whole design. It was deemed impossible to sensibly address the experimental error any further. The choice was to at least use the last day of experimentation to resolve the main effects for the eventual analysis of the heat transfer coefficient. Essentially the argument is based on, let’s-at-least-try-anyways, thinking. A lot of experimentation is also based on unforeseen observations. The combined designs, i.e. the original design together with its full fold-over is of resolution IV with I=BCDE=ABEF=ACDF as the defining relation. This allowed for clear estimation of main effects assuming that three factor interactions are negligible but also the potentially significant two factor interactions are confounded with other interactions that are assumed inert based on process knowledge.

2.1 Measurement System

The measurements were made with the special multisensory equipment, Scorpion 2 (Markel Food Group, USA), with the ability to measure; airflow, temperature, convective and radiative heat flux. The equipment was packed in an insulated box and needed to be cooled after each experiment both to protect the electronics and to cool the reference temperature loggers inside the equipment. The measuring equipment is placed inside the oven for 10 minutes. Only the last 5 minutes of the recorded data was used, to exclude the impact on the system from the placement of the measuring equipment. This can be seen in Fig. 2 by the example of recording where the first couple of minutes show a transition to steady state. The equipment is supplied with special software for extracting and plotting the recorded data. The responses of interest for this study were the total heat flux and the convective heat transfer coefficient. The responses are derived values based on calculations made on special temperature probes and reference temperatures. The special probes make it possible to quantify the amount of energy supplied via radiation and convection separately which it is of interest for process design. It was found during the experimentation that the equipment gave faulty estimates for the heat transfer coefficient by a roughly a factor 3 bigger values. This did not fit with what could theoretically be expected and had been observed in previous experiments using similar measuring principles. The adjustments were made for all measurements by using a different reference temperature recorded in the equipment. This gave reasonable estimates but some concern should be raised as to the validity due to the fact that the precise setup inside the equipment is unknown. The problem could also stem from the choice of reference temperature and thus reference system which can differ between engineering sciences. Finally the scale of the measurements with the heat flux measured in kW was recorded with two significant digits and the heat transfer coefficient in W. So, this posed an issue of differences in scale. The precision of the corrected heat transfer coefficient should be most dependent on the temperature measurements, but no uncertainty analysis has been performed, again due to the unknown setup inside the measurement equipment.

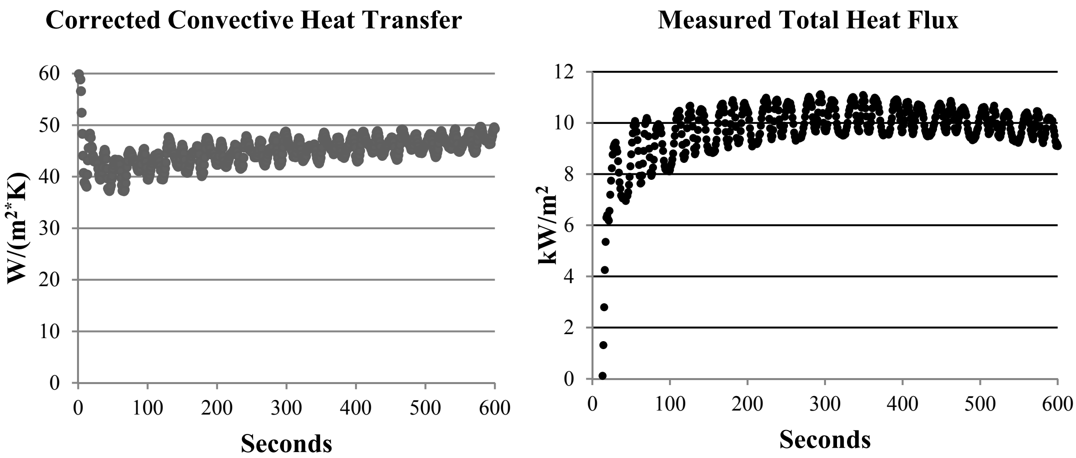

Figure 2 Shown on the right is an example of the calculated heat transfer coefficient based on the measurements shown in the plot on the left.

An assumption concerning the system was that the heat transfer magnitude would be homogenous across the baking sheet surface. This means that the possible gradient would be relatively low between different locations on the baking sheet. Therefore the exact location of the measurement device was irrelevant. However the outcome of the experimentation changed this view and in the future it is recommended to consider obtaining the measurements at the same location on the baking sheet. Optimally some consideration should be made as to what constitutes a significant difference in heat transfer coefficient between location which will be very much product dependent. This could also motivate a new study into determining how factors influence the observed gradient in the measured heat transfer coefficient between locations. Also the response which was the average over the sampled time span should be reassessed. The response does not capture the oscillatory trend. Again this also falls back to the question of whether the oscillatory trend is important in relation to baking.

3 Results

The analysis of the full design (both fractions combined Table 2 and 5) is given in Table 6 with the center points and Table 7 without. The results show that as expectedly the heat flux is influenced by changing the temperature, which means that it is not an appropriate response to consider for evaluating design configurations impact on the process. Two sets of analysis were performed both with and without the center points. The argument being that the center points one set of the center points were made on a day 2 and the fold over was on day 3. The analysis made is very much ad hoc. The results shown are for the condensed design where some of the interaction terms have iteratively been dropped. The results show that many of the terms are insignificant. The terms chosen to display in the tables of results for all cases are the main effects and the interaction effect of most interest from an engineering view point (Table 6 – 9).

Table 5 The fold over design performed on the second day of experimentation

	Factors						Average
Run	A	B	C	D	E	F	Y (q) kW/m^2
abc	+1	+1	+1	-1	-1	-1	10.79
bcde	-1	+1	+1	+1	+1	-1	11.13
acef	+1	-1	+1	+1	-1	+1	7.97
cef	-1	-1	+1	-1	+1	+1	8.11
abef	+1	+1	-1	-1	+1	+1	9.98
bdf	-1	+1	-1	+1	-1	+1	9.83
ade	+1	-1	-1	+1	+1	-1	8.19
(1)	-1	-1	-1	-1	-1	-1	7.00

Table 6 ANOVA for the heat flux effects with center points included.					
Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*
Block	0.02	1			
Height	0.03	1	0.03	0.14	0.98
Temp	21.01	1	21.01	120.02	0.07
Airspeed	0.91	1	0.91	5.19	0.33
Fresh %	0.01	1	0.01	0.06	1.00
T-B fraction	0.14	1	0.14	0.80	0.72
Bandspeed	0.42	1	0.42	2.39	0.47
Error (residual)	2.28	13	0.18		
Lack of fit	2.20	8	0.27		
Pure error	0.08	5			
Total	24.81	20			

Table 7 ANOVA for the heat flux measurements					
Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*
Block	0.02	1			
Height	0.03	1	0.03	0.09	0.99
Temp	21.01	1	21.01	76.55	0.00
Airspeed	0.91	1	0.91	3.31	0.11
Fresh %	0.01	1	0.01	0.04	0.85
T-B fraction	0.14	1	0.14	0.51	0.50
Bandspeed	0.42	1	0.42	1.52	0.25
Error (residual)	2.20	8	0.27		
Total	24.73	15			

Table 8 and 9 shows the results from the analysis performed on the corrected measured heat transfer coefficient with and without the center points respectively.

Table 8 ANOVA for the corrected heat transfer coefficient with center points.					
Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*
Block	48.62	1			
Height	0.81	1	0.81	0.07	0.80
Temp	14.95	1	14.95	1.29	0.29
Airspeed	45.39	1	45.39	3.91	0.09
Fresh %	8.80	1	8.80	0.76	0.41
Bandspeed	4.28	1	4.28	0.37	0.56
Height * Airspeed	16.81	1	16.81	1.45	0.27
Bandspeed * Airspeed	9.85	1	9.85	0.85	0.39
Error (residual)	139.33	12	1.,61		
Lack of fit	13.97	7			
pure error	125.36	5			
Total	288.84	20			

Table 9 ANOVA for the corrected heat transfer					
Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*
Block	48.62	1			
Height	0.81	1	0.81	0.41	0.54
Temp	14.95	1	14.95	7.49	0.03
Airspeed	45.39	1	45.39	22.74	0.00
Fresh %	8.80	1	8.80	4.41	0.07
Bandspeed	4.28	1	4.28	2.14	0.19
Height * Airspeed	16.81	1	16.81	8.42	0.02
Bandspeed * Airspeed	9.85	1	9.85	4.94	0.06
Error (residual)	13.97	7	2.00		
Total	163.49	15			

The conclusions are drawn from the analysis from the ANOVA model without center points. The question seemed open-ended whether or not it was valid to include the center points in the same analysis when we also block for a different set of days between the two fractions. The model includes the main effects and the most interesting two factor interactions with respect to the objective of assessing the influence of the height between the air ducts and baking sheet. The effect estimates are although relatively small compared to the intercept when looking at the heat transfer coefficient. This implies from an engineering standpoint that there are marginal impacts from changing the settings over the experimental region studied, which can be seen by the regression model coefficients shown in Table 10. From a first glance it would seem like the changes induced on the heat transfer coefficient over different processing conditions are too low to have an impact of processing of products. The interesting observation was the interaction effect observed between the height of inlet holes above the baking sheet and the airspeed. An interaction plot of the two variables (height and air speed) is shown in Fig. 3.

Table 10 Heat transfer coefficient effect estimates based on the final model based on the model formulated without center points

Factor	Effect estimate	Standard error
Intercept	55.11 W/m^2T	
Height	-0.22	±0.35
Temp	-0.97	
Airspeed	1.68	
Fresh %	0.74	
Bandspeed	-0.52	
Height * Airspeed	1.03	
Bandspeed * Airspeed	0.78	

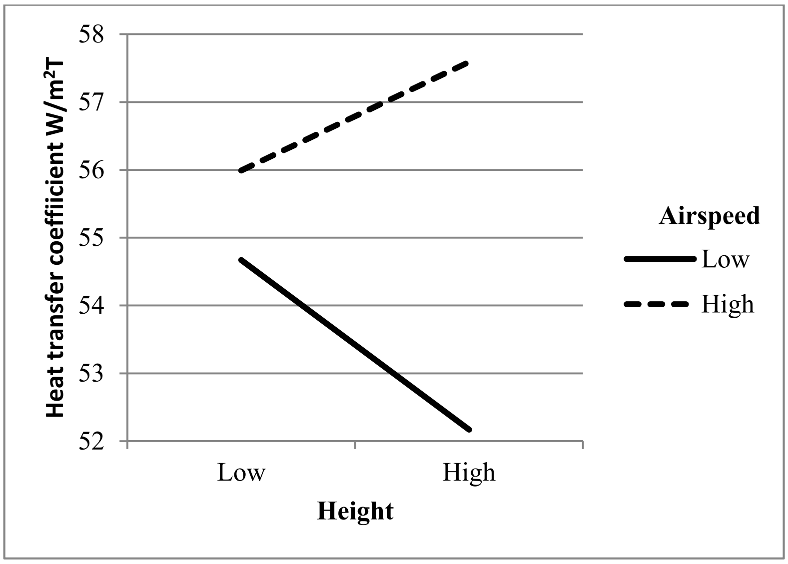

Figure 3 Interaction plot between height of the inlet holes and the airspeed on the measured heat transfer coefficient.

This effect was very relevant in showing that the question concerning the impact of height between baking sheet and inlet holes is possibly important and needs further study. The interaction plot in Fig. 3 shows that there are certain height and airspeed conditions that have higher heat transfer which was also the expectation. Although, it still is another question whether it has any impact when baking products especially when considering the magnitudes of the effects (Table 10). So even though the design chosen were possibly not the "best" and the problems before, during and after experimentation were abundant, the analysis coupled with engineer knowledge gave valuable experience on the equipment and measurement tool and most importantly findings pertaining to improving future experimentation setups.

4 Summary

This small case study shows some of the issue the experimenter has to consider during the execution of an experiment. The ease of execution, the time frame, and the number questions that the experimenter seeks to answer are important points to have as clear as possible from the start. Thinking simple and in terms of flexible designs such as fractional factorials can be proven quite effective when the execution is plagued with a lot uncertainties and hurdles. It is hoped that the description provides a narrative that other people can recognize. The findings of the reported experiment provide the experimenters and company the following discussion points which could be further studied. The pilot plan oven needs further refinement. The use of the structured design and executing it, gave valuable insight in the limitations for the future use of the experimental platform and also possible new design points for consideration. The interaction of the air speed and height needs further studying. The analysis of the response should be worked out. The question is whether the average over the sampled timespan is a useful description of the impingement system.

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Example 2: Discussion

The example documents the many practical consequences that can dictate the design of an experiment. Many of these considerations are directly discussed in the example such as the changing time frame, the measurement equipment issue, etc. A key takeaway point which will be the focus of the discussion is the considerations towards the use of the split plot design protocol. As described in part 2 the split plot design can be used in industrial/process experimentation to account for limitations in how freely or easily certain variables can be changed (Kowalski et al. 2007). It has even been argued that all industrial experimentation is in actuality split plot experimentation due to these limitations on freely changing settings in the processes, attributed to Cuthbert Daniel (Jones & Nachtsheim 2009; Box et al. 2005). In relation to the oven experiment the variable was the height between air inlet and baking sheet that required the dismounting of the oven door. Several arguments were mentioned for using either split plot or the chosen fractional factorial design. In hindsight the choice of using an un-replicated split plot design could have been a safer and less laborious choice in the execution of the experiment. The considerations made against using split plot were the intricacies in designing the experiments which would have to have a fractional subplot design. The intricacies would also be there in designing follow-up experimentation and then the complexity of the analysis.

By performing the experiment experience is gained which also influences future considerations. The choice of using the complete randomization of the treatments with the fractional design gave valuable insight. The door dismounts procedure between each experimental run allowed for a faster cool down and thus resetting of the system between experimental runs. So unbeknownst, the choice between split plot and fractional design actually constituted a choice between fewer door dismounts (safer and easier execution) against the actual performed experiment (faster overall execution and easier analysis). The intention of the argument presented is not to give a wrongful impression that one type of design is superior to another but rather to detail the many practical considerations and observations that can and should influence the experimental design.

Example 3: RSM inspired approach for investigation of dough mechanics (Report II)

This example is based on the use of blocking, factorial and central composite designs. The investigation is the first of its kind trying to assess the influence of using scrap dough on sheeted dough properties. In some industrial bakeries dough is sheeted, cut and shaped. These operations can produce a side stream of dough (called either reuse dough or scrap dough) as an example the sides of the dough sheet trimmed off in order to control shape and generate straight edges. The side stream of dough can then be reintroduced back into one of the preceding process steps. The issue is then how does the scrap dough influence the entire sheeted dough mixture in processability and final product quality. The process is the focus point in this example with the rheological characteristics chosen as an indicator. The theory concerning dough properties is explained in the report. There is however important information worth mentioning in the introduction to the case for framing the design process. As mentioned the investigation is the first of its kind. There are anecdotal reports stating that the use of scrap dough could reduce the resting time. Resting time is a nonspecific term used where the dough after a certain unit operation is "rested" which can for some types of dough or recipes be a necessary and slowing process step. The resting time allows the forces and tension built up in the dough to release and makes the dough more malleable again. This can influence the ability to maintain steady mass flow in automatic sheeting processes. Although these findings are based on model laboratory experimentation and numerical simulation with the caveat that the ability to model and quantify such phenomena still has some way to go (Chakrabarti-Bell et al. 2010). So the experiment was an unstudied application with certain expectations to possible findings, and adding to the complexity the measurement techniques were also under assessment themselves. The report and the following discussion will show the strategy used and learning outcome.

