La diffusion sélective lors de la déshydratation osmotique de la mangue: Impact de la solution hypertonique et des prétraitements

Thèse

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Résumé

La déshydratation osmotique est une technique de séchage partiel ayant lieu à de basses températures (moins de 50°C), permettant de préserver la qualité des produits comparativement au séchage conventionnel. De plus, une formulation du produit peut être réalisée en utilisant des solutions riches en composés bénéfiques, comme le sirop d'agave qui contient des prébiotiques (inuline). Elle est pratique pour conserver les fruits saisonniers comme la mangue dont les caractéristiques organoleptiques et nutritionnelles la placent parmi les fruits les plus consommés au monde. Cependant, l'imprégnation de solutés (sucres ou sels) augmente la teneur calorique des produits après la déshydratation osmotique. De plus le sucrose, le soluté le plus utilisé, n'est pas adapté à certains consommateurs qui y sont intolérants. Enfin, la teneur élevée en sucres dans les aliments est incriminée dans les maladies cardiovasculaires et l'obésité. C'est pourquoi, ce projet avait pour but d'optimiser le procédé de déshydratation osmotique de la mangue afin de produire des mangues déshydratées osmotiquement avec du sirop d'agave et ayant une teneur ajoutée en sucres réduite. Dans un premier temps, la viscosité et la rhéologie des solutions osmotiques de composition différentes ont été caractérisées, suivi de la déshydratation osmotique des morceaux de mangues Tommy Atkins d'épaisseurs 0.4 cm et 1.5 cm. Les résultats ont montré que l'augmentation de la viscosité, de la taille des molécules de solutés ainsi que de l'épaisseur de la mangue peuvent permettre de réduire le gain en sucres ajoutés tout en maintenant une quantité suffisante de perte en eau. En second lieu, des analyses par chromatographie liquide haute performance de la quantité et du profil en sucres individuels des mangues déshydratées dans les différentes solutions osmotiques, ont montré que la composition initiale de la mangue en différents sucres ainsi que la composition de la solution osmotique influencent le profil final en sucres. Une perte en sucrose et un gain en fructose et glucose ont été observés dans la mangue lorsque des solutions pauvres en sucrose ont été utilisées, permettant ainsi de moduler le profil final de sucres du produit. La présence d'inuline a été détectée dans la mangue après la déshydratation osmotique, ce composé prébiotique est bénéfique pour la flore intestinale et est une valeur ajoutée dans le produit final. Une analyse par microscopie électronique à balayage a permis d'observer le mode de dépôt des différents solutés sur la mangue au cours de la déshydratation osmotique, et ainsi déterminer les mécanismes par lesquels une réduction d'entrée de solides est possible. Finalement, des prétraitements de congélation/décongélation et de champ électrique pulsé ont permis de modifier la structure microscopique de la mangue avant de la soumettre à la déshydratation osmotique. Cette étape a montré que le type de prétraitement impacte l'effet sur le transfert de matières. La congélation/décongélation a augmenté le gain en sucres au détriment de la perte en eau, et l'effet du champ électrique pulsé (dans les écarts des variables utilisées dans cette

étude) était négligeable sur le transfert de matières en général. Cependant, l'utilisation de solutions osmotiques à viscosité élevée a permis de réduire le gain en sucres dans le cas des mangues dont la structure cellulaire a été sévèrement endommagée par la congélation/décongélation.

Cette thèse constitue une contribution dans la production de mangues déshydratées (et de fruits en général) ayant une teneur en sucres ajoutés réduite et des ingrédients fonctionnels tels que l'inuline qui est bénéfique pour l'organisme.

Abstract

Osmotic dehydration is a partial drying technique which necessitates low temperatures (less than 50°C), allowing product quality to be preserved compared to conventional drying. It allows product formulation throughout solutions rich in beneficial compounds, such as agave syrup which contains prebiotics (inulin). It is practical for preserving seasonal fruits such as mango which organoleptic and nutritional characteristics rank it among the most consumed fruits in the world. However, the impregnation of solutes (sugars or salts) increases the caloric content of products after osmotic dehydration. In addition, sucrose, the most used solute, is not suitable for certain consumers who are sucrose intolerant. Finally, the high content of sugars in food is incriminated in cardiovascular diseases and obesity. Therefore, this project aimed at optimizing mango osmotic dehydration process to produce osmotically dehydrated mangoes in agave syrup and with low sugar content. Firstly, viscosity and rheology of osmotic solutions of different compositions were characterized, followed by the osmotic dehydration of Tommy Atkins mangoes with thicknesses of 0.4 cm and 1.5 cm. The results showed that increasing solution viscosity, solutes molecules size, as well as mango thickness can reduce sugar gain while maintaining enough water loss. Secondly, high performance liquid chromatography results showed that initial composition of mango sugars as well as composition of osmotic solution influence the final sugar profiles of dehydrated mango. A loss in sucrose together with a gain in fructose and glucose have been reported in mango when osmotic solutions with low concentration of sucrose were used. Inulin was found in mango after osmotic dehydration in solutions containing inulin, this prebiotic compound is beneficial for gut microbiota and is therefore an added value in the final product. An analysis by scanning electron microscopy demonstrated the behavior of different solutes on the mango surface during osmotic dehydration allowing the understanding of the mechanisms by which solids gain could be reduced. Finally, freeze-thawing, and pulsed electric field pretreatments were applied to mango to modify its tissue structure before osmotic dehydration. Results indicated that the type of pretreatment impacts the mass transfer differently. Freeze-thawing increased sugar gain and negatively affects water loss, whereas pulsed electric field effect was negligible on mass transfer in general. However, high viscosity osmotic solutions reduced sugar gain for frozen-thawed mango. This thesis contributes to the research field of processed mangoes and in general, processed fruits, with low sugar content together with added functional ingredients such as inulin which is beneficial for the gut microbiota.

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US	Ultrasound
PEF	Pulsed electric field
FOS	Fructooligosaccharides
HPLC	High performance liquid chromatography
USDA	United States Department of Agriculture
TA	Titratable acidity
TSS	Total soluble solids
HP	High pressure
VI	Vacuum impregnation
G	Gain
L	Loss
DP	Degree of polymerization
ODE	Osmotic dehydration efficiency
LMP	Low-methoxyl pectinate
CMC	Carboxyl-methyl cellulose
SEM	Scanning electron microscopy
OS	Osmotic solution
E	Electric field strength
Z	Disintegration index
OD	Osmotic dehydration
N/A	Non applicable
CC25	Concentric cylinder 25
CC40	Concentric cylinder 40
CSS	Corn syrup solids
XG	Xanthan gum
AS	Agave syrup
I	Inulin
F-T	Freeze-thawing
L_0	Lenght
SG	Sugar gain
WL	Water loss
SGeq	Sugar gain at equilibrium
WLeq	Water loss at equilibrium
p	Probability value
RID	Refractive index detector
cv.	Cultivar

A ma mère

L'éducation vous libèrera et vous ouvrira la voie de l'indépendance

Compaoré Bibata

La plus belle récompense: les fruits de la force et la persévérance de notre travail

Thione Nyang

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Avant-propos

La présente thèse comporte cinq chapitres, dont 3 chapitres constituent des articles scientifiques publiés ou déjà soumis pour publication. J'ai proposé l'hypothèse et élaboré les protocoles expérimentaux, effectué les essais au laboratoire, réalisé les analyses statistiques des résultats, la rédaction et la correction de la thèse. La professeure Cristina Ratti, ma directrice de thèse, est l'initiatrice de ce projet et a effectué avec la contribution du professeur Seddik Khalloufi qui est mon co-directeur, la conception et la validation des protocoles expérimentaux, l'analyse et discussion des résultats, la correction et la révision des articles scientifiques et des autres parties de la thèse.

Le chapitre 1 représente une revue critique de la littérature sur la déshydratation osmotique et décrit les principes, les facteurs importants ainsi que les avancées dans la déshydratation osmotique des composés végétaux.

Le chapitre 2 regroupe la problématique de la recherche, les hypothèses et les objectifs généraux et spécifiques de cette thèse. Les trois autres chapitres suivants sont des articles scientifiques issus des résultats obtenus au cours des travaux de recherche réalisés pendant la thèse.

Le chapitre 3 intitulé « *Effect of viscosity and rheological behavior on selective mass transfer during osmotic dehydration of mango slices in natural syrups* » a été publié dans le *Journal of Food Processing Engineering* en mai 2021. Il rapporte les résultats de l'impact **de la composition et de la rhéologie de la solution osmotique ainsi que de l'épaisseur des tranches de mangue** sur la réduction de solides et le transfert de matières lors de la déshydratation osmotique des mangues.

Le chapitre 4 dont le titre est *« Sugar profiles modulation of mango during osmotic dehydration in agave syrup solutions »* présente l'effet des différentes solutions osmotiques sur le **profil en sucres individuels de la mangue déshydratée**. Il signale les possibles mécanismes par lesquels une réduction de gain de sucres est possible durant la déshydratation osmotique. Il a été soumis au *Journal of Food Science* en mars 2022 et a été révisé en août 2022 (en attente d'acceptation finale).

Le chapitre 5 intitulé « *Pulsed electric field and freeze-thawing pretreatments for solids uptake modulation during osmotic dehydration of mango* » s'intéresse à l'application de **technologies de prétraitement (congélation/décongélation et champ électrique pulsé)** sur le transfert de matières lors de la déshydratation osmotique de la mangue. Il a été soumis au Journal *Foods* en juillet 2022, et a été révisé et accepté en août 2022 (en attente de publication). Ce dernier article scientifique a été réalisé, de la conception aux expérimentations et révision, avec la collaboration du Professeur Sergey Mikhaylin qui a bien voulu me former et me permettre d'utiliser l'équipement du champ électrique pulsé au labo pilote du Département de Sciences des aliments (Université Laval).

Enfin, cette thèse se termine par les conclusions générales issues des résultats de cette thèse ainsi que les perspectives de travaux futurs sur la déshydratation osmotique de la mangue.

Introduction

La conservation des aliments est un défi constant pour l'alimentation humaine. En effet, les produits alimentaires à l'état frais particulièrement les fruits sont susceptibles à des dégradations de natures microbiologiques et biochimiques qui altèrent rapidement leurs qualités gustatives et hygiéniques les rendant impropres à la consommation. En effet, la plupart des aliments frais notamment les fruits tels que la mangue possèdent une teneur en eau supérieure à 80%. Cette eau disponible facilite les réactions enzymatiques de dégradation ainsi que la prolifération d'organismes microbiens telles que les bactéries, les champignons et moisissures.

Depuis longtemps, l'élimination de l'eau connue sous le nom de déshydratation est la méthode la plus utilisée pour prolonger la conservation des denrées alimentaires (Phisut, 2012). Le procédé de déshydratation le plus utilisé est celui du séchage conventionnel à air chaud qui nécessite des températures élevées souvent supérieures à 60°C (Shi & Xue, 2008). Toutefois les produits obtenus par ce type de séchage sont reconnus pour avoir une diminution de la qualité au niveau gustatif (goût, arôme), visuel (couleur), textural (fermeté) et nutritionnel (vitamines, antioxydants…) (Lazarides, 2001). Certains autres désavantages du séchage à air chaud sont la durée procédé, la consommation énergétique et le coût des équipements (Bchir et al. 2011) qui le rendent souvent peu accessibles aux pays à faibles revenus où la plupart des fruits tropicaux sont produits.

De plus en plus, les industriels de par le monde, recherchent des procédés peu coûteux en énergie et permettant d'obtenir des produits alimentaires déshydratés de qualités proches de celles des produits frais. Ainsi, des procédés utilisant des températures modérées (<50°C) sont maintenant recherchés afin de réduire les dommages thermiques qui sont associés à l'utilisation de hautes températures. Dans cette optique, la déshydratation osmotique, qui se déroule non seulement à température ambiante à modérée mais aussi en absence d'oxygène est une technique prometteuse.