Study on the influence of resting time and dough reuse on sheeted dough rheology by response surface methodology

Søren Juhl Pedersen, Stephanie Henriksen, and Stina Frosch

November 2015

Wheat Dough • Rheology • Scrap Dough • Recycling • Resting Time

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1 Introduction

Wheat flour doughs form the basis of many types of baked products ranging from biscuits and pizzas to products with complex structures such as Danish pastries. The rheological properties of dough are central in understanding the processing conditions for achieving a product with the optimal quality using a minimal amount of work, time and waste. The quantification of the material and rheological properties requires a deeper understanding of the impact and characteristics of typical bakery unit operations, such as mixing, sheeting, lamination, cutting etc., on production efficiency and the product quality (Patel & Campanella 2014). The various types of dough produced for different types of end products depend both on the ingredients applied and the subsequent processing. Several studies on how ingredients influence the dough structure have been published for sugar (Indrani & Venkateswara Rao 2007), salt (Angioloni & Dalla Rosa 2005), enzymes (Collar et al. 2000), fat (Pareyt et al. 2011) and fibers (Collar et al. 2007).

A combination of rheological and textural measurement techniques are often applied due to the parameters for dough, which characterize its ability for handling and baking (Dobraszczyk & Morgenstern 2003; Armero & Collar 1997; Schiedt et al. 2013). However, as others have acknowledged (Patel & Chakrabarti-Bell 2013), there is little information on how constituent dough properties affect sheet-ability although new studies for laminated puff pastry has investigated the effect of the inclusion of fat, the type of fat used and number of fat layers on baking properties (Renzetti et al. 2015; Bousquieres et al. 2014). Furthermore, there are in general few recommendations on what constitute optimal conditions for sheeting, shaping and cutting operations in continuous manufacturing of bakery products (Patel & Chakrabarti-Bell 2013).

Elasticity and extensibility are two important parameters for improving the control of sheeting and cutting operations. In an automated continuous operation, the cutting and piecewise handling of dough is critical for obtaining the set piece weight and shape. The piece weight and shape are also related to sheeting, where the material properties of dough will interact with the rolling operations and thereby influence the mass flow rate along the conveyer. (Chakrabarti-Bell et al. 2010) modelled the sheeting of wheat flour dough and found indications that the flow rate and sheeting properties will depend on the history of the roller operations performed on the dough sheet.

The aim of this study is to investigate the influence of re-using dough trimmings (scrap dough) and variating the resting time after mixing of the freshly prepared dough on the characterizing rheological parameters of the dough sheet. No such studies have been found by the authors. In industrial production lines for bakery products for both savory and sweet products, a side-stream of dough trimmings from the cutting and shaping operations is observed. A common practice is to reintroduce the scrap dough into either the main feed or in the mixing unit thus combining freshly mixed dough with scrap dough. The intention of this research is to bring attention to the use of recycling side streams of dough. This study focuses on the influence of adding scrap dough and variating the resting time of dough on material properties of the dough sheet measured by uniaxial stretching, oscillation and stickiness measurements.

2 Materials and Methods

2.1 Dough preparation

The wheat flour applied for this study is of the type Manitoba (Valsemøllen, Køge, DK). The wheat flour had a protein content of 12.1% with 25-27% wet gluten and a water content of 15.5%. The wheat flour was produced with an extraction rate of 85%. Dough was prepared from 60.6% wheat flour, 8.5% icing sugar (Nordic Sugar, DK), 1.2% salt, 3.6% vegetable margarine (AMA, Thisted, DK) and 26.0% ice / demineralized water mix, where iced tap water was melted in demineralized water. The ingredients were mixed by a TEDDY Bear Varimixer with a dough hook for 6 minutes at speed setting 1. For dough with a resting time greater than 0 minutes, the dough was rested at room temperature with the bowl covered with a wet kitchen cloth according to the designated time of the experimental protocol. The dough was passed through a roller table at sheeting setting 9, folded and twisted 90° with seven repetitions, which resulted in a dough thickness of 20 mm. For experiments including trimmings / scrap dough, the scrap dough was rested for one hour at room temperature covered with a wet kitchen cloth. The scrap dough was added to a new batch of freshly prepared dough during mixing. The freshly prepared dough was mixed for 5 minutes at speed setting 1 with the scrap dough added for one minute of mixing at speed setting 1 for a total mixing time of 6 minutes. The dough was passed through a roller table at sheeting setting 9, folded and twisted 90° with seven repetitions, which resulted in a dough thickness of 20 mm. For the screening experiments, three samples were cut for the oscillatory measurements and then the sheet was rolled at setting 3 to a thickness of 5 mm and three samples were cut for the uniaxial stretching measurements. For extended experiments, dough was sheeted at setting 3 and three samples were cut for each type of measurements (uniaxial stretching, oscillatory and stickiness). A flow chart of the dough preparation process is presented in Figure 2.

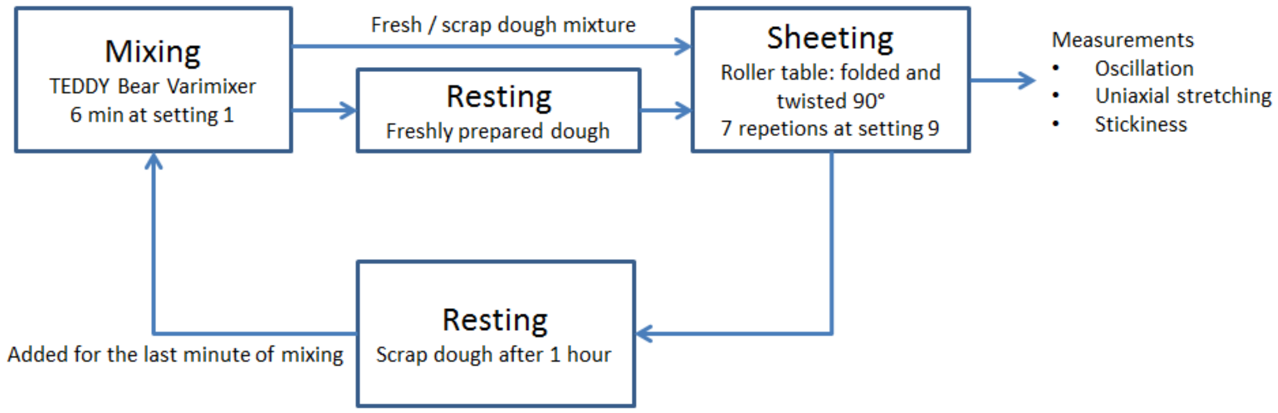

Figure 2: The dough preparation process applied in this study

2.2 Oscillatory measurements

Oscillatory measurements were performed on a Haake Mars Rheometer II (Thermo Haake, Karlsruhe, Germany) using flat serrated plates with a diameter of 6 cm and a gap of 4 mm. Polyethylen-polypropylenglycol (PPG), a rheological modifier, was applied to the exposed dough edges to prevent evaporation. Measurements were performed at a strain level of 0.1%, which is within the linear viscoelastic region, and with a frequency level ranging from 0.05-100 Hz. An example of the resulting elastic and loss moduli and the apparent viscosity as a function of frequency is shown in Fig. 2.

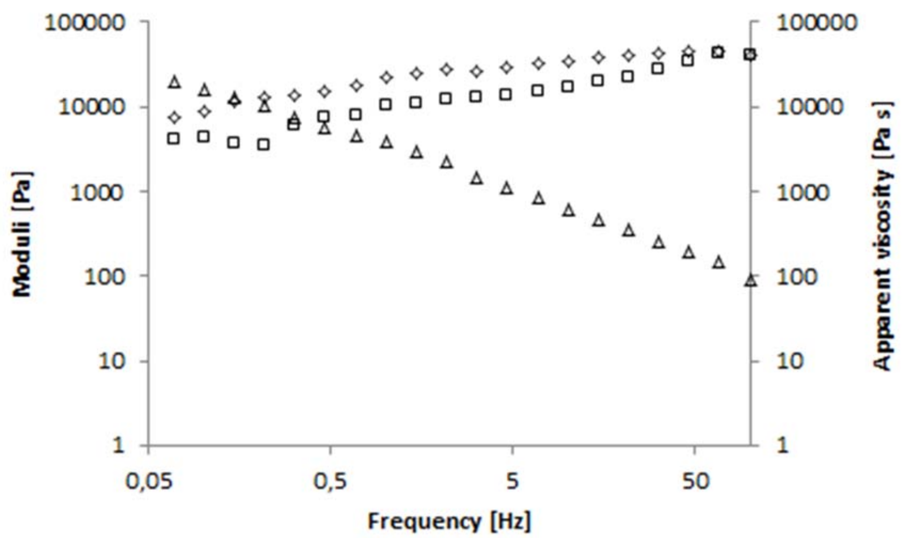

Figure 3: Example of the resulting storage and loss moduli and the apparent viscosity according to the chosen frequency range from the oscillatory measurement. The diamond (◊) resembles the storage modulus (G'), the square (□) resembles the loss modulus (G''), and the triangle (Δ) resembles the apparent viscosity (|η|). The graph is originating from a dough sheet with 25% scrap dough that has rested for 15 minutes.*

The levels of the storage and loss moduli at 10 Hz were noted. A power law regression was fitted to the moduli in the frequency range of 0.1-10 Hz, and the slope, and intercept parameters were calculated. The tangent to damping factor was calculated from the slope (Tanner, 2000):

$$tan\delta = tan\frac{p\pi}{2} \quad \text{(Eq. 1)}$$

Where the power law slope is p.

One measurement was performed for each of the three samples from the dough sheet.

2.3 Uniaxial stretching measurements

A setup mimicking the burst rig (Larrosa et al. 2013) setup was utilized using a Texture Analyzer TA-XT plus (Stable Micro Systems, UK). Sheeted dough was placed on top of a circle carved platform with a diameter of 7 mm. The dough was covered with a vinyl coated flask stabilizing ring weighting 997 g with a diameter of 10 mm. A 1" Spherical Probe Plastic (P/sp) (Stable Micro Systems, UK) was forced into the center of the dough with a velocity of 1 mm s^{-1} until reaching a travelled distance of 50 mm. An example of the resulting force-distance graph for the uniaxial stretching measurement can be seen in Fig. 3.

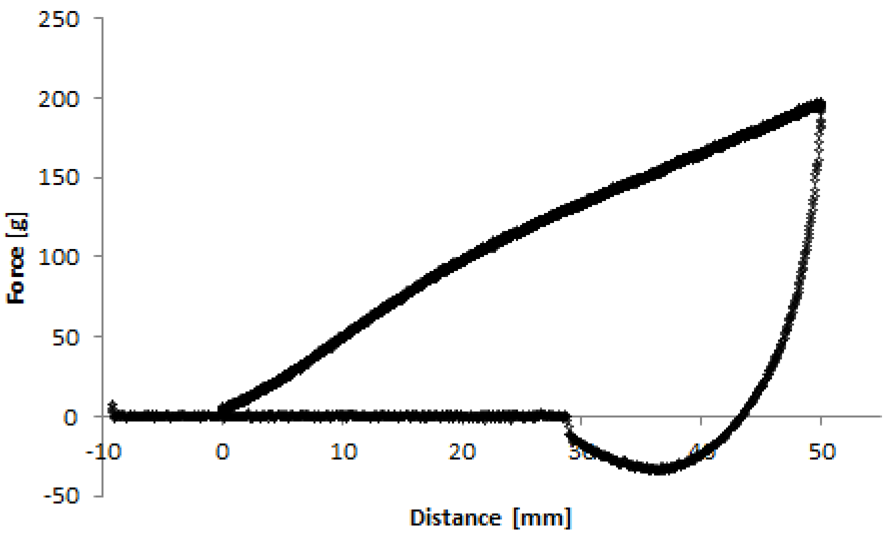

Figure 4: Example of the resulting force-distance graph from the uniaxial stretching measurement. The graph is originating from a dough sheet with 25% scrap dough that has rested for 15 minutes.

A linear regression was fitted in the regions of 2-10 mm and 20-45 mm distance, and the maximum force reached was noted. The work from distances of 0-50 mm, 50 mm to the force intercept and from the first to the second force intercept was calculated. One measurement was performed for each of the three samples from the dough sheet.

2.4 Stickiness measurements

The stickiness measurements were conducted using Texture Analyzer TA-XT plus (Stable Micro Systems, UK) by a SMS/Chen-Hoseney Dough Stickiness Cell (A/DSC) and a 25 mm Perspex Cylinder Probe (P/25P). Dough was placed within the rig and screwed for the dough to move through the holes of the rig. The first dough moving through the rig was removed in order to create an even height of the dough strips, and then the dough sample was screwed to the top of the cell. The sample was rested for 30 seconds prior to measurement. The probe was set up to travel 4 mm down into the sample with a velocity of 0.5 mm/s. The probe maintained its position for 0.1 seconds at a force of 40 g and moved upwards with a velocity of 10 mm/s. An example of the resulting force-distance graph for the stickiness measurement is illustrated in Fig. 4.

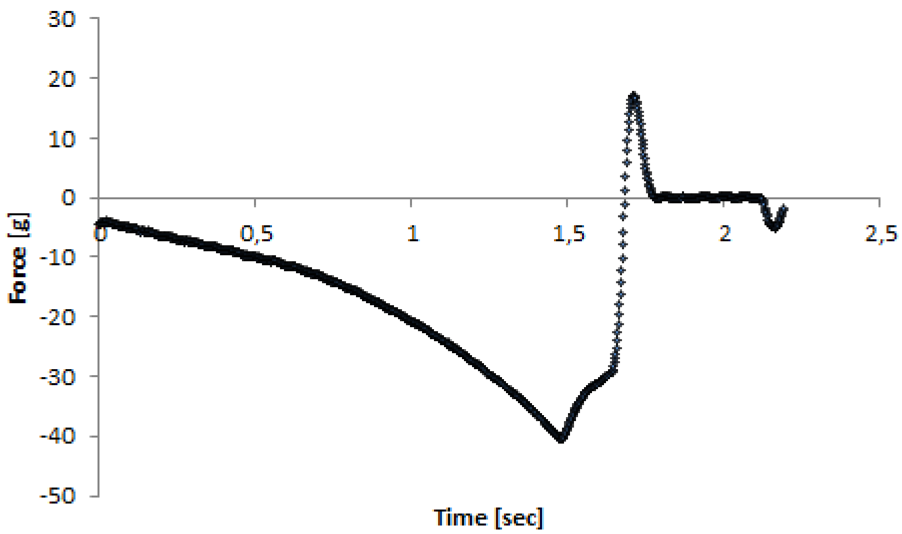

Figure 5: Example of the resulting force-distance graph from the stickiness measurement. The graph is originating from a dough sheet with 25% scrap dough that has rested for 15 minutes.

The maximum force and distance travelled were noted, and the stickiness force work from the minimum force to the distance intercept was calculated. One measurement was performed for each of the three samples from the dough sheet.

2.5 Design of experiments

The experiments performed were divided into a screening experiment and an extended experiment. The levels of scrap dough and resting time for the screening experiment was chosen to assess the influence of scrap dough on the resting time, and also to assess whether there would be difference between a designated no scrap dough, and the dough that would become the scrap dough. The last assessment was important to detect any possible effects of scrap dough. For the extended experiment the scrap dough and resting time levels where chosen to expand the response along the resting time axis and to allow for a complex model structure to be fitted to the observations. An overlap of the experiments on the workspace of the used factors can be seen in Fig. 5.

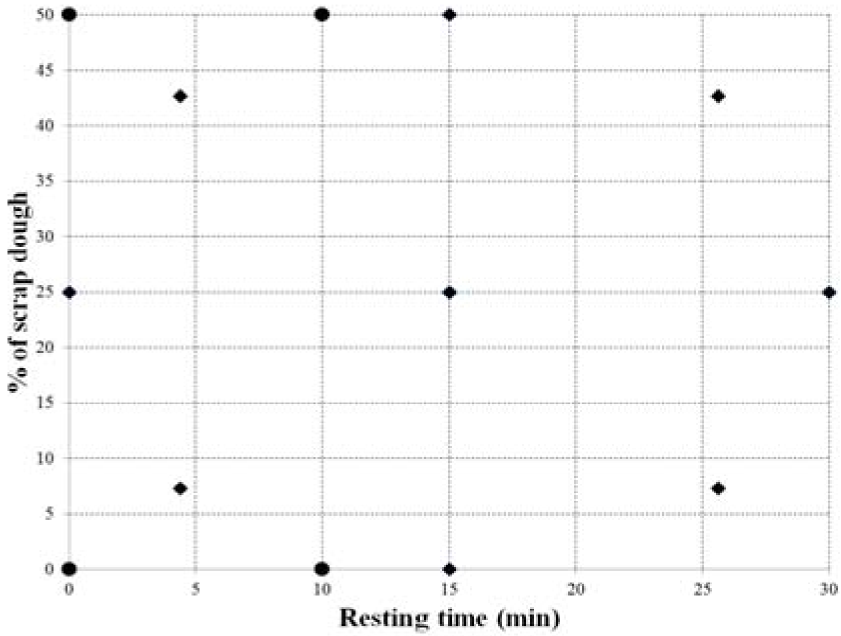

Figure 6: The experimental region showing the levels for the two experiments performed. The 2^2 factorial screening experiments are indicated by a solid dot (●) and the CCD extended experiments are indicated by a solid diamond (♦).