La déshydratation osmotique consiste en l'élimination partielle de l'eau d'un tissu végétal ou animal par immersion dans une solution hypertonique de solutés (sucres ou de sels) (Shi & Xue, 2008). Elle se déroule à températures basses et ne nécessite pas de changement de

phase de l'eau (Tortoe, 2010). L'élimination de l'eau se fait du milieu le moins concentré en solutés (tissu cellulaire) vers le milieu le plus concentré (solution hypertonique) à travers la membrane semiperméable du tissu cellulaire de l'aliment, et elle est accompagnée d'un gain de solutés (Torregiani, 1993). La déshydratation osmotique permet d'obtenir des produits de meilleures qualités gustatives, visuelles et nutritionnelles. De plus elle permet d'économiser en énergie et de réduire le temps d'une déshydratation conventionnelle subséquente (Bchir et al. 2011). Elle intègre donc la plupart des critères recherchés par les industriels et les consommateurs pour des technologies de transformation alimentaire plus vertes, peu coûteuses et avec des produits finis de bonnes qualités. Cependant, la déshydratation osmotique a aussi ses limites. D'abord, c'est un procédé lent pour lequel la sortie d'eau s'accompagne d'une perte en nutriments solubles, quoique celle-ci est minime par rapport aux autres procédés de séchage, elle a un impact sur la qualité nutritionnelle du produit final. Aussi, le gain en sucres dans l'aliment soulève des critiques liées à la relation entre la consommation de sucre et les maladies cardiovasculaires et l'obésité.

De plus en plus, le sucre conventionnel ou sucrose est remplacé par d'autres types de solutés qui apportent moins de calories et/ou ayant des ingrédients fonctionnels reconnus pour avoir un impact positif sur la santé, c'est l'exemple des prébiotiques. Le sirop d'agave est une source naturelle de sucres qui contient des nutriments (minéraux, vitamines) ainsi que l'inuline et les fructo-oligosaccharides, qui sont des prébiotiques (Corrales Escobosa et al., 2014a). Dans la littérature, d'autres sirops naturels tels que le sirop d'érable et le miel ont été utilisés dans la déshydratation osmotique. Cependant avec le sirop d'agave, il n'y a pas selon nos recherches des travaux scientifiques disponibles. Des études ont été faites soit pour réduire le temps de déshydratation osmotique par accélération de la perte en eau, soit pour réduire le gain en sucres. En contrôlant les paramètres influençant la déshydratation osmotique tels que la concentration, le poids moléculaire du soluté, la température, la présence d'agitation et les prétraitements, plusieurs études ont pu augmenter la vitesse de déshydratation osmotique des fruits. Mais la réduction de la diffusion en sucres reste une problématique actuelle, car plusieurs études ont relevé l'augmentation de la perte en eau qui s'accompagne d'une élévation du gain en sucres (Rastogi et al., 2002).

Parmi les techniques les plus documentées pour contrôler la teneur en sucres ajoutés, le revêtement comestible avec des lipides, des polysaccharides ou de la résine, a permis de réduire le gain en sucres lors de la déshydratation osmotique (Matuska et al., 2006). Quoique le revêtement comestible ait donné de bons résultats sur la performance de la déshydratation osmotique (rapport élevé de perte en eau sur gain en sucres), c'est une méthode où il y a un ajout supplémentaire d'additifs aux fruits. Les consommateurs recherchent toutefois des produits avec le moins d'additifs possibles. Face à cette problématique, quel moyen peut être utilisé pour réduire le gain en sucres, réduire le temps de déshydratation osmotique et améliorer la qualité nutritionnelle et organoleptique des fruits déshydratés osmotiquement avec peu ou pas d'additifs? La synthèse de la littérature a permis d'identifier les facteurs importants pour moduler le transfert de matières lors de la déshydratation osmotique.

Ainsi, les propriétés du tissu végétal ainsi que les caractéristiques de la solution osmotique peuvent être optimisés pour contrôler le gain en sucres. Particulièrement, la composition en solutés et la viscosité de la solution osmotique ainsi que l'épaisseur du fruit pourraient être utilisées pour changer le contrôle (interne ou externe) de transfert de matières. En ce qui concerne la modification du tissu végétal, des prétraitements de modification de la structure cellulaire tels que le champ électrique pulsé, l'ultrason, les microondes, les hautes pressions hydrostatiques ont donné des résultats prometteurs en améliorant la qualité des produits obtenus (Ahmed et al., 2016). Ces méthodes modifient la structure cellulaire des tissus en créant des pores ou des canaux qui facilitent la sortie de l'eau. Cette propriété de modification du tissu végétal pourrait être exploitée pour contrôler la diffusion en sucres dans l'aliment au cours de la déshydratation osmotique. La mangue étant l'un des fruits les plus consommés au monde, avec une production mondiale supérieure à 80 millions de tonnes par an (Evans et al., 2017), pour éviter le gaspillage alimentaire, la déshydratation osmotique est un procédé qui pourrait contribuer à sa conservation tout au long de l'année tout en gardant le mieux possible ses qualités.

C'est dans ce cadre, que ce projet s'est intéressé d'une part à la déshydratation osmotique de la mangue dans le but de produire des mangues déshydratées osmotiquement avec une faible teneur en sucres ajoutées comparées à celles disponibles sur le marché. Et d'autre part, le projet vise également à utiliser le sirop d'agave pour améliorer les propriétés nutritionnelles

des mangues déshydratées produites. Les connaissances et les outils ainsi développés permettront aux industriels de produire des mangues et par extension des fruits déshydratés qui correspondent aux critères des consommateurs.

Chapter 1: Literature review

1.1 World production of mango

Mango (*Mangifera indica*) belongs to the family of *anacardiaceae* and it grows in tropical and subtropical regions (Nono et al., 2001; Zou et al., 2013) mainly in developing economies where it contributes to million people incomes (Mitra, 2016). It represents a fruit of great appeal worldwide due to its pleasant taste, aroma, flavor and nutritional components (Maldonado-Celis et al., 2019b). Thus, mango is equally popular in developed countries where it is consumed fresh or processed. In most countries, its production remains seasonal (Abano, 2016). Its production as illustrated in Figure 1.1 is growing constantly from 24.71 × 10^3 tons in 2000 to 42.66 × 10^3 tons in 2013 (Evans et al., 2017).

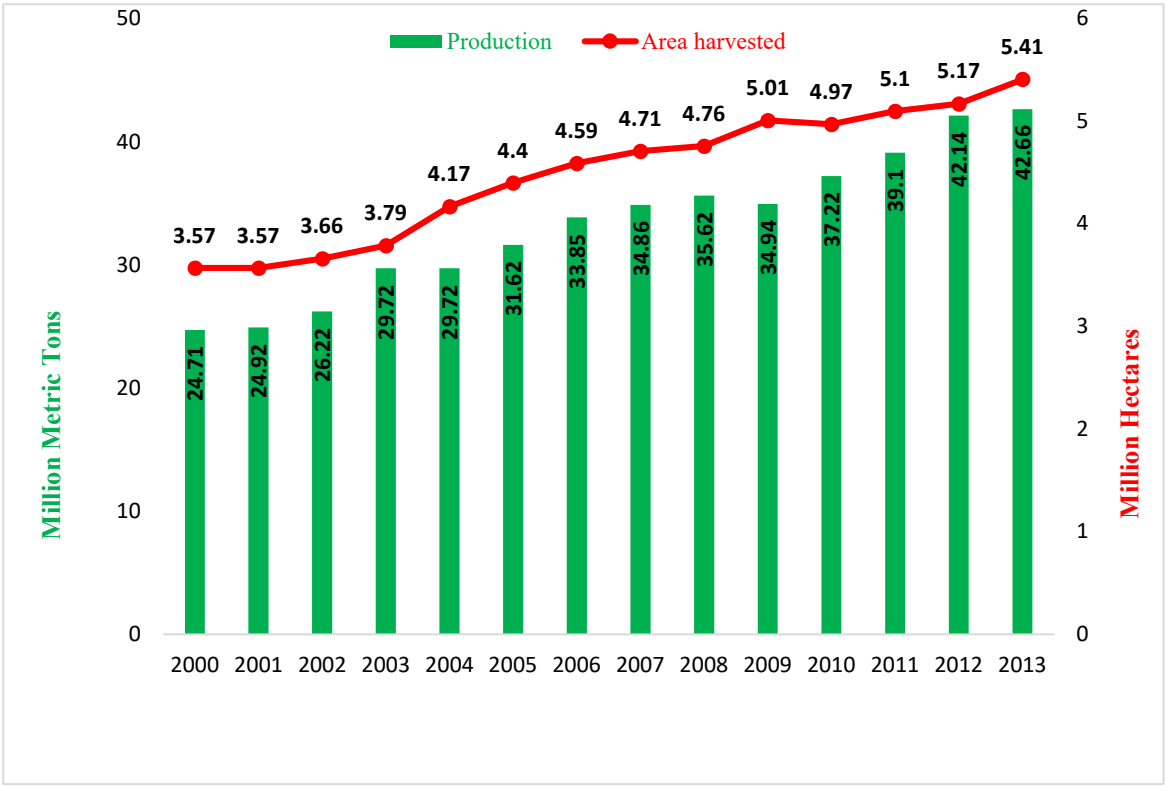

Figure 1.1: World mango production and area harvested (2000-2013), adapted from (Evans et al. 2017)

Mango is cultivated in more than 100 countries (Evans et al., 2017; Jahurul et al., 2015; Jiménez-Hernández et al., 2017a) with production shared as 77.17% in Asia, 12.2% in Americas, 10.50% in Africa and only 0.11% in Oceania (Evans et al., 2017). Thousands varieties are cultivated, among them Haden, Kent, Keit, Palmer and Tommy Atkins cultivars (Sanjinez-Argandoña et al., 2018) dominate the global export market (Sauco, 2017). Further increase in mango production is expected in future years due to the world market increasing demand and innovative agricultures techniques which favors planting in more diverse climates (Sauco, 2017).

1.2 Mango nutritional components

Mango average composition (American varieties) is presented in Table 1.1 adapted from (Maldonado-Celis et al., 2019b; Tharanathan et al., 2006). It highlights that mango is a source of nutrients such as carbohydrate, protein, lipid, minerals, and dietary fiber (pectin). It should be pointed out that nutrients proportion vary for different varieties due to climacteric conditions and agriculture processes (Evans et al., 2017).

Table 1.1: Mango composition*

Compound	Content (g per 100 g of fruit dry weight basis)
Water	78.9-82.8
Ashes	0.34-0.52
Total lipid	0.30-0.53
Total protein	0.36-0.40
Total carbohydrate	16.20-17.18
Total dietary fiber	0.85-1.06
Energy (Kcal)	62.1-190

*From (Maldonado-Celis et al., 2019b; Tharanathan et al., 2006)

1.2.1 Antioxidants and polyphenol

Polyphenols are found in mango pulp, peel, seed, kernel, leaves, stem bark and flower (Burton-Freeman et al., 2017; Masibo & He, 2008). Table 1.2 from Berardini et al. (2005), presents mango polyphenols distribution in different mango varieties in mg/kg dry matter. The major polyphenols are mangiferin, quercetin, isoquercetin, beta-glucogallen, gallic and ellagic acids (Berardini et al., 2005; Masibo & He, 2008). Common varieties found in American countries (i.e., Tommy Atkins, Kent, Haden, Heidi) contain mainly mangiferin, quercetin and rhamnetin. Most of these phytochemical compounds possess antioxidants properties which protect human cells against lipid peroxidation, DNA damage and many degenerative diseases (Masibo & He, 2008).