The screening experiment utilized a blocked 2-level factorial experiment with replication. The experiment was replicated the following day as shown in Table 1.

Table 1: The experimental design for the screening experiment. The table shows the settings and the run order for the two blocks.

		Coded		*Settings*	
Block	**Run order**	**Resting time (min)**	**% re-cycled dough**	**Resting time (min)**	**% re-cycled dough**
B1	2	-1	-1	0	0
	1	1	-1	10	0
	4	-1	1	0	50
	3	1	1	10	50
B2	3	-1	-1	0	0
	2	1	-1	10	0
	4	-1	1	0	50
	1	-1	1	10	50

For the extended experiment, the response surface design chosen was a central composite design (CCD) performed in two blocks allowing the design to be both rotatable and orthogonal (Box & Draper 2007). The levels for the CCD experiment were chosen to improve the estimation within the varying amount of scrap dough from 0-50% and resting time from 0-30 min. The CCD design can be seen in Table 2.

Table 2: The experimental central composite design for the extended experiment. The table shows the settings and the run order for the two blocks. The design is both rotatable and orthogonally blocked.

		Coded		*Settings*	
Block	**Run order**	**Resting time (min)**	**% re-cycled dough**	**Resting time (min)**	**% re-cycled dough**
B1	5	-1	-1	4	7
	4	1	-1	26	7
	6	-1	1	4	43
	1	1	1	26	43
	2	0	0	15	25
	3	0	0	15	25
B2	4	1,414	0	30	25
	5	-1,414	0	0	25
	2	0	1,414	15	50
	3	0	-1,414	15	0
	1	0	0	15	25
	6	0	0	15	25

2.6 Statistical Analysis

The statistical analysis was conducted with the use of Microsoft Excel 2010 (Microsoft Corporation, Sacramento, USA) and R (R development core team, 2014). ANOVA was applied for assessing factors for the two level factorial experiments. ANOVA and student's t-test was used for the comparison between control points and dough designated for scrap dough (Fig. 1). For the response surface modelling (CCD experiment) regression and ANOVA, the statistics and contour plots model for the resulting significant models were assessed. The alpha value used for test statistics were 0.05 and model hierarchy if the parameter interaction was significant.

3 Results and Discussion

The dough used for scrap dough was compared with the dough without scrap dough as a control test in order check whether the starting doughs were reasonably similar to the doughs without scrap dough addition. The results showed no significant differences for both textural- and rheological parameters. The blocked 2-level factorial experiments had measurements on the uniaxial stretching and rheological parameters. The handling of the dough still needed improvement the sampling for the texture and rheological measurements were performed at wrong settings. This did not influence the control test but did mean that the results could not be used for comparison with the expanded CCD experiments. They were only relevant in and of themselves. The results did indicate that scrap dough and resting time had a similar effect on the material properties but that these effects were not additive. This was due to the points at no addition of scrap dough and no resting time gave similar results as when the settings were with scrap dough and resting time. These findings indicate that there could be some validity in the anecdotal comments concerning the relationship between resting time and the use of scrap dough.

The full set of experiments from the CCD experiments were tested by ANOVA and regression modelling in order to determine if the content of scrap dough or resting time period had a significant impact on the measured rheological and textural parameters. The rheological and textural parameters investigated are listed in table 3 and crosses mark whether the parameters was found to be influenced by scrap dough / resting time or not.

Table 3: Table showing whether the scrap dough content or the resting time had a significant impact on the measured rheological and textural parameters. The crosses mark whether the parameters are significantly impacted or not.

	Significant impact	**No significant impact**
RHEOLOGICAL PARAMETERS		
G' (frequency=10 Hz)	x	
G'' (frequency=10 Hz)	x	
G' intercept for power regression	x	
G' slope for power regression		x
G'' intercept for power regression	x	
G'' slope for power regression		x
Tan δ		x
TEXTURAL PARAMETERS		
Uniaxial stretching		
Slope 1: 2 to 10 mm	x	
Slope 2: 20 to 45 mm		x
Max force	x	
Work down: 0 to 50 mm	x	
Work up: 50 mm to force intercept		x
Adhesiveness: First force intercept to second force intercept		x
Stickiness		
Max force		x
Work		x
Distance travelled		x

3.1 Rheology

From Table 4 it is seen that the levels of the loss (G'') and storage (G') moduli at a frequency of 10 Hz and the modelled intercept are significantly affected by the content of scrap dough and the resting time. However, the slopes of the moduli within a frequency region of 0.1-10 Hz are not affected, and the same is the case for the tangent to the damping factor.

The equations for the mean of the depending rheological parameters are listed in Table 4. The standard error of the regression coefficients are shown for the fitted models on the responses directly measured. The models are fitted according to a heirarchy principle that if the interaction is significant then the main factors are also included in the final model.

Table 4: Listed equations for the mean of the resting time- and scrap dough content depending rheological parameters.

	Intercept	**Resting time**	**% Reuse**	**Interaction**	**(Resting time)^2**	**(% Reuse)^2**	**Adjusted R^2**
G' (frequency=10 Hz)	49824	188	-775	-2015	ns	ns	0.5402
Std. Error	3612	626	626	885			
G'' (frequency=10 Hz)	23407	74	-309	-918	ns	ns	0.5089
Std. Error	1730	300	300	424			
G' power regression intercept	27771	22	-454	-1000	ns	ns	0.5482
G'' power regression intercept	13610	30	-217	-450	ns	ns	0.6841

It can be observed from Table 4 that a longer resting time will increase the levels of the storage and loss moduli. On the other hand, by increasing the amount of scrap dough, the parameters will be lowered. The same is seen for the interaction effect. The squared effects of resting time and the amount of scrap dough did not show a significant effect. Contour plots illustrating the mean levels of dependent rheological parameters at different resting times and scrap dough contents are presented in Fig. 6.

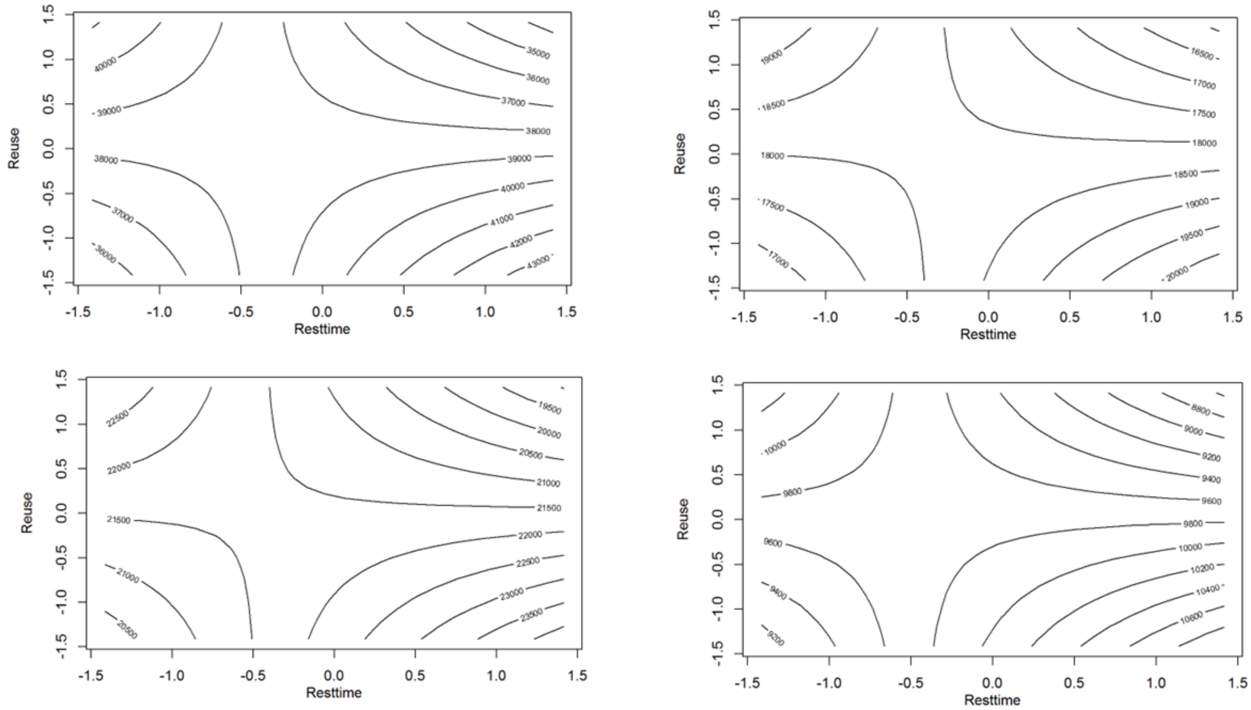

Figure 7: Contour plot illustrating the levels of the depending rheological parameters at different resting times and scrap dough contents. In the top left corner is the contour plot for G' (frequency=10 Hz), in the top right corner for G'' (frequency=10 Hz), G' power regression intercept in the bottom left corner, and the contour plot for G'' power regression intercept in the bottom right corner.

As seen at Fig. 6, for the storage and loss moduli, the surface fitted showed that the experimental region covered a saddle point. The gradient of G' and G'' increases from the control point to the center point and decreases at the other end of the diagonal. The opposite is seen for the reversed diagonal. The results indicate that the quantities of the moduli will be at approximately the same level if scrap dough is added as seen by applying rest time without the addition of scrap dough. For example, a similar level for the storage modulus is seen for a resting time at approximately 15 minutes and no scrap dough as seen for dough containing approximately 43% scrap dough without any resting time. The same behavior is seen for the loss modulus. The results of regression models on G' and G'' in Table 4 when considering the standard errors indicate that the resting time could be too short to detect any change. The standard errors also show that the models fitted are due to the significant interaction terms.

3.2 Texture

From Table 5 it is seen that for the uniaxial stretching measurements, the slope from 2 to 10 mm, the maximum force, and the work into the dough sheet from 0 to 50 mm were all significantly impacted by the changing resting times and content of scrap dough. However, the slope from 20 to 45 mm into the dough sheet, the work from 50 mm to the force intercept and the work related to stickiness were not significantly dependent on the changing scrap dough content and resting time. For the specific stickiness measurements, none of the noted parameters proved to be significantly impacted.

The scrap dough content and resting time dependent uniaxial stretching parameters are all related. If the first slope is lower, so will the maximum force reached be, as long as the second slope is somewhat constant. When the maximum force reached is lowered, so will the work area be when the distance travelled is constant. The stickiness related work from the uniaxial stretching measurements did not prove to be significant; the same behavior was seen for the parameters from the specific stickiness measurements.

The equations for the mean of the depending textural parameters are listed in Table 5.

Table 5: Listed equations for the mean of the resting time- and scrap dough content depending textural parameters.

	Intercept	Resting time	% Reuse	Interaction	(Resting time)^2	(% Reuse)^2	Adjusted R^2
Slope 1	5.47	0.25	-0.31	0.49	ns	ns	0.5853
Std. Error	0.70	0.12	0.12	0.17			
Max force	1.89	0.09	-0.14	ns	ns	ns	0.5328
Std. Error	0.24	0.04	0.04				
Work down for uniaxial stretch	5952	241	-350	ns	ns	ns	0.7575
Std. Error	406	70	70				

From Table 5 it is seen that by prolonging the resting time, all the depending textural parameters will increase. On the other hand, by adding scrap dough, the parameters will be lowered. The interaction effect will cause an increase of the slope 1 parameter, whereas the interaction effect is not significant for the remaining parameters. The squared effects of resting time and scrap dough did not show a significant effect for any of the parameters. Contour plots illustrating the levels of dependent textural parameters at specific resting times and scrap dough contents are presented in Fig. 7.

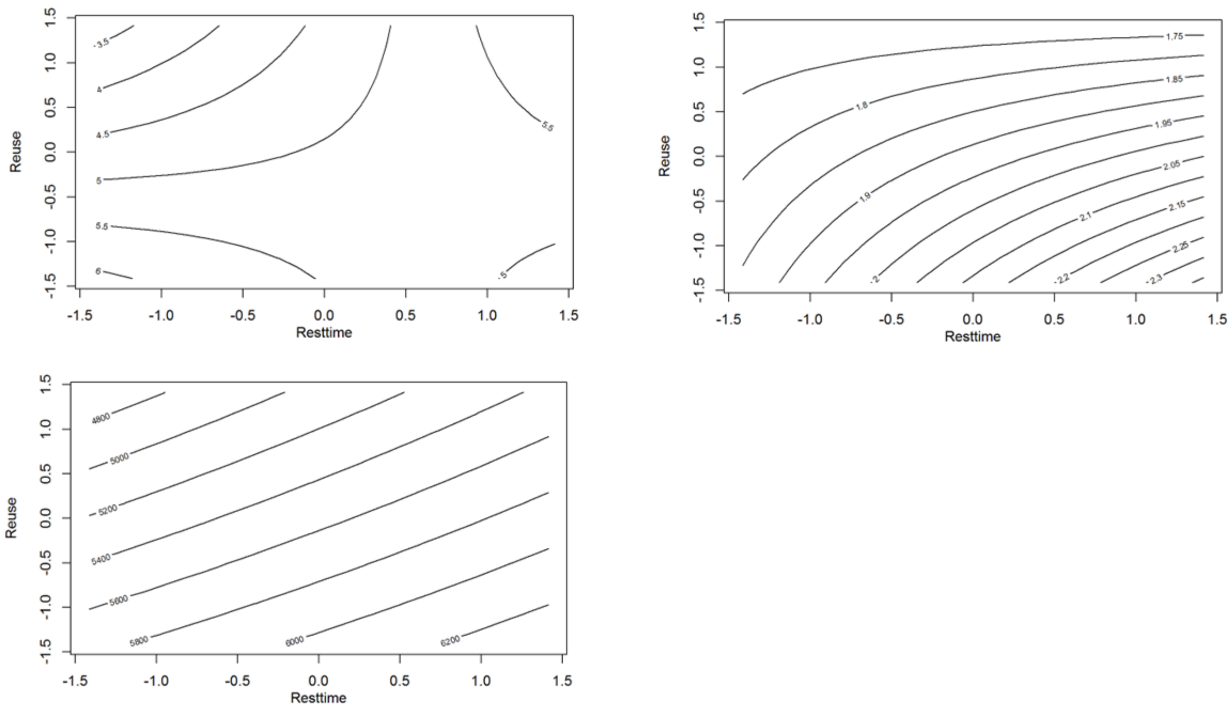

Figure 7: Contour plot illustrating the levels of dependent textural parameters at specific resting times and scrap dough contents. In the top left corner is the contour plot for slope 1, in the top right corner for the max force reached, and the work from 0-50 mm travel into the dough sheet in the bottom left corner.

As seen at Fig. 7, the slope of the force from 2-10 mm into the dough sheet covered a saddle point, whereas the maximum force reached follows an exponentially curved plane, and the work from 0-50 mm travel into the dough sheet shows a linear plane. The saddle point for slope 1 is displaced down and to the right compared to a reflection around the extreme diagonal from the control point to a resting time of 30 min and a scrap dough content of 50%. The gradient of slope 1 increases from the upper left corner to the lower left corner. The highest slope value is thus found at the control point. For the maximum force, the curvature causes the value of the maximum force to be dependent on the resting time, when no scrap dough is added, and less dependent on the resting time for higher scrap dough contents with no resting time. The highest value of the maximum force is found for a resting time of approximately 30 min without the additiong of scrap dough. For the work down into the dough sheet, the plane is increasing as the resting time increases and the slope is not depending on the resting time. The biggest work is thus seen for a longer resting time without the addition of scrap dough.