Table 1.2: Polyphenol content (mg/kg dry matter) in different varieties of mango*

Compound	Tommy Atkins	Manila	Kent	Jose	Mini mango	Haden	Heidi
Mangiferin	1263.2	43.5	13.9	983.6	449.9	11.2	108.9
Isomangiferin	40.3	11.5	4	45.5	13.3	21	8
Mangiferingallate	87.3	7.8	-	25.2	31.6	-	-
Isomangiferingallate	12.3	3	-	-	-	-	-
Quercetin-diglycosidol	55.1	145.9	-	40.3	-	1309.1	25.9
Quercetin-3-0-gal	1217.3	430.6	944.5	1467.7	1147.1	912.7	1275.7
Quercetin-3-0-glc	882	282.5	890	1045.3	767.8	179.1	814.5
Quercetin-3-0-xyl	239.5	39.2	150.7	278.6	10.2	104.9	225.7
Quercetin-3-0-arap	163.5	27.6	91.6	191.8	3.5	70.5	131.9
Quercetin-3-0-araf	152.4	17.9	84.8	119.6	-	52.7	123.5
Quercetin-3-0-rha	38.2	15.6	58.1	116.4	-	43.7	41.6
Kaempferol-3-0-glc	77.3	16.8	30.6	171.7	-	228.6	73
Rhamnetin 3-0-gal/glc	215.6	14.6	70.6	374.4	49.8	2.8	57.4
Quercetin	-	1.7	3.3		19.3	-	11.9

*From (Berardini et al., 2005)

1.2.2 Vitamins and Minerals

Tables 1.3 and 1.4 indicate mango vitamins and minerals profiles respectively. The main vitamin found in Table 1.3 is ascorbic acid, but mango also contains vitamin A and vitamin E among others. These compounds possess antioxidant properties that promote healthy immune function (Evans et al. 2017).

Table 1.3: Vitamin composition in 100 g of mango pulp*

Vitamin	Value per 100 g
Ascorbic acid (Vit. C)	13.2-92.8 mg
Thiamine (Vit. B1)	0.01-0.04 mg
Riboflavin (Vit. B2)	0.02-0.07 mg
Niacin (Vit. B3)	0.2-1.31 mg
Vitamin E (alpha tocopherol)	0.79-1.02 mg
Panthotenic acid (Vit. B5)	0.16-0.24 mg
Pyridoxine (Vit. B6)	0.05-0.16 mg
Folat total	20.69 ug
Vitamin A	54 ug
Vitamin K	4.24 ug

*From (Maldonado-Celis et al., 2019b)

Moreover, mango contains mineral compounds (Table 1.4) which offers medicinal and nutritional benefits. The main one is potassium followed by phosphorus, magnesium and calcium. Their roles are important for the body functionality and are related to blood, muscles, bones, DNA and enzymatic systems among others (Bhutto et al., 2005).

Table 1.4: Mineral composition in 100 g of mango pulp *

Minerals	Value (mg) per 100 g
Calcium	7-16
Iron	0.09-0.41
Magnesium	8-19
Phosphorus	10-18
Potassium	120-211
Sodium	0-3
Zinc	0.06-0.15
Cooper	0.04-0.32
Manganese	0.03-0.12
Selenium	0-0.6

*From (Maldonado-Celis et al., 2019b)

1.2.3 Carbohydrates

Mango carbohydrate composition evolves through the ripening process (Maldonado-Celis et al., 2019b) from complex starch to monosaccharides (glucose, fructose) and disaccharide (sucrose) (Bello-Pérez et al., 2007). During ripening, starch is hydrolyzed into glucose (Derese, 2017), through phosphorylation, subsequently glucose contributes to sucrose synthesis and degradation (Geigenberger & Stitt, 1991) leading to modulation of sucrose, glucose, and fructose content as shown for kiwi by Moscatello et al. (2011). Ripening increases mango total sugar content which may vary according to cultivars. American varieties (Tommy Atkins, Haden, Kent, Keitt) reportedly has a content of sugar per 100 g of fruit distributed as, 14.98 g of total carbohydrates, 13.66 g of sugars (6.97 g of sucrose; 4.68 g of fructose; 2.01 g of glucose) and 1.6 g of dietary fiber (USDA, 2018). Overall, in majority of cultivars, mango predominant sugars are sucrose, fructose and glucose in decreasing order (Bello-Pérez et al., 2007).

1.3 Health benefits and medicinal use

As shown above, mango is a good source of vitamins, minerals, dietary fibers, carbohydrates and phytochemical components which are beneficial for human health in term of energy, growth and maintenance (Dar et al., 2016). A wide range of research was conducted for mango consumption regarding health benefits related to diverse diseases illustrated in Figure 1.2: cancer, diabetes, obesity, cardiac, brain, skin and intestinal diseases (Burton-Freeman et al., 2017). Research conducted on animals (*in vitro* and *in vivo*) has proven mango anti-inflammatory and anti-oxidative properties (Ornelas-Paz et al., 2010). In human studies, glycemic control, probably related to insulin action and/or glycogen synthesis was shown (Burton-Freeman et al., 2017). Positive effect of mango consumption include benefits for the gut microbiota, increase of cutaneous flow, providing antioxidants to the skin to strengthen collagen, an important component of the epidermis (Burton-Freeman et al., 2017).

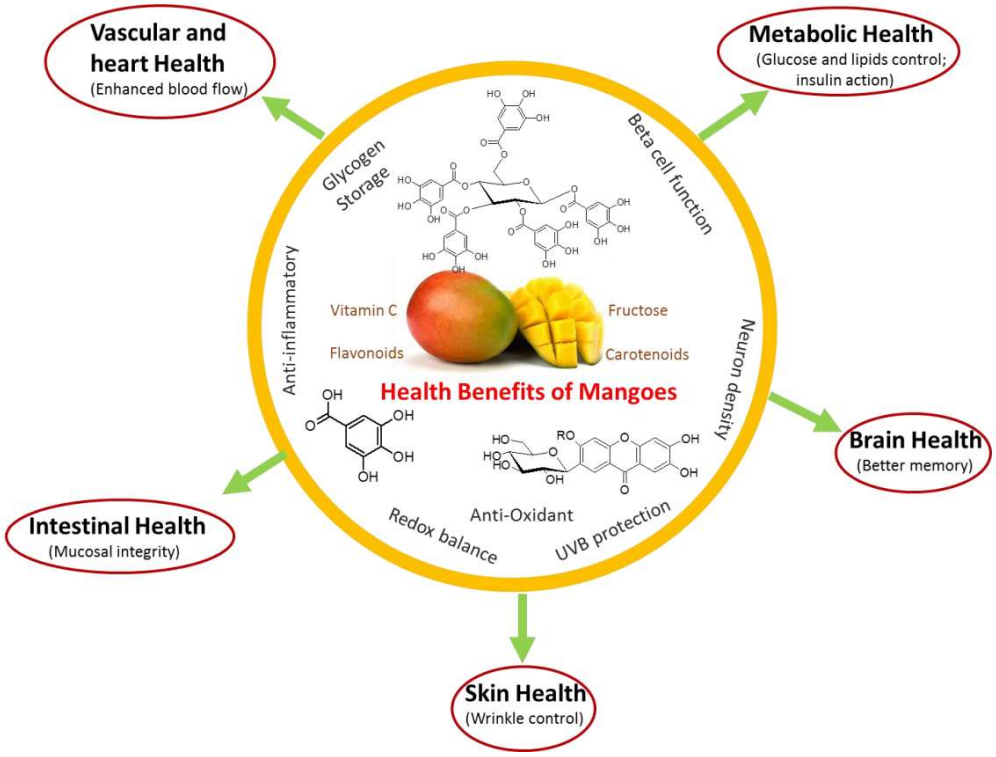

Figure 1.2: Potential health benefits of mango consumption, from (Burton-Freeman et al., 2017).

1.4 Quality parameters of mango

Raw material properties affect the overall final product quality after processing. Hence, methods have been developed to select suitable mangoes before applying any processing technology. Mango maturity depends on factors such as cultivars, time of harvesting, conditions of storage and transport (Nunes, 2007). These factors affect chemical composition and sensory quality of the fruit. Main maturity indicators of mango are the decline of acidity and firmness, higher sugar content level (Sirisomboon et al., 2008) and increase of moisture content (Suhaime et al., 2018). During mango ripening process, the change in firmness induces softening that affects cell membrane's structure which plays a major role in mass transfer processes.

1.4.1 Titratable acidity and pH

Content of acids in mango decreases throughout the ripening process due to their conversion into sugars by physiological and biochemical changes (Shafique et al., 2006). Organic acids in mango are mainly malic and citric acids with the latter being predominant (Medlicott & Thompson, 1985; Shashirekha & Patwardhan, 1976). Acidity of mango has generally been assessed by titratable acidity (TA) through measure of the acid citric percentage (Medlicott & Thompson, 1985) and pH in the flesh (Liu et al., 2013). Numerous factors can affect mango titratable acidity: cultivars, growth conditions, storage, climate, regions, etc. (Liu et al. 2013). Mango is considered as an acidic fruit with pH generally lower than 6 (Santos et al., 2008). Values of pH in literature for ripened Tommy Atkins mango ranged from 3.2 to 4.5. (Dutra et al., 2005; Lucena et al., 2000; Rocha et al., 2001; Santos et al., 2008).

1.4.2 Total soluble solids

During the ripening process, gluconeogenesis and hydrolysis of polysaccharides especially starch into sugars lead to increase in total soluble solids (TSS) and decrease of titratable acidity (TA) (Tharanathan et al., 2006; Thompson, 1992). Change in TSS has a positive correlation with sweetness and can be an indicator of mango ripening (Padda et al., 2011). It varies according to cultivars and can reach up to 11%-13.9% for Tommy Atkins to 17% for Ataulfo mango (Medlicott & Thompson, 1985; Ochoa-Martínez et al., 2012). It is measured with a refractometer and consists of g soluble solids per 100 g of solution. A ratio of total soluble solids to titratable acidity (TSS/TA) is used to indicates ripeness of mango (Sivakumar et al., 2011). Rodríguez Pleguezuelo et al. (2012), estimated Tommy Atkins TSS/TA ratio of about 148 at maturity stage.

1.4.3 Color

Color change during fruit ripening is caused by degradation of chlorophyll which reveals presence of other pigments (Tucker & Grierson, 2013) accompanied by accumulation of carotenoids such as β-carotene, xanthophyll esters, xanthophylls and lycopene in the plastids

or synthesis of different types of anthocyanins and their accumulation in vacuoles (Medlicott et al., 1987). Mango **peel** color vary according to the cultivars, red (Tommy Atkins), green or yellow (Bally et al., 2009). Due to peel color variation and the fact it can remain unchanged during the ripening process (Padda et al., 2011), it cannot always correlated with maturity, ripeness or internal eating quality (Medlicott et al., 1992). Therefore, **pulp** coloration is often used to estimate the ripeness of the fruit. Figures 1.3 shows 5 stages (from left to right) of mango ripening process. Ripe mango has yellow to orange pulp tone with intensity increasing during ripening (Medlicott et al., 1992). Pigments such as carotene (Pott et al., 2003) and orange antocyanins (Proctor & Creasy, 1969) are both responsible for the pulp coloration. The higher the carotene content, the most pronounced is the red colour of the pulp (Vásquez-Caicedo et al., 2005). These pigments conferred to mango its attractiveness and also health benefits due to their antioxidant capacities and were proven to contribute against cancers, atherosclerosis and macular degeneration. A colorimeter is often used for color measurement.