4 Conclusions

ANOVA and regression modelling was applied in order to detect whether the noted rheological and textural parameters were significantly affected by a change in the content of scrap dough or in the resting time. Four rheological and three textural parameters proved to be significantly impacted. The stickiness measurements should not be used for doughs of the gluten strength as used in this experiment. If a weaker wheat flour or doughs with higher water or fat content then it would still be worthwhile to include the stickiness measurement. It was noted throughout the experimentation that there were several steps such as the mixing and sheeting that had be fine-tuned. The conclusion was that high degrees of craftsmanship or lab experience with doughs were needed in order to further investigate reuse/scrap doughs influence on sheeting and processability. This has been a general finding for previous work on dough rheology.

For the scrap dough and resting time significantly depending rheological parameters (G' and G'' at a frequency of 10 Hz, and G' and G'' at the power law intercept), the models showed that the resting time increased the moduli and the same but to lesser degree was seen for the reuse dough. Furthermore, the interaction effect between the scrap dough and resting time causes a greater lowering of the parameters than seen for the addition of scrap dough. From the mean values, it was indicated that it was possible to obtain similar parameter levels by adding scrap dough without any resting time as when resting time was applied but no scrap dough added. However the uncertainties in the main effects of the model parameters were high so the results are deemed speculative.

For the scrap dough and resting time significantly depending textural parameters (slope from 2-10 mm into the sample, maximum force, and work 0-50 mm into the sample), the statistical analysis showed that a longer resting time would result in higher parameter levels, while an increased amount of scrap dough would cause lower parameter levels. Furthermore, the interaction effect between the scrap dough and resting time would increase one of the parameters. From the mean values, the possibility to add scrap dough with no resting time instead of having a resting time with no scrap dough added was not seen for any of the parameters.

So, there is a speculative potential to save production time and lower the waste by leaving out the resting time and instead add scrap dough. However, the method applied for this study does not follow the industrial production, since the resting time designated to the experimental protocol would be placed after the addition of scrap dough as illustrated in Fig. 8.

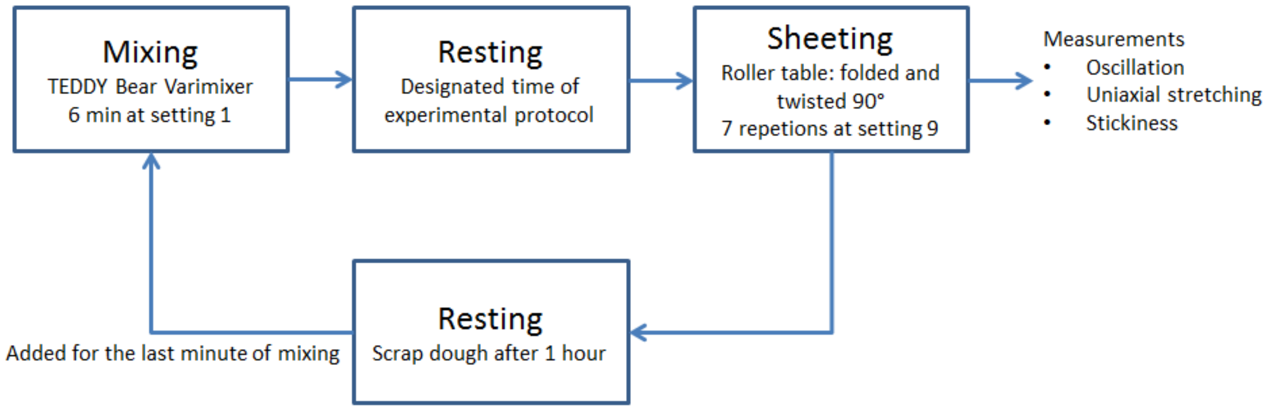

Figure 8: Flow chart following the industrial dough preparation.

5 Future work

This study did show some interesting results about the relationship between the addition of scrap dough and the length of the resting time, since the resting time can potentially be shortened or eliminated by adding scrap dough. However, since the resting time have been inserted before the addition of scrap dough instead of after the addition of scrap dough, as would be seen in the industry, it would be an idea to change this in the method, for obtaining results that are more useful for the industry. The rheological measurement technique could also be reevaluated. Maybe amplitude sweep would be better a measurement although this would take some further calibration work on the rheometers. Techniques like large amplitude sweep have been reported to describe the material properties of dough more accurately with respect to the complexity of the material (Ng et al. 2011).

Furthermore, one could calculate the work for the uniaxial stretching by the work from 0-50 mm attracted the work from 50 mm to the force intercept, which would be a more accurate measure. The uniaxial platform should also be improved with a proper clamping system to hold the dough. Slippage had occurred at some instances, unfortunately.

Measurements at industry should be made in order to get reference values of the material properties.

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Example 3: Discussion

The experiments consisted of 2 sets of experiments. The first was the 2-level factorial experiment wherein there was included a control sampling. This was the preliminary experiment where the key question where if the experiments could be properly executed. The experiment included a sampling of fresh dough mix from the dough which was going to become scrap dough. These samples were for checking whether the dough that later would become scrap dough, was initial similar in material properties as the 0% scrap dough experimental runs. The sampling and measurements control check of the dough was time consuming. The results were that the all fresh dough mixture was reasonably similar. This meant that the sampling and measurements could be scraped in the following experiments. The major constraint again for all the experiments was time. It was approximated and later verified that roughly 6 experimental runs could be executed per day. As mentioned it was decided that the sampling and measurements of freshly mixed dough designated for scrap dough was dropped. This meant 2 extra experimental runs were freed, so that all possible 6 experimental runs could be utilized for actual design points.

For the following experiment it was chosen to extend the experimental region with respect to the time factor so that the highest setting was 30 mins. The design chosen was a central composite design because there was expected some curvature in response surfaces for some of the measured properties. This would at least fit with the anecdotal comments concerning the use of scrap dough and resting time. The results of the experiments did indicate that there could be some validity to the anecdotes. The major issue with these experiments was the difficulty in working with the dough. The issue of the use of scrap dough is important with respect to processability specifically lowering waste and improving production efficiency. The issue with the lab experimentation of such scenarios is that to replicate industrial conditions is almost impossible mainly due to scale differences which are important when working with material that is as history dependent as wheat dough is known to be. The results from these experiments could only be used for indicating which factors on industry scale should be looked at.

Example 4: Designed experiment with a multivariate response (Paper B)

This example is focused on utilizing a new exploratory analysis procedure, ASCA, in the analysis of designed experiments. The novel analysis approach combining principal component analysis and ANOVA has been used on images as the response. The experiment had two factors, air speed and type of baking sheet with the aim on understanding the implications choosing a baking sheet type. Nuisance factor such as blend variation, duration of experimentation and position on baking sheets were all concerns that had to be dealt with. The batch to batch variation of cookie dough mixture and duration of experimentation was handled simultaneously by blocking for days and having one batch of dough mixed for each of those days. This meant that the batch to batch effect was confounded with day to day effect i.e. part of the same blocking factor. The within day duration effect on experimentation was dealt with by randomization. The position on baking sheet was recorded with respect to a reference frame so that information about the position could be included in the analysis. The design used was simple although there are some special concerns with blocking a mix level factorial experiment (ref) which was not dealt with. The intricacies were in the analysis of the results. The design falls in the category of mixed level factorials i.e. one factor has 2 levels and another 3levels. No considerations in designing the experiments were made towards the type of response generated by the images. The image analysis steps were ad hoc procedures for extracting the information of importance from the image. The two priorities were that it could be analyzed with respect to the multivariate approach chosen and at the same time have some direct interpretability. The discussion following the paper will try to give some more information concerning the analysis method ASCA.

Analysis of designed experiment with cookie images as multivariate response using ASCA for assessing process effects on surface appearance

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Abstract

The focus of this study was on assessing the impacts of airflow and baking sheet design on the appearances of butter cookies baked under air impingement conditions. The appearances of the cookies were assessed by objective measures of digital images of each individual cookie. The experiment was to vary 2 air flow settings and 3 types of baking sheet material. The cookies were baked at a temperature of 220^{o}C for 5 mins. Images were taken 24 hours after baking. The image data were converted into color histograms for the entire surface of the cookies and for each subdivision in circular regions of the images. For the analysis a novel methodology, ASCA, was implemented in order to utilize the experimental structure and to analyze the multivariate response. The analysis showed that there were different spatial impacts for each of the experimental factors; the air flow had an effect on most of the surface whereas the baking sheet was confined to impacting the edge of the cookies. There also was a nontrivial interaction of air flow and baking sheet. The methodology and the analysis were so general in nature that they can be applied to many different experimental situations where the response is multivariate as in the example with images as the response.

Keywords

Image analysis ; ASCA ; Baking; Multivariate response

1 Introduction

In this study we propose a novel approach to the problem of assessing process parameters impact on finished product appearance. The challenging task is to address the appearance of the product, which in this case are cookies, and how the processing conditions impact color and shape. The approach presented in this paper is based on the coupling of classical design of experiments with a multivariate analysis.

Physics based modeling assists in understanding and predicting the rate of change of mass and temperature under different processing conditions (Datta, 2008). However the connection between modeling activities and actual appearance is an issue where physical experimentation is needed for building equations relating quality and process conditions (van Boekel, 2008). This paper uses a novel analysis method for analyzing the images of cookies produced under different conditions where temperature and time is kept constant. The conditions directly related to the heat and mass transfer processes which impact the appearance of baked goods (Purlis, 2010), such as airspeed and baking sheet design, were systematically varied. Zareifard et al. (2009) showed that by changing the convective component but keeping the total heat flux constant, different product appearances were obtained which could be seen in the images and in the calculated total color differences ΔE based on the L,a,b color space. As the authors Zareifard et al.(2009) note, oven producers focus on optimizing energy utilization in baking ovens and heat flux, but they pay little attention to the resulting product appearance. Hadiyanto et al. (2007) describe the need for models based both on physical principles as well as empirical models in order to elucidate additionally the spatial quality attributes of the food product. The need for coupling product kinetics with process modeling in order to optimize both the usage of utilities and product quality is of major importance for the improvement of food processing.

The appearance of a food product is the first quality parameter assessed by the consumer (Wu & Sun, 2013). Thus, it is of critical importance to be able to control the product appearance and also to ascertain relations between defects and processing conditions in order to produce the desired product. The color formation of cookies is due to browning processes at the surface of the cookies. Purlis (2010) presents an exhaustive review of the browning development for baked goods. The paper emphasizes the importance of understanding the browning formation for the process design and the improvement of existing processes which have also been studied by Hadiyanto et al. (2007) and Hadiyanto et al. (2008). Gökmen et al. (2007), Gökmen et al. (2008), and Lu & Zheng (2012), for example, show the correlation between color formation in baked goods and the generation of possible carcinogens. It is therefore important to control color formation not only to supply a high quality product but also to reduce the formation of harmful byproducts.

Vision systems are a growing field of application within the food industry especially for quality control, monitoring and classification for contamination risks (Mogol & Gökmen 2014). The baking industry has many applications both in quality measurement techniques and also in monitoring (Sun, D-W.2012). Several papers demonstrate promising techniques for the evaluation of color of cookies using image data. Nashat & Abdullah (2010) tested the use of charge-coupled device (CCD) cameras using the red-green-blue (RGB) image combined with support vector machines (SVM) for classification of cookies into groups that correspond to degrees of baking, from light for underbaked to dark for overbaked cookies. Andresen et al. (2013) used RGB images combined with browning score evaluation by a sensory panel to construct a classification model that predicts the pixel state of the cookie. Moghaddam et al. (2015) illustrate how to convert RGB images into LAB scale and estimated kinetic parameters for the lightness (L) and ΔE values for cookies that were deep fat fried.

The focus of this study is to apply an objective measure on the appearance of cookies in order to assess the effects of process conditions. This paper's measure uses digital images, which have a 3 dimensional array structure of data for each image. This type of data structure is atypical for the classical tools used in analyzing experimental data where the response is usually single valued. In order to analyze the experiment we apply the ANOVA-Simultaneous-Component-Analysis (ASCA) method which is an analysis combining the ideas of analysis of variance (ANOVA) and principal component analysis (PCA) (Smilde et al. 2005; Smilde et al. 2008; Jansen et al. 2005). The issue lies in the problems of combining the structure of the experimental design with the multivariate data set in order to analyze the different factors' impacts independently. Multivariate analysis of variance (MANOVA) is the multivariate extension of ANOVA but the method is not trivial in application to models with the inclusion of interaction terms and there are also problems in choosing test statistics (Ståhle & Wold, 1990). Other proposed methods include partial least squares (PLS) and principal component ANOVA (PCA-ANOVA) which all have their merits and caveats. We refer the reader to the literature on these topics discussed in papers by Sarembaud et al. (2007), Ståhle & Wold (1990) and Zwanenburg et al. (2011). The use of ASCA provides a more general methodology for the analysis of the experiment when the response is multivariate. In the application we also test the stratification of cookie surface regions with the intention of assessing whether the different process factors impact different parts of the surface area.

2 Materials and Methods

2.1 Preparation of butter cookies

The dough for the butter cookies were made in one batch for each day of baking respectively. The recipe consisted of 1200g wheat flour (12 % protein, Strong-flour, Valsemøllen, Denmark), 330g pasteurized whole eggs (Danæg, Denmark), 450g icing sugar (Nordic Sugar, Denmark) and 900g salted butter (Arla Foods, Denmark). Each batch of dough was prepared following the same procedure. The flour and icing sugar were stored at ambient temperature in sealed containers, and the eggs and butter at normal refrigeration of 5^{o}C. The icing sugar and butter were whipped for 2 minutes, and then whole eggs were added and then mixed further for 1 minute. Finally the flour was added, and the dough was mixed further for 2 minutes. The mixing was made with a kitchen-top mixer and a spade beater, 5L Bear Varimixer Teddy (Varimixer, USA). After mixing, the dough was tightly covered with cling film and stored for 48 hours at refrigeration (5^{o}C). After storage, the dough was portioned out into smaller batch sizes. Each small batch was rolled with a Rollmatic Manual Sheeter (Rollmatic Srl, Italy) to a thin sheet of 6mm height. The cookies were then cut with a cylinder steel cutter of 42 mm in diameter. The resulting dimensions of the cookies were 6 mm in height and 42 mm in diameter. 16 cookies were cut from each sheet and stored on the same tray covered with cling film at refrigeration temperature (5^{o}C) before baking. The sheeting and cutting of the cookies were done on the same day of baking. The placement of the cookies on the baking sheets was controlled and recorded (Table 1).

Table 1 shows the position code and the placement on the baking sheet.

<table>
<tr><td></td><td colspan="4">Back of oven</td><td></td></tr>
<tr><td rowspan="4">left</td><td>1</td><td>2</td><td>9</td><td>10</td><td rowspan="4">right</td></tr>
<tr><td>3</td><td>4</td><td>11</td><td>12</td></tr>
<tr><td>5</td><td>6</td><td>13</td><td>14</td></tr>
<tr><td>7</td><td>8</td><td>15</td><td>16</td></tr>
<tr><td></td><td colspan="4">Front (Door)</td><td></td></tr>
</table>

2.2 Baking

This study used a pilot scale oven of an industrial convection type (Andresen et al. 2013). The air flow impinged on the baking sheet from both top down and bottom up. The inlet nozzle array was set to oscillate at 1.5 mm/s to mimic continuous oven baking. The oven was preheated for 1 hour before setting the air flow conditions for the first experiment, followed by a 30 minute period for the system to reach steady state for the first experimental setting. Between each experimental run / baking, the oven was run empty for 30 minutes to flush out minor residual humidity and to stabilize before the next run. The baking time (5 min) and temperature (220^{o}C) were kept the same for all the experiments. Three types of baking sheet material were used, normal steel baking sheet, perforated steel baking sheet and stainless steel chain link sheet. Two airflow settings were used, corresponding approximately to 7 m/s (low) and 9 m/s (high) at the inlet nozzles. All combinations of air speed and baking sheet type were used, and the experiment was replicated 3 times over a 2 week period. The factor combinations and the protocol of the design can be seen in Table 2 and Table 3, respectively. After baking, the cookies were cooled at ambient temperature and then stored 24 hours in sealed containers.