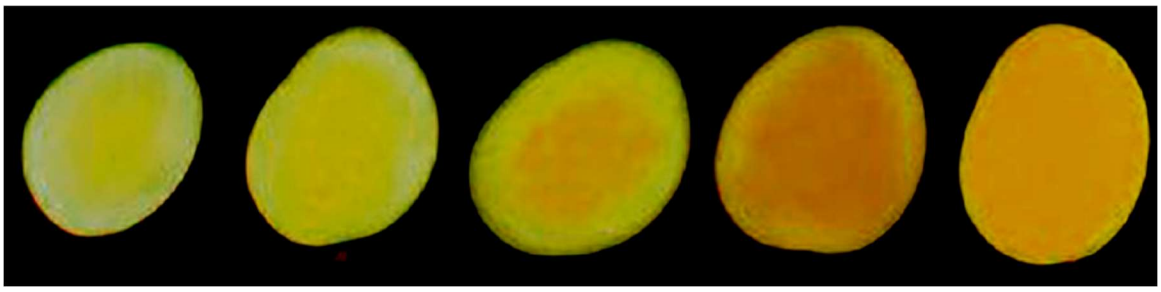

Figure 1.3: Mango flesh color development stages, from (Brecht et al. 2014)

1.4.4 Moisture content

Moisture content can contribute in assessing fruit maturity and is correlated to the final total soluble solids content achieved in ripe fruit, ranging above 80% during the ripe stage for most varieties (Suhaime et al., 2018) including Tommy Atkins (Ochoa-Martínez et al., 2012).

1.4.5 Firmness

Quality parameters (TA, pH, TSS, color, moisture) in the previous sections depend on fruit maturity. Usually, mango is harvested earlier than fully maturity stage which is before the onset of climacteric respiration (Lakshminarayana et al., 1970) for shipping to long distances (Jha et al., 2013). Lack of maturity in fruit during harvesting, led to poor quality of mango and non-uniformity in ripening and less sweetness (Jha et al. 2007, Jha et al. 2010b). Late maturity fruits led to damage during ripening (Medlicott et al., 1988). Therefore, it is important to select fruit with an adequate maturity degree in the commercial supply chain. And textural characteristics such as firmness are one of the most important quality factors because it is related to the maturity, ripening stage and shelf life duration of mango (Jha et al., 2010). A decrease of firmness is observed as fruit becomes more mature and a rapid decrease occurs during ripening. However, firmness evaluation is a challenge due to variation with cultivars (Jarimopas & Kitthawee, 2007), agricultural conditions and environment of ripening chamber (Jha et al., 2006).

Numerous methods exist to assess mango firmness. For instance, Padda et al. (2011) compared different methods to measure mango firmness: destructive (penetrometer, durometer) and nondestructive methods (hand squeezing, impact firmness sensor, low-mass elastic method). They found out that the penetrometer was the best method to test firmness for early ripening stage in the destructive category method, while durometer and hand squeezing were fit to assess mango firmness during late ripening stage.

Pulp softening modifies mango mechanical properties. These properties can be measured with a texture analyzer (Sirisomboon et al., 2008) through puncture (penetrometer) or compression. The texture analyzer main parts are the force transducer, the probe, and sample platform. Liu et al. (2019) described the principle of the texture analyzer as the arm force moving down onto the sample and the strength applied creates a pressure or deformation force. The probe transmits the change of pressure or deformation force to the transducers and the parameters (firmness, hardness, fractures, toughness…) are registered by the software. Puncture and compression probes are commonly used for vegetables and fruits firmness (Liu et al., 2019). The parameters to read depend on the probe used. Needle probes or puncture probes are used for penetration test where the probe perforates the skin of the fruit in a

localized part. This method is used to test the maturity of fruit through hardness and firmness measurements. Flat probes or compression probes are used for a compression test where the sample is placed onto a flat surface and an upper fixture, larger than the sample, and then it is lowered into the sample.

1.5 Processing technologies of mango

Mango is a climacteric fruit, which continues to ripe after harvest. Its availability as fresh product is restricted to a short period throughout the year. Thus, due to its important nutrient and sugar content after harvesting, it is exposed to physico-chemical and microbiological degradations reducing its shelf life. Preservation methods have been therefore developed to prolong its availability and retain nutrients, facilitating its incorporation in consumers diets in diverse forms and enhancing the economies of those countries which produce mango.

Two categories of mango by-products are found on the market. Minimally processed products are fresh-cut fruits usually pretreated with ascorbic acid, citric acid, calcium chloride and chitosan coating (Siddiq et al., 2012), which need special packaging (PET, modified-atmosphere) and temperature of 4 °C to avoid enzymatic and microbiological degradation (Malik et al., 2017). The other category is processed mango which has the advantage of extended shelf life compared to the fresh-cut products. Processed mangoes are mango juice and juice concentrate, nectar, concentrated pulp and puree, leather, pickles, jam, jelly, chutney with added sugar, flakes, chips, frozen and dried mango (Deepti et al., 2017; Malik et al., 2017). Processed mangoes are popular products, especially for exportation. Air drying or freezing are among the most used processing technologies (Rawson et al., 2011).

1.5.1 Air drying

Drying, the process of thermally removing water from a product (Mujumdar & Menon, 1995), is one of the most common preservation technologies used for dehydration of a foodstuff, especially for fruits and vegetables. Drying extends storage life by reducing water activity. For instance, microorganisms and insects' development is inhibited below 70% humidity (Sokhansanj, 1995) and, at lower water activities, chemical reactions are stopped

or slowed down (Barbosa-Cánovas & Vega-Mercado, 1996). Quality is improved through palatability and digestibility, but it could also be negatively affected (i.e. color and flavor) depending on the processing conditions (Sokhansanj & Jayas, 1995) when using high temperatures for example. Drying ease products handling by reducing weight and volume consequently facilitating transportation (Sokhansanj & Jayas, 1995).