Table 2 the code given the different combinations of factors

	Steel	Chain link	Perforated steel
Air speed low	1	2	3
Air speed high	4	5	6

Table 3 shows the actual run order the experiments with the numbers referring to the code in Table 2.

	Blocks			
Run order	1	2	3	4
1	5	6	4	1
2	3	2	1	6
3	1	5	2	4
4	4	1	5	3
5	2	3	6	5
6	6	4	3	2

2.3 Videometer lab pictures

The equipment used for image acquisition was a combined camera and lighting system called VideometerLab (Videometer A/S, Denmark). The images were taken using a Point Grey Scorpion SCOR-20SOM greyscale camera. The cookie was placed in a plastic petri dish inside an integrating sphere (Ulbricht sphere) with light emitting diodes placed in the rim around the sphere (1). The curvature of the sphere and its matte-white coating ensured a uniform diffuse light so that specular effects were avoided and the amount of shadow was minimized. The device was calibrated radiometrically following light and exposure calibration according to the National Institute of Standards and Technology (NIST). The system was also geometrically calibrated to ensure pixel correspondence for all spectral bands (2). The Scorpion camera had a 12 bit analogue to digital converter (ACD), and the system used an 8 bit data output from the camera. The correction for calibration gave a reflectance intensity output of 32 bit precision. The image resolution was 2056 x 2056. In this situation one pixel equaled approximately 45 microns, hence the image represented approximately a 9.252 x 9.252 cm area. The RGB images were generated by averages of reflectance intensities of the 9 lower wavelengths (400-700 nm), first 3 gives the blue color, second 3 gives green and the final 3 are averaged giving the red color intensity.

2.4 Image processing

The image processing was performed using Matlab (2014b, MathWorks, USA) and the image processing toolbox. The region of interest (ROI) was the entire cookie in each picture. The steps for capturing the ROI are shown in Fig. 1. The technique employed is a border search function inbuilt in Matlab´s image processing toolbox. The first step uses an image slice (i.e. one of the wavelengths) and partitions the image by setting intensities below a specified value as 0 (Fig. 1. Image A to B). The next step performs a boundary search for the biggest object in the image (Fig. 1. Image B to C). This step first removes data known to be redundant. It next uses the intensity values to better delineate cookies from the background and at the same time search for "big" regions (identifying a cookie and not a crumb). The complete procedure automates the steps and generates distinct ROI's for each image.

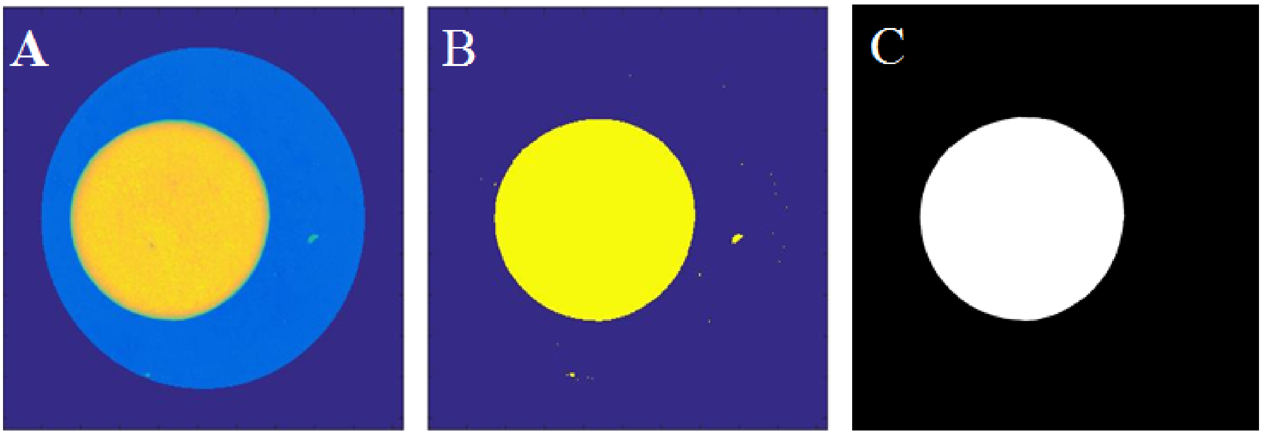

Figure 1 shows the automated steps in defining the ROI of a cookie image. Pic. A Shows a wavelength image slice used to delineate background from cookie giving the resulting Pic B. Pic. C shows the border search result performed in pic. B extracting the biggest object found in the image.

Morphological parameters were calculated using built-in image processing toolbox functions for each cookie. The parameters were Area, Major and Minor axis lengths, and Eccentricity. The color information of the RGB images was analyzed using histograms of the color channels both separately and combined. The combined histograms were set so that the individual histograms were concatenated left to right in the order red, green and blue. A further sub sampling was also performed in order to evaluate spatial differences in color. The subsampling stratifies each cookie image into specified number of annuluses and a center region (Fig. 2). The range of subdivisions go from 1 (the entire cookie surface), 2 (an annulus containing the border and a center region), up to 6 sub regions. For each sub-sampled region the color channel histograms were made, and then for each cookie the respective region histograms were appended from left to right by the outer most regions into the center in a matrix. Fig. 3 shows an example of the concatenated histograms of the red color from the picture of a cookie.

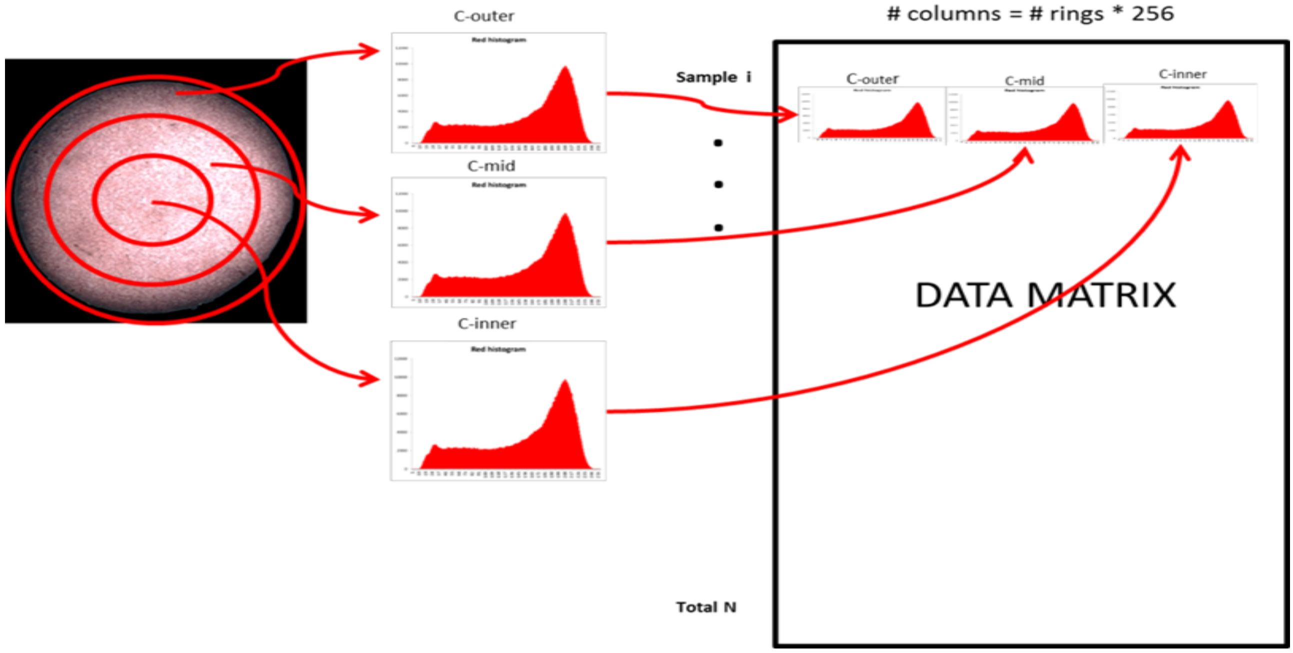

Figure 2 shows the stratification by subdivision of a cookie image and the construction of the data matrix by concatenating the histograms from left to right by the outer most to the inner regions.

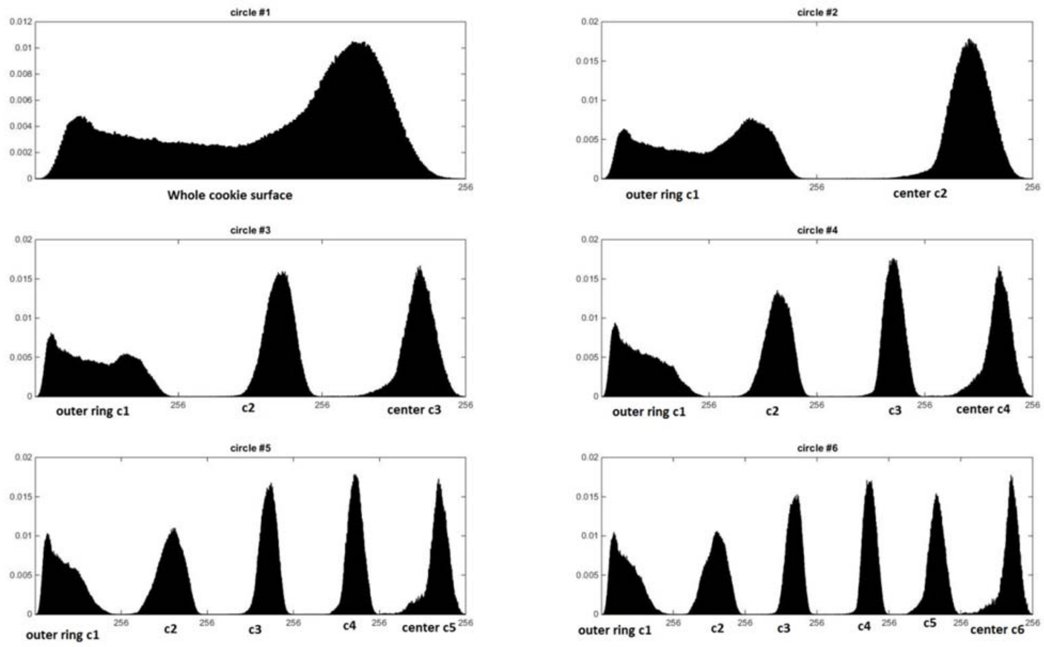

Figure 3 histograms of the red color from an example picture of a cookie. The circle number represents the number of subdivisions made.

2.5 Data analysis

The morphological data were analyzed using both normal scatter diagrams and Principal Component Analysis (PCA) (Hotelling, 1933). For the PCA model the morphological data were normalized. The RGB histogram analysis was performed using ANOVA Simultaneous Component Analysis (Jansen et al. 2005). The Matlab script by the author: Gooitzen Zwanenburg (Biosystems Data Analysis group, University of Amsterdam, The Netherlands) was used with only minor modifications. The preprocessing of the RGB histogram was confided to stratification method of the image (mentioned in part 2.4), and auto-scaling. The ASCA model (Eq. 1) was based on main effects for air, baking sheet and position, an interaction term for air and baking sheet and a blocking factor for days.

$$\mathbf{X}_{obs} = 1 * \bar{\bar{x}}_{\dots m} + \mathbf{X}_{air} + \mathbf{X}_{baking\ sheet} + \mathbf{X}_{block\ (day)} + \mathbf{X}_{interaction} + \mathbf{X}_{position} + \mathbf{X}_{residual} \qquad \text{Eq. 1}$$

3 Results & Discussion

3.1 Morphology

Scatter diagrams of the morphological data of the images are presented in Fig. 4, with the samples colored according to baking sheet type. The scatter diagrams in Fig. 4 show clear grouping of the cookies due to the baking sheet type. The cookies baked on the normal sheet have a bigger surface area, seen by the red points grouped on the higher measures for axis lengths and area, than the two other sheet types. The perforated and chain link baking sheets give the same resulting shapes of the cookies. The interesting difference between the perforated and chain link baking sheet is the smaller spread of the points for the perforated baking sheet. This is evident in all of the subplots in Fig 4, which means that cookies produced using this baking sheet would be more similar to each other. It should be mentioned that this does not necessarily translate into a preferred shape as seen from the consumer’s point. The roundness of the cookies (eccentricity, 0 is a circle and 1 is a line) seems unaffected by the sheet type used.

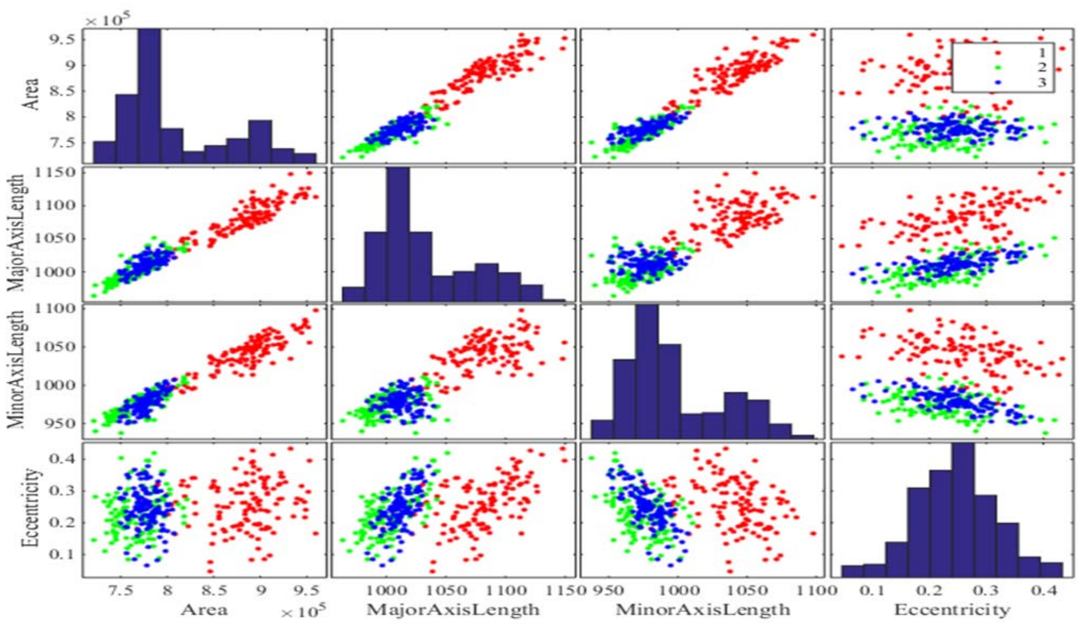

Figure 4 shows scatter plots of the shape measures with the points colored by the baking sheet used; 1 = steel, 2=chain-link and 3= punctured steel.

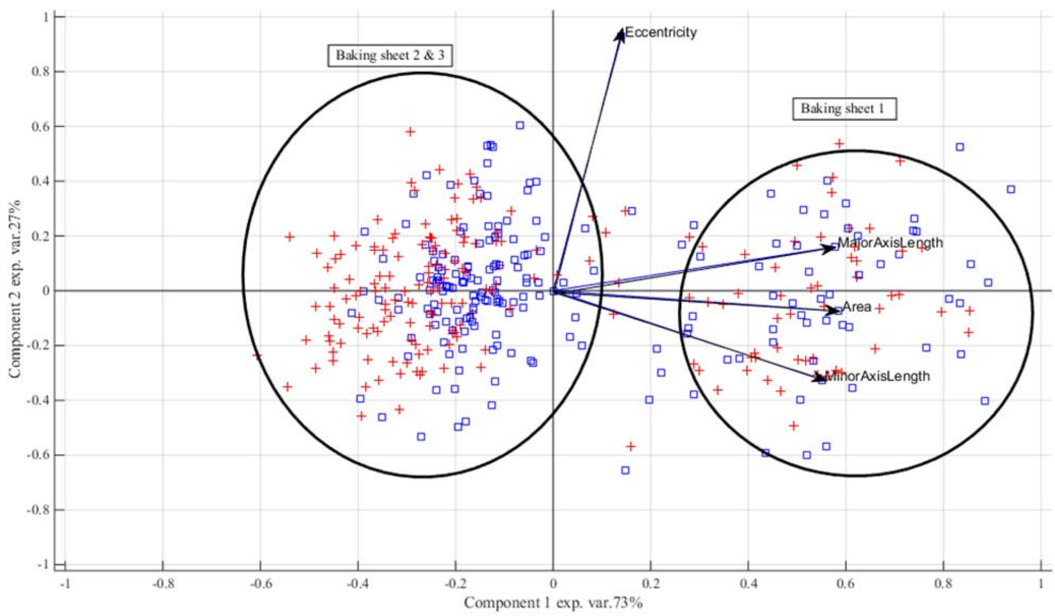

Figure 5 bi-plot of the PCA performed on the scaled shape data where the points are colored by air speed settings with blue is low and red is high air speed. The arrows show the loadings.

Fig. 5 shows a bi-plot of scores and loadings for PC1 versus PC2 from the PCA made on the morphological data. The samples are colored according to air speed settings. The plot shows the same tendencies as the scatter plots in Fig. 4. The perforated sheet and chain link sheet produce cookies with a smaller surface area than the normal baking sheet. Neither the air flow speed nor the type of baking sheet seems to influence the roundness of the cookies seen by the orthogonal placement of the eccentricity loading in the bi-plot. These results show that the perforated and the chain link baking sheets result in similar sizes of cookies. The reason is that the cookies can to some degree melt during the baking and seep into the chain link and the perforated sheets. The results show that the cookies would flatten out on normal baking sheet as would reasonably be expected.

3.2 ASCA

The ASCA model of the color differences between the cookies shows that air flow speed, baking sheet type, and the interaction between the two parameters have an effect on the color formation. In Figs. 6-7 the loadings and scores for PC 1 and PC 2 of the respective factors are investigated for the red channel and the concatenated RGB histograms shown when using the whole cookie surface as input. The score plots include both the mean effect score points and the back projected residuals and the explained variance is with respect to the mean effect scores. The effect due to changing airspeed is shown in both the score plots of the red channel and the RGB and is indicated by the grouping of the respective residuals close to their corresponding mean effect point and the distance between the mean points (Figs. 6 and 7). The residual overlap is very small between the two groups seen by the residual points colored by their corresponding factor level. This could indicate that the within group variation is smaller than the effect of changing the air speed which is shown by the distance between the mean effect points. To illustrate this, an example of the two cookies with the lowest and highest residual score values along PC 1 is shown in Fig. 8. The exemplified cookies show that the higher air speed gives lower red intensities across the surface compared to the low air speed cookie. The lower intensities for all three color channels red, green and blue correspond to surfaces having a darker appearance.

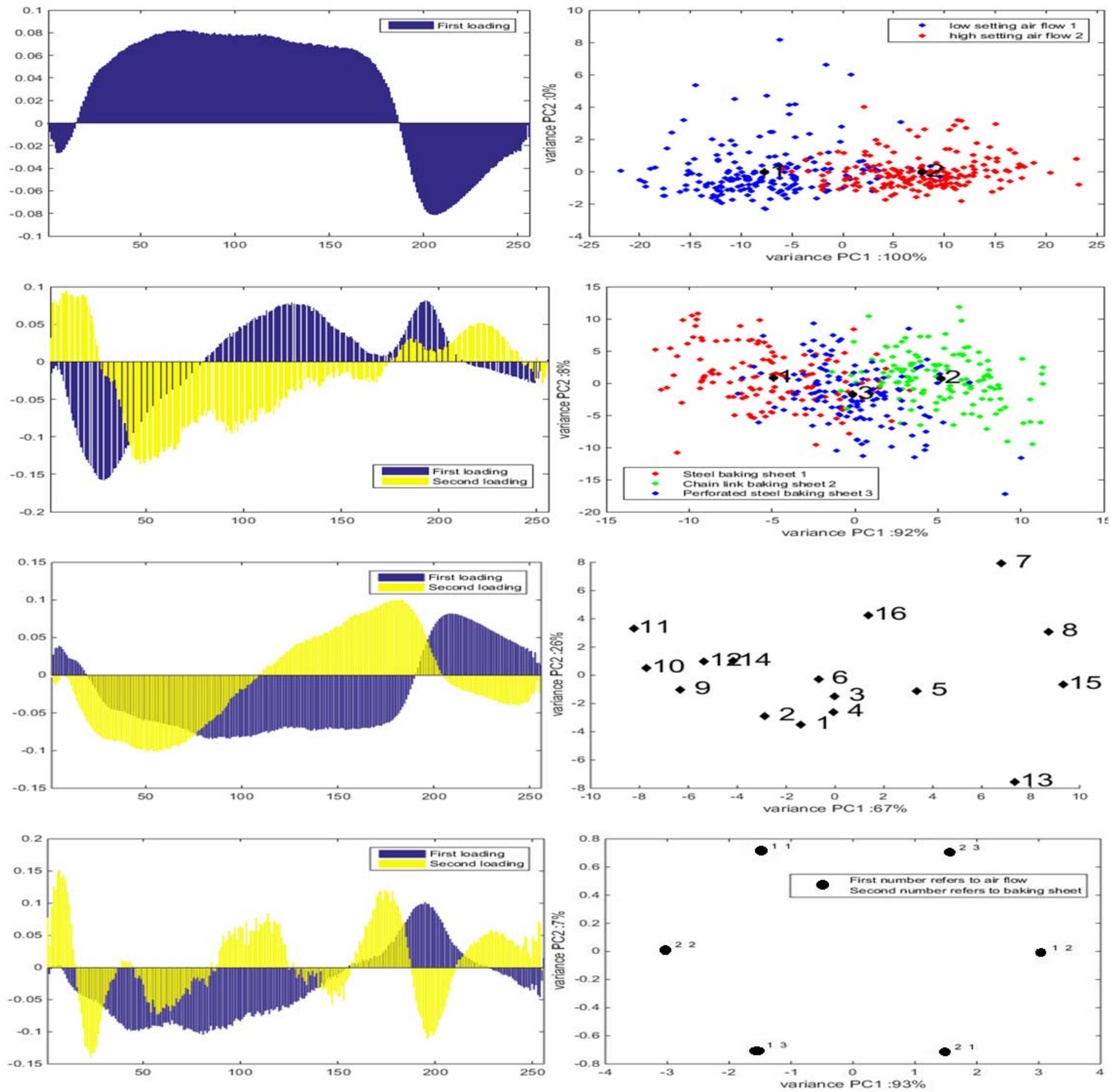

Figure 6 shows the loadings and scores plot for the ASCA model on the red color histogram of the entire cookie surface. The mean effect score values are indicated by the black points and the residuals are the colored and grouped by their respective factor level. From top to bottom the plots are paired with the first set for the air flow factor, then baking sheet, position and finally interaction between air flow and baking sheet.

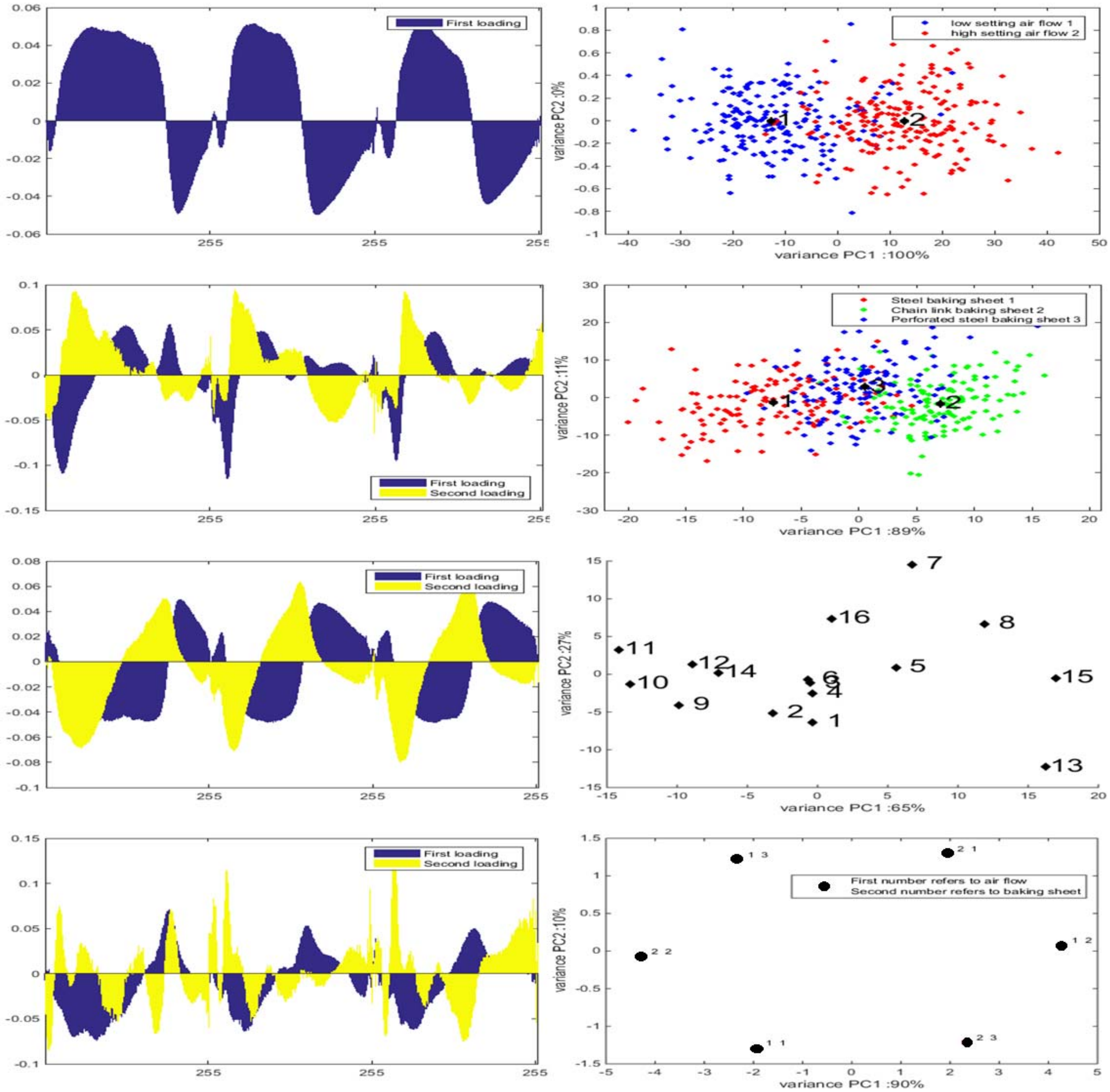

Figure 7 shows the analysis of the RGB histograms concatenated in the ordering of the name e.g. first the red histogram, then green and blue. The layout is the same as Fig. 6.

For the baking sheet factor the results show that the effect scores and residuals align with how the baking sheets were perforated. This can be seen by the location of the marked score values for the means in the scores plots for baking sheet along PC 1, going from left to right with normal sheet, perforated sheet and chain-link respectively in Figs. 6 and 7. Additionally, it can be seen that the interaction between air speed setting and baking sheet type influences the final color of the product.

This is evident from observing what happens along the first principal component of the interaction score plots in Fig. 6 and 7 where the chain link baking sheet is placed at opposite end, surrounding the points for both the normal and punctured baking sheets. Along PC 2 the normal and perforated baking sheets are paired by the low and high level air speed. This is seen by the high airspeed normal baking sheet and low air speed perforated baking sheet combinations having the same score value along PC 2. The chain link responses are only affected along PC1. As an example see Fig. 6 showing the red color histogram low level air speed combined with either the normal or punctured baking sheet will give a response more resembling the chain link sheet at the high air speed when looking at PC 1 axis. The interaction effect could be due to some of the cookies melting into the baking sheets, but a definitive conclusion is not possible.

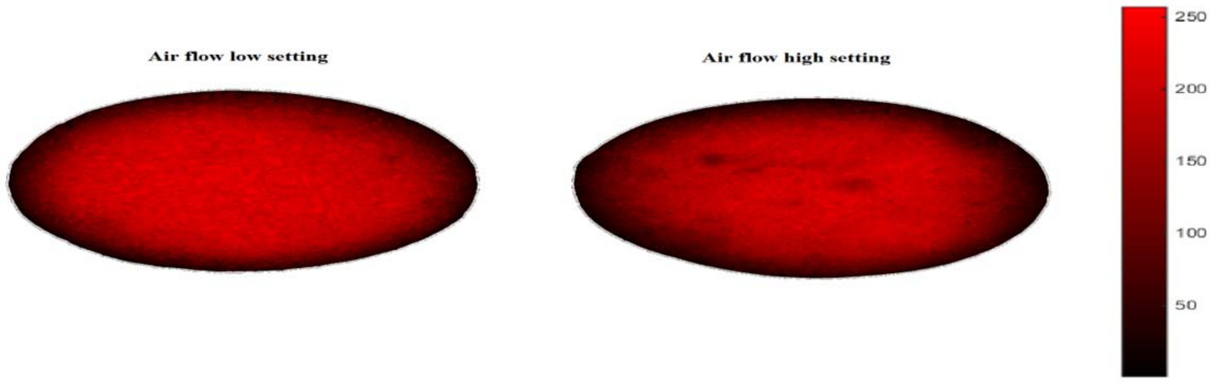

Figure 8 example of the red color channel from pictures of two cookies. The pictures show the higher air flow setting giving more low intensity red color across the surface compared to the low air flow setting.

Additionally, a positional grouping of the cookies can be seen where the cookies closer to the oven lid (7, 8, and 15) are more closely associated (Figs. 6 and 7) or distanced from the rest of the points. The positional difference is related to the frequency of high intensity colors compared to the lower intensity colors. This could roughly be translated to an overall brighter cookie. The positions 7, 8 and 15 (Table 1) on the baking sheet are closer to the oven lid, which could possibly have resulted in a draft that would change the impinging air flow compared to positions 1, 2 and 9, 10. If this explanation is allowed to be extrapolated a little, it could also explain the overall trend seen for the positional points where the points closer to the back of the oven are also correspondingly closer to each other in score points as seen in Fig. 6 and 7 compared to the points closer to the oven lid.

3.3 Image stratification

By subdividing the cookies into circular segments, the spatial correlation of color with the different factors is captured (Figs. 9-12). Fig. 3 shows an example of the stratification by subdividing and appending the regional histograms of a cookie. In the loadings plots for the factors air speed (Fig. 9), baking sheet (Figs. 10 and 11) and the interaction (Fig. 12), the subdivision of the cookies shows that the effects due to the respective factor changes is confined to different spatial regions. The air speed level influences the region between border and center seen in higher loadings for the corresponding regions especially evident with the increasing number of subdivisions used (Fig. 9). The loadings plots in Fig. 10 show that by applying the subdivision of the cookies, the effects of the baking sheet type are mainly confined to the border region of the cookie as seen by the higher loadings in the left most part of the plots. The loadings of the second component seem to only capture noise since no clear pattern is seen in the evolution of the sub plots (Fig. 11). This is further discussed later.

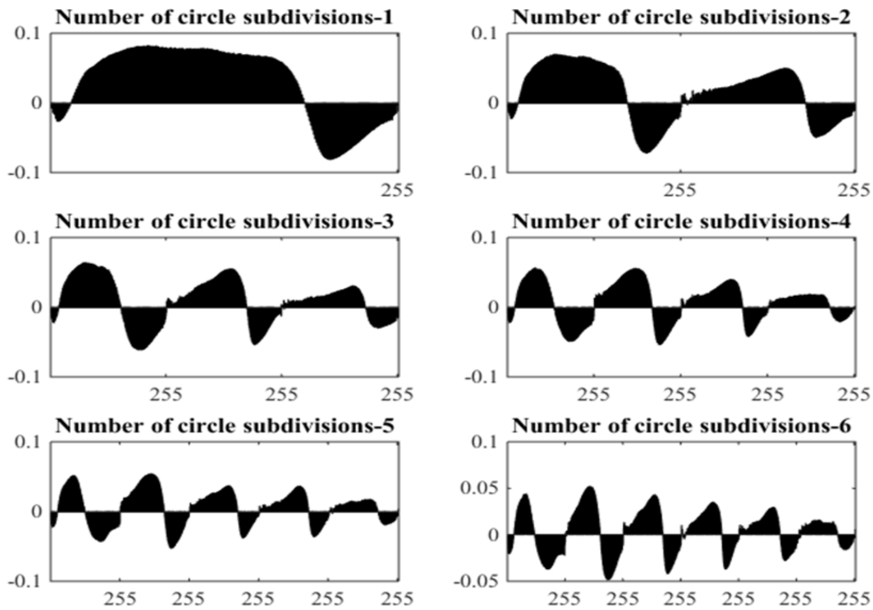

Figure 9 plots of the loadings of PC1 for the air flow factor of red color histograms. The plots show how the stratification of the cookie image into sub regions influences the loading weights. The order of from left to right on the horizontal axis is correspondingly from the border region to center of the cookie. The plots show that the center region has progressively smaller influence on the color differences due to air flow.

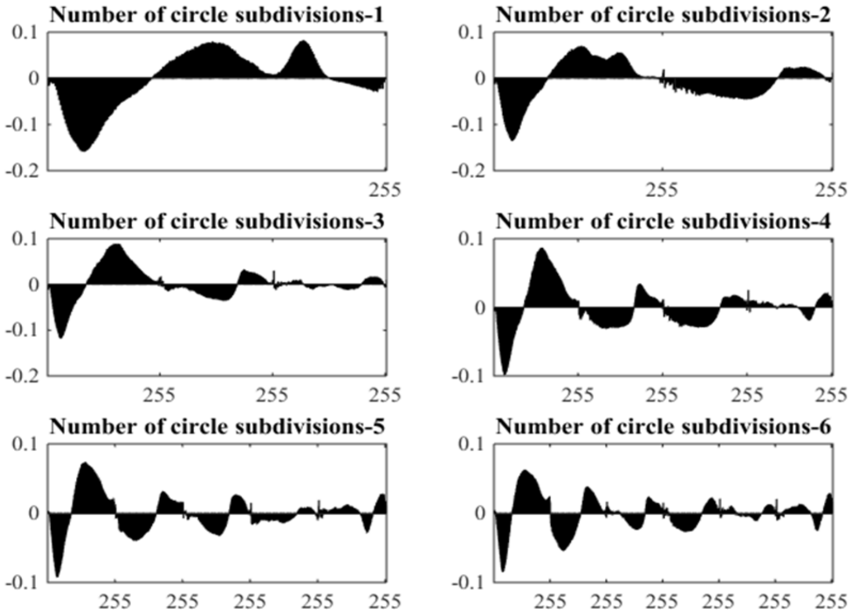

Figure 10 plots of the loadings of PC1 for the baking sheet factor. The layout is the same as Fig. 9. with the ordering of the regions towards the center of the cookie. The plots show that the outer most ring or subdivision is the defining region for the influence of baking sheet on color differences.

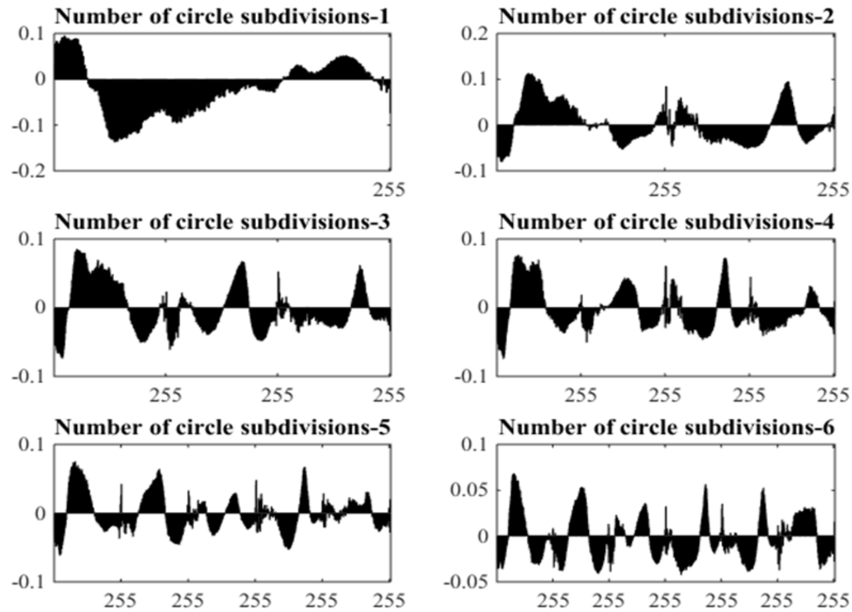

Figure 11 plots of the loadings of PC2 for the baking sheet factor. The layout is the same as Fig. 9. The plots show that the outer most ring or subdivision is the defining region for the influence of baking sheet on color differences.

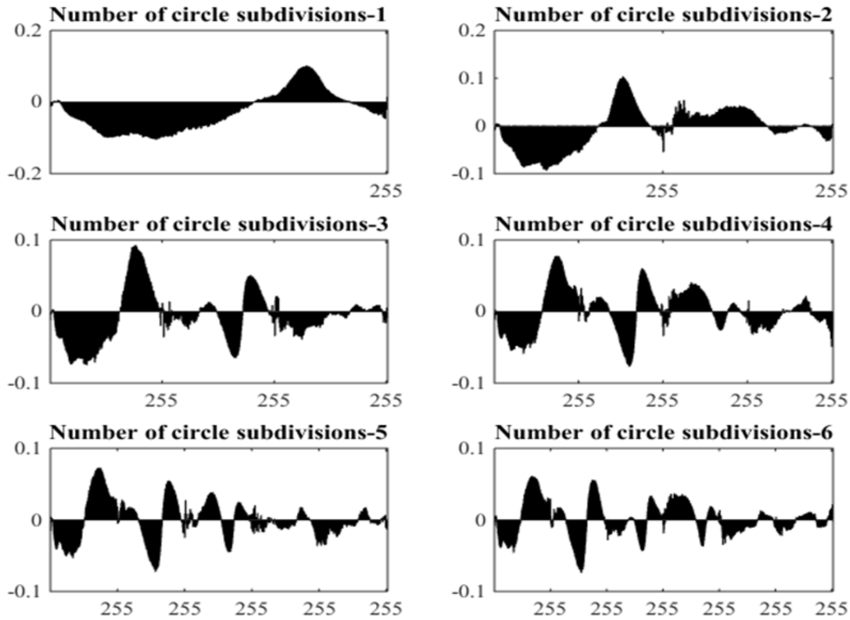

Figure 12 plots of the loadings of PC1 for the interaction between baking sheet and air flow. The layout is the same as Fig. 9. The plots show the border regions influence the differences in color by the trend of higher weights in the left most of the plots.

The center regions of the cookies consistently get lower loadings as the numbers of sub regions are increased across all the color channels (results not shown). This trend is seen for green and blue color histograms assessed individually (as exemplified with the red color histogram) or when combined in the RGB concatenated histogram analysis. From the overall results of stratifying the cookie image, it can be seen that the mean effect score increases with the number of subdivisions.

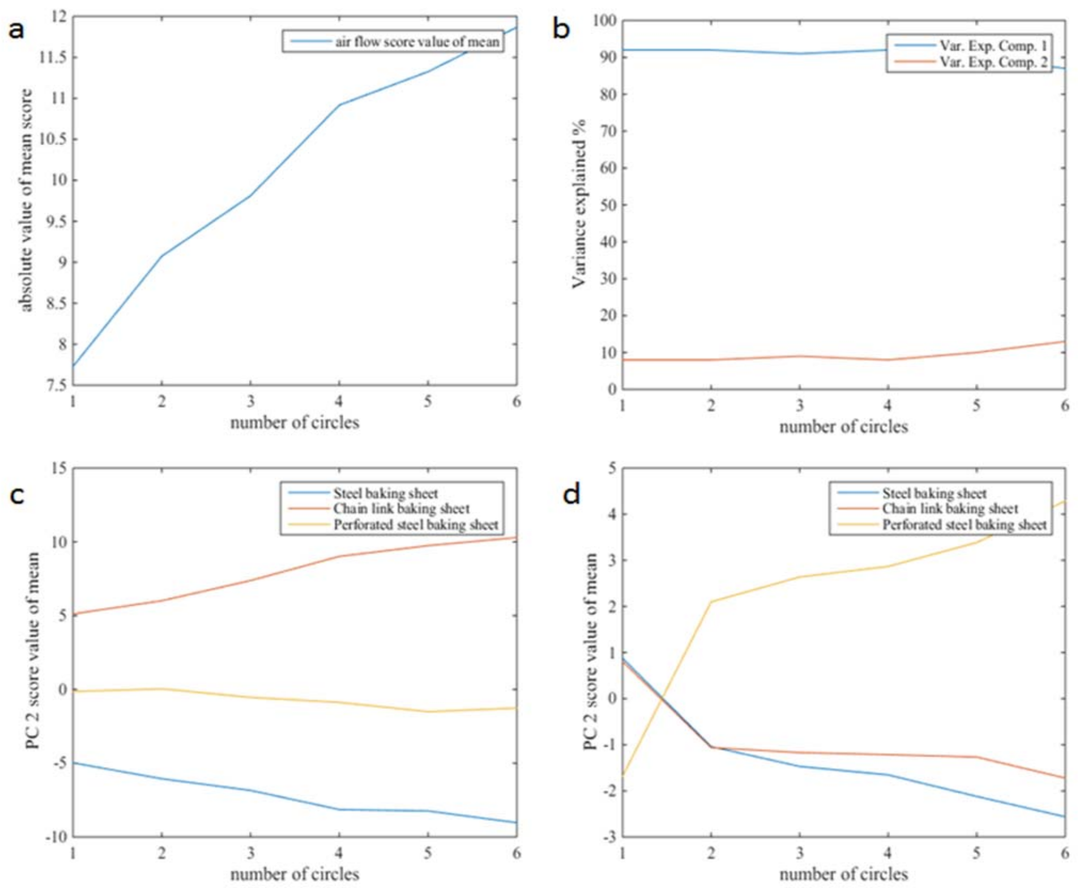

Figure 13 a, shows the score value of the mean response for air flow for the concatenated RGB analysis. b, variance explained for comp 1 and comp 2 for the baking sheet factors. c, PC1 mean score values for baking sheet factor. d, PC2 mean score values for baking sheet factor.

In Fig. 13 (a), the effect from the change of airspeed increases in score value with the increase of number of subdivisions used. The increase of the air effect score values tapers off at higher numbers of subdivisions. This is explained by the loadings for the outer rings of the cookie, which seem to be the distinguishing feature. The values of the loadings across the inner regions show similar pattern as the number of circles used goes up in Fig. 9. For the baking sheet the information is reserved for the two outer most rings i.e. the edge of the cookie and the ring immediately following (Fig. 13b, c, d). The loadings for these sections are especially high in the lower intensities of the histogram indicating that the effect of the baking sheet is confided to the darkness of the cookie edges. The plot of the explained variance of the components and of the second component (PC2) effect mean scores (Fig. 13c, d) distinguishes the perforated baking sheet from the other two. The differentiation of the baking sheets along the second component is small compared to the first component when considering the explained variance being below 20% for all the sub divisions.

3.4 General results

The overall analysis shows that the combination of the ASCA method and the stratification helps in identifying how factors could influence the spatial color distribution. Although the method is still qualitative, the results demonstrate its usefulness in designing or optimization of unit operations at least when surface appearance is of interest. The results show that the combination of the experimental design and the sub sampling make it possible to indicate which factors influence the color distribution and to give an indication of difference across the baking sheet.

The results from the ASCA analysis of the cookie images (sec. 3.2 and 3.3) show that the spatial color formation is highly dependent on design choices disregarding temperature. These results match the findings of Zareifard et al. (2009) where the convective component of the baking process was varied. The overall darkening of the surface with higher convection which corresponds to the results of the high air speed can be seen in both cases.

The combination of morphological parameters and color can give valuable information with regards to process design. The results from the morphological analysis gave information about choice of baking sheet and the color analysis showed which areas are affected by the different combinations of sheet designs and flow conditions.

4 Perspectives

The utilization of PCA for analysis of multivariate responses already has a long history of insightful results. The results show that digital image information can be used as an experimental response for studying the connections between process and product. The use of image for processing monitoring is a fast growing field with many documented applications already. It is hoped that this paper sparks interest in coupling image data with experimental design, which is still a new field, and in general the coupling of multivariate data analysis and experimental design. The coupling could help to provide a tool for increasing the understanding of color formation and process conditions. This paper demonstrates that the analysis method gives indications as to which process factor influences the color and also the region of the cookie. This information is invaluable for both the design and the operation of new process equipment, for improving the control of product appearances, and also in the diagnostics of process faults.

Acknowledgements

This work was performed within the research platform inSPIRe. The financing of the work by The Danish Council for Strategic Research and The Danish Council for Technology and Innovation is gratefully acknowledged. A special thanks to Dr José Amigo Rubio, University of Copenhagen for introducing the ASCA technique.

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Example 4: Discussion

The focus is on the analysis of the experiment and the new type of response for use in investigating of process variables. In honesty, not much consideration was made towards the design structure used with respect to blocking of mixed level experiment. This was simply due to it being an unknown issue. There were three types of baking sheet which could be tested. The air speed was chosen due to considerations mentioned in the article concerning the interest in wanting to change convective conditions in the oven chamber. The choice of 2 levels for the air speed was made so that all treatment combinations (i.e. all combinations of baking sheet and air speed) could be tested within a day. It is worth highlighting that it is the same oven used in Report I thus it was known that the cooling in between experimental runs in order to reset would take time. Thus it was expected that 6 experimental runs could be made in one day. The approach in the design of the experiment followed an intuition based approach, if however we strictly followed the recommend procedure for mixed level factorial the number of experimental runs needed would have been 8 per day (Montgomery 2009a).

The choice of 4 replications was a combined mix of curiosity and symmetry. The schedules for the preparation of the dough, the experimentation day and the day of measurements meant that 2 sets of experiment days (blocks) would require a week. It was therefore chosen to extend it with 2 more replications or blocks. So the experiments would span 2 weeks. There were several other measurements not shown such as texture and mass loss of the cookies. It was in the mass loss that the difference could be seen between the weeks matching with the metrological data for the period. Although it was not formally possible to test such effects the graphical analysis of the day blocks did show the mentioned trends. The example proposes the opportunities in combining the DoE concepts with the exploratory data analysis techniques, which help in the stratification of the results into the known groupings used in the construction of the design.

The ASCA method is one of the "generalized" newer methods under development applicable for analysis of experimental designs with multivariate data analysis tools (Smilde et al. 2008). The steps in the ASCA method are: *firstly* use the design structure for the orthogonal decomposition of the data set into matrices pertaining to each factor of the design. Each such factor matrices are characterized by column wise means for each of the levels of the respective factor. After all of the factor mean matrices are computed the residuals are computed by the subtraction of the original data set with each of the mean matrices. *In the second step*, now that all the experimental factors have their respective mean matrices, principal components are computed for each of them individually and analyzed by their scores and loadings plots. The scores are represented by a point for each level of a factor so for a 2 level factor there will be 2 score points for each level mean. The loadings show the maximal variation across all the column wise means which is the subspace of multidimensional effect. *The third step* is to plot and interpret the scores and loadings plots for each design factor, with the residuals projected into each scores subspace for the factors. The analysis is graphical and compromises both the study of the individual scores with residuals and loadings plots and the overall assessment of all the plots collectively. The scores plot for each factor with residuals projected into the principal subspace allows the user to graphically determine whether the factor and the level changes of the factor gives an impact. The scores plot is a between and within group analysis where the spread of the residuals for each group (within) is compared against the spread of the mean effect score points (between).

The loadings plot gives a diagnostics plot of which of the measured variables define the maximal spread of the factor means. Thus, loadings with the highest absolute weighting are where the respective factor is most clearly distinguished in its level changes. Extrapolating on this, it also possibly indicates which responses are affected by the factor.

The analysis can be summarized in two main equations eq. 3.1 and eq. 3.4. Firstly, the steps from eq. 3.1 to eq. 3.4 are shown. In eq. 3.1 using the example's experiment, the matrix is decomposed into each factors level mean matrix.

$$\mathbf{X}_{obs} = 1 * \bar{\bar{x}}_{....m} + \mathbf{X}_{air} + \mathbf{X}_{baking\ sheet} + \mathbf{X}_{block\ (day)} + \mathbf{X}_{interaction} + \mathbf{X}_{position} + \mathbf{E}_{residual} \quad (3.1)$$

For each of these factor matrices a PCA is performed, as an example the air factor ($\mathbf{X}_{air}$) in eq. 3.2 is performed a PCA on:

$$\mathbf{X}_{air} = \mathbf{TP^T}_{air} \qquad (3.2)$$

The loadings in eq. 3.2 is used in the projection of the residuals with the effect scores which gives the following equation eq. 3.3

$$\mathbf{Y}_{air} = (\mathbf{X}_{air} + \mathbf{E})\mathbf{P^T}_{air} = \mathbf{T} + \mathbf{EP^T}_{air} \quad (3.3)$$

$\mathbf{Y}_{air}$, containing the projected residuals, is used for the graphical analysis of scores. The crucial detail is that it is in eq. 3.2 where the loadings have been defined by the maximum variation in the effect means matrix. The residuals do not contribute to this, and are first used in eq. 3.3 for the visual analysis. These steps are repeated for each effect matrix in the model, which can be summarized into the overall equation of the analysis eq. 3.4.

$$\mathbf{X}_{obs} = 1 * \bar{\bar{x}}_{....m} + \mathbf{TP^T}_{air} + \mathbf{TP^T}_{baking\ sheet} + \mathbf{X}_{block\ (day)} + \mathbf{TP^T}_{interaction} + \mathbf{TP^T}_{position} + \mathbf{E}_{residual} \quad (3.4)$$

Another method which could have been used instead is the ANOVA-PCA (Sarembaud et al. 2007; Zwanenburg et al. 2011; Harrington et al. 2005). The difference between ANOVA-PCA and ASCA is in the step involving eq. 3.2. ANOVA-PCA proposes that the PCA should be made on each means matrix plus the residuals (eq. 3.5), where in the steps shown for ASCA the loadings are fitted on the means matrix alone.

$$\mathbf{X}_{air} + \mathbf{E} = \widetilde{\mathbf{T}}\widetilde{\mathbf{P}}^{\mathbf{T}}{}_{air} \qquad (3.5)$$

The difference will be in whether the within group variation should be included directly when fitting the loadings. The reason for using the ASCA method is in the proposed discriminating power compared to ANOVA-PCA, although it should be mentioned that some concerns have been raised on ASCA possibly overfitting (Zwanenburg et al. 2011). The proposition is also that ASCA fits the loadings on the between variation only, where in ANOVA-PCA the loadings are fitted to both between and within variation.

As shown in the example the exploratory analysis approach combined with DoE shows promising possible applications and developments for studying more complex problems. The issues are as always balancing the complexities of the analysis with the clarity in conclusions.

Discussion

The main objective of this thesis was to frame the use of statistically designed experiments for the use of improving food quality. The food engineer's toolbox should contain a plethora of tools and techniques to address the diverse problems in the food industry. The narrative of this thesis proposes that Design of Experiments (DoE) gives a valuable tool for learning about problems. DoE is relevant for all scales of experiments from lab to production, especially due to the developments within Quality Engineering (QE) using DoE for industrial process and production improvement. The learning combined with empirical and mechanistic understanding of problems is the important first quality improvement step.

The problems of the food engineer often pertain to quality of food either directly or indirectly. The complexity in the relationship of understanding all the intricate kinetics of intrinsic quality parameters is difficult. Coupling the intrinsic quality with how it will affect the extrinsic quality or vice versa adds to the difficulty. One of the issues seen has been the proposed dived between empirical modelling and physical based modelling or mechanistic understanding. The thesis proposes that this dived is nonsensical and that the approaches are part of the same overall strategy especially when issues of combined intrinsic and extrinsic quality attributes are tackled.

The experimental approach

The process of combining the mechanistic and empirical approach is abductive reasoning. Abductive reasoning can roughly be understood as the parallel process between deductive and inductive reasoning. The abductive reasoning refers to inference of simplest explanation. The reason for addressing the concepts of induction, deduction and abduction are that they form conceptual frameworks for the scientific process which is part of engineering. The understanding of: how problems are formulated, how solutions are proposed, and how decisions are made from these solutions influences the proposed strategies for designing experiments (Box et al. 2005) for formulating and solving physical models (Hadiyanto 2007) and how engineers can combine these frameworks (Box 2001). The work and environment of this thesis is on the applied version based on recognition or interpretation of the collaborative companies' situations and resources. As previously mentioned, the varying demands of the food engineer possess constraints on how complex issues can be formulated. A rough description of the approach which has been the "mental framework" is as follows: Based on physical understanding of the systems typically described by physically based models or robust engineering models, a mental model is formed. This mental model gives condensed or approximate understanding of the system. The mental process is then to imagine how the system could work and from there which responses could give the most knowledge. The experimental results could then confirm a concept or it could give a new quantitative description of phenomena as in Paper B. As examples are the shrimp experiments (Paper A) and the oven heat transfer experiment (Report I). In the shrimp experiments, mental models on how ohmic heating should in theory work influenced the research questions proposed. As mentioned in the paper (Paper A) the physical understanding of the situation says that within certain regions of experimental factors the temperature rise should be reasonably linear and more importantly volumetric. The experiments had the intention of verifying some of the existing theory and from there include the effects of the unit operation on the quality i.e. weight loss, press juice and texture. This reflects the approach of understanding the intrinsic quality attributes of the shrimp (weight loss and heating method) and trying to relate it to the extrinsic quality attributes (texture). The oven experiment was based on an understanding of the convective heat transfer process and the factors affecting it. The experiments were formulated so to include factors which were reasonably expected to affect the air flow but also to include variables which were not expected to influence the system directly. If, these indirect variables were insignificant, then by the projective properties of the design it could be projected down into fewer factors making full factorial design possibly with replication. Although this was not formally included in the design choices it was a known opportune byproduct of the design used. The understanding of the heat transfer provides insight of the possibilities of the setup for studying the quality of baked goods. The extension of this was shown in the cookie image example (Paper B). Here the knowledge of the heat transfer influenced the motivation for the experiment and from there the choice of factors. The study used images as the response requiring implementation of novel analysis procedures combing DoE and multivariate statistical techniques. This application using basic DoE principles converged with novel QE research in the using image data to answer basic FE questions. The future of image data was also found as an important application for QE research (Paper C).

The experimenting subject matter expert

The emphasis in the thesis was on the practical interpretation of the basic DoE concepts. Most of the interpretation took form through discussion of DoE application to different experimental situations. As has been discussed throughout the thesis the basic techniques are seldom used, reported and at times understood. This could result in an unfortunate expenditure in experimental efforts and possibly missing out on interesting effects. It can possibly be the reason for delineating empirical and mechanistic methods with Saguy (2015) emphasizing the value of the latter. This emphasis can to some degree also be understood when looking at the description of planning an experiment which is more complex than alluded to in Granato & de Araújo Calado (2014). Coleman & Montgomery (1993) describing the process of planning an experiment listed the possible bad consequences of the knowledge gap between statistician and experimenter where two points are worth reiterating in this discussion. The statistician's lack of physical understanding which therefore can result in not exploiting such knowledge. The second point, the experimenter's wrongful interpretation of previous experimental results. The examples from this thesis are when the statistician and the experimenter is the same person. The majority of the papers presenting the planning process does so from a team focus viewpoint (Viles et al. 2008; Freeman et al. 2013). This could be the reason for the problematic issues behind the apprehension towards statistical thinking and of DoE represented by Saguy (2015). The democratization of statistics means that more "non-statisticians" (other educational/technical background) function as the statistics experts. This binds back to the statement concerning the issue of the subject specialist and the statistician being the same person. The recommended approach is to present more documented discussions of applications as was noted by Tanco et al. (2009). The flow diagrams and team focus has major merit but the need for more examples of DoE cases discussed from the subject matter specialist view point are needed. This could help in progressing more directed cooperation with QE and statisticians. It is hoped that the presentation in this thesis can provide the food engineer with an appreciation of the DoE tools and from there be aware of the necessity in statistical thinking when addressing the broad issues of quality and quality improvement. As discussed in Part 1 the concept of quality lends itself to a statistical interpretation. The recognition of the complex relationship of the different quality attributes especially due to the transitionary state that food is in requires statistical thinking for quantifying these relationships (van Boekel 1996).

Experiments and simplicity

Instead of formalizing a mental processes model in a diagram of when the statistician and the engineer is the same person, a different presentation style was used. The method used here was to describe as many different scenarios and cases as possible together with a discussion of the choices. It is the author's opinion that providing examples of the methods in use is the best way to present the design process. It is not beneficial to say that one tool works for all problems. This is exemplified when looking at the difference between the experiment concerning the pilot plant oven (Report I) and the experimentation with the use of scrap dough (Report II). Both experiments concern unit operations within bakery industry and investigation of the process characteristics. The oven experiment had a simpler response compared to the dough experiment where several responses are needed to describe the material properties. Also the difference in the execution the experiments show how different situations the food engineer can experience within one industry sector. The dough experiments required mixing and sheeting and in general much more handling. The exposure to so many different types of process or production situations the needs for some basic experimental principles become crucial. Experimentation is a crucial part of learning which is a required activity in order to understand and improve food processes and from there food quality. It is not uncommon that the food engineer at the companies will also have to take the role as the quality engineer. Also typically the engineer does not have a team as such to corporate with. This places a certain need on flexibility of the methodologies at disposal. The engineer's task should be the ability to combine the tools in order to create new technologies and to solve problems. The presentation of process diagrams of the planning process has great value in the context of coordinating teams, designing experiments or more broadly solving problems. The intention with the format of the presentation is to give the user an inkling of how they themselves can start using DoE.

The focus on the basic principles was intended to provide a fair representation of experimental design so as others can implement the tools. The discussed statistical issue of the problems seen in food research (Granato et al. 2014; Nunes et al. 2015) was focused on the analysis which was also the case for the material provided on DoE (Granato & de Araújo Calado 2014). Here we have focused on the preceding steps before analysis; the actual process of designing an experiment. As presented by Box et al. (2005) experimental designs can be made which lends itself to simple interpretations. The simple designs and analyses are crucial in order to combine empirical results with mechanistic understanding to in turn provide feasible solutions to real problems.

Conclusion

It was found that DoE is a growing application within FE (FE). At the same time a divergence in the methodology is seen in FE with regards to empirical methods. The literature indicates that the issues in understanding statistical methods could be at fault. It was seen that the basic concepts of DoE are scarcely used. The main body of literature for food research has been more focused on the analysis of experiments than DoE. In this thesis I have tried to present basic principles concerning factorial experiment, randomization and blocking and applying the techniques. The focus of the presentation has been on the practical interpretations in order to show the relevance for other FE research applications. I also present the application of these principles within novel food research. The experimental designs used have been discussed and the choices presented, showing the various ways of addressing experimentation and research questions.

As was shown QE concepts are being used and reinterpreted for tackling food quality. The complexity of food quality demands various strategies which will be case dependent. One strategy is to define a process or product operating space which does not eliciting rejection by the consumer and then work within this space. This translates into focusing more on describing process and product interactions rather than actual formal optimization at least with respect to consumer preferences. The development of DoE especially with the contributions from QE is relevant for future FE problems. The techniques used by QE have been developed with special considerations towards designing experiments for industrial problems. The design of an experiment should consider complexity, time and resources. The applications within this thesis document some of many different approaches which can be taken within DoE. The iterative learning process is the common theme of all these approaches. In every case some mechanistic understanding has driven the experimental design or has been the focus of the design question. I hope that the documented applications will reframe the DoE and empirical methods, and stimulate the appreciation of the interconnectedness between empiricism and mechanistic knowledge when solving applied problems. Especially when trying to solve food quality problems the combination of tools is a necessity.

Perspectives

It is hoped with this presentation that the food engineer can appreciate what QE and DoE has to offer. By using a more informal route for presentation of the DoE concepts it is hoped that future users can be inspired to try some of these concepts. The importance of supplying "practical" examples cannot be overstated. These examples could also inspire researchers in QE or statisticians to try and tackle some of the issues present in FE. It is recognized that it is more feasible that food engineers are the instigators in starting such collaborations. In this dialog between food engineers and statisticians the presentation here can help in giving some examples of what practical choices are to be made in designing experiments. It is important that some mutual understanding of concepts is present. It is also important that the users of statistical tools start to give demands on the presentation of the tools. This also has implications to towards the consultancy industry. The proper teaching of DoE and guidance in applying it to the user's problems is of upmost importance. This will always present itself as recurring issues as new engineers are educated and new companies are started.

I also hope that it is shown how the "old basics" and the newer analysis tools can be combined for such issues as objective appearance assessment. During the course of the work for this thesis several advanced cases presented itself for study but the importance of supplying simple and tangible cases outweighed them. The future of FE is a promising ground for research in quality improvement. The growing field of applied mechanistic models presents itself also for applications in design of computer experiments. Also the well documented spread of chemometric techniques should be built on as a further gateway for introducing statistical thinking. Finally, the food engineers, as users of statistics, should take effort in trying to clarify their needs in order to improve the research and communication made by quality engineers and statisticians who should be held accountable in supporting proper understanding of DoE.

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Appendix

The following appendices are included:

Appendix 1: Recent Advances and Future Directions for Quality Engineering (Paper C)

Appendix 2: Studying fluid-to-particle heat transfer coefficients in vessel cooking processes using potatoes as measuring devices (Paper D)

Appendix 1: Paper C

Geoff Vining, Murat Kulahci and Søren Pedersen. (2015). Recent Advances and Future Directions for Quality Engineering. *Quality and Reliability Engineering International,* 32 (3) pp. 863-875

Abstract:

The origins of quality engineering are in manufacturing, where quality engineers apply basic statistical methodologies to improve the quality and productivity of products and processes. In the past decade, people have discovered that these methodologies are effective for improving almost any type of system or process, such as financial, health care, and supply chains.

This paper begins with a review of key advances and trends within quality engineering over the past decade. The second part uses the first part as a foundation to outline new application areas for the field. It also discusses how quality engineering needs to evolve in order to make significant contributions to these new areas

Keywords:

Statistics for complex systems; massive data sets; innovation; statistical thinking; statistical engineering

Appendix 2: Paper D

Aberham Hailu Feyissa, Martin Gram Christensen, Søren Juhl Pedersen, Minka Hickman, Jens Adler-Nissen. (2015). Studying fluid-to-particle heat transfer coefficients in vessel cooking processes using potatoes as measuring devices. *Journal of Food Engineering,* 163, pp. 71-78

Abstract:

This paper presents and demonstrates a novel idea of using spherical potatoes as a dispensable, cheap device for determining the fluid-to-particle heat transfer coefficient, hfp in vessel cooking processes. The transmission of heat through the potato can be traced by measuring the distance from the surface to the gelatinization front, which is easy to identify visually. Knowing this distance, the gelatinization temperature, the period of immersion, and the average radius of the potato, the heat transfer coefficient can be calculated. Either a numerical model based on the Finite Element Method (FEM) or an analytical solution of the Fourier equation can be applied for the calculation. The gelatinization temperature of the potatoes used was determined to be 67 °C by a direct temperature measurement and by visual inspection of the progression of the gelatinization front. A sensitivity analysis demonstrates that the method is rather precise at relevant values of hfp in vessel cooking (100–300 [W/m2 K]), allowing a prediction of the centre temperature within ±0.6 °C.

Keywords:

Heat transfer coefficient; Potato; Fluid-to-particle heat transfer; Gelatinization; FEM

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ISBN: 978-87-93109-83-4