Hot air drying consists of applying a hot stream of air to evaporate moisture in food material, it is the most common drying process and it allows the dry product to be stored for a year (Ratti, 2001). Hot air drying is generally used for foodstuff dehydration because it is a traditional method known for centuries, also it is considered as simple and economical compared to methods such as freezing and freeze-drying. Hot air drying is commonly used in tropical countries of sub-Saharan Africa to preserve mango due to these factors. However, drawbacks of water removal through such method lie on the marked decrease of organoleptic, chemical, and nutritional qualities of the food product. Typical food degradation is caused by high temperatures and contact with oxygen which can affect color and aroma through browning and lipid oxidation, in addition to undesirable modification of texture, volume through shrinkage, loss of sensitive nutrients such as ascorbic acid and so on. As an example, Sehrawat et al. (2018) reported that hot air drying (60,70,80 °C) decreased ascorbic acid, beta-carotene, total phenolic content, and antioxidant activity of mango along with color and texture undesirable changes. Other authors have found similar conclusions after mango air drying (Chen et al., 2007; Izli et al., 2017; Sogi et al., 2015). The unwanted color modification of mango after hot air drying was attributed to Maillard reaction due to high temperatures employed (Sehrawat et al., 2018). Additionally, the presence of oxygen led to browning of pigments. Hot air-drying method led to structure collapse and rigid texture of mango which was indicated by a low rehydration ratio (Sehrawat et al., 2018). Acid ascorbic retention decreased by 36-48% and beta-carotene decrease were due to oxidation (Sehrawat et al., 2018). Decrease of beta-carotene affects the dried mango appearance because beta-carotene is responsible for the yellowish color in mango (Sehrawat et al., 2018). Total phenolic content decreased with temperature and total antioxidant activity decreased due to thermal and oxidative degradation of phenolic compounds (Izli et al., 2017; Sehrawat et al., 2018). In addition, air drying requires high energy input during lengthy periods (Izli et al., 2017; Ratti, 2001; Sokhansanj & Jayas, 1995; Yi et al., 2017). For instance, Singh et al. (2022) studied

the energy consumption of mango during hot air drying to find out that up to 83.74 kJ were required for a 50 liters capacity air dryer equipment, the highest energy consumption among the other methods tested.

Based on these findings on mango drying, air-drying is not recommended to process heat sensitive biological material or high-value foodstuffs (Lewicki, 2006). Therefore other methods operating at low temperature and less oxygen availability can be better alternatives for mango to enhance retention of ascorbic acid, color and phenolic compounds (Izli et al., 2017). Such conditions are found in osmotic dehydration.

1.5.2 Freezing

Freezing, another process to preserve mango, reduces food temperature to at least -18 °C leading to water crystallization (Barbosa-Cánovas et al., 2005; De Ancos et al., 2006). The temperature reduction happens in three separate phases : pre-cooling or chilling phase that leads the material to reach its freezing point temperature; phase change of water from liquid to crystallized (ice); and finally a tempering phase where the product reaches its final established temperature (Delgado & Sun, 2001). The crystallization fixes the tissue structure along with preventing water availability for microorganisms' growth, chemical and cellular metabolic reactions leading to long-term storage, particularly for seasonal fruits such as berries, litchi, cherries and mango (Kaur & Kumar, 2020). However, size and form of ice crystals (large and needle-shaped) and their location may lead to severe tissue damage and can be avoided by speeding up crystallization phase and freezing rate (Brennan, 1990; Ramaswamy & Tung, 1984) through lower temperatures (< -18°C).

Mango is a perishable fruit, and has a short storage period at ambient temperature (only 2-3 days) and about 2-3 weeks at 10-15°C (Yahia, 1999). Freezing can help extend its storage (Sriwimon & Boonsupthip, 2011). Mango can be frozen as fresh or with added syrup or dried sugar to benefit from the cryoprotectant effect of sugars. This can be seen in the study by Isaacs (1986) which used slices of mango *cv*. Kensington pretreated by sucrose syrup or dry sugar, compared to fresh mango without sugar addition as control. Mango samples were frozen at -30 °C and -18 °C and stored for 3 months before analysis of quality retention. The

pretreatment with syrup and dry sugar showed it is possible to obtain frozen-thawed mango with better quality retention (appearance and flavor) than the frozen-thawed mango without sugar addition. This is due to the cryoprotectant effect of sugar which prevents reaction with oxygen and thus avoiding browning and enzymatic reaction that could deteriorate flavor and color. However, due to granular mouth perception of dry sugar, it was the least appreciated after the panel study with a preference for frozen-thawed mango pretreated in syrup followed by the frozen-thawed fresh mango. Another example is a relatively more recent study by Sriwimon & Boonsupthip (2011) which compared partially ripe and ripe mangoes (*cv. Nam Dok Mai*) with or without impregnation of mango juice and other sugars mixture (sucrose, glucose and fructose) after freeze-thawing. This study resulted in an overall better-quality product of frozen-thawed mangoes with syrup impregnation pretreatment before freezing. Pretreated mangoes had higher firmness, color, and sensory retention than frozen mango without pretreatment in syrup due to the cryoprotectant effect of sugars.

Other studies targeted freezing conditions (freezing rate, temperature) on the quality of frozen-thawed mango. Zhang et al. (2017) studied state/phase transitions through freeze-thaw cycles on *cv.* Keitt mango quality. They compared frozen mango at different temperatures, i.e., glassy state (-65°C, -60°C, -49°C), partially freeze-concentrated state (-38°C) and rubbery state (-28°C). Freeze-thawing affected mango quality compared to fresh mango in all freezing conditions. Hardness decreased after freeze-thawing, a drip loss was observed, accompanied with color change, drop of acid ascorbic and pH increase. However, glassy state (-65°C, -60°C, -49°C) and partially freeze-concentrated state (-38°C) showed superior quality for mango over rubbery state (-28°C). This could be explained by the fact that high freezing rate reduced freezing damage due to the smaller ice crystals formed compared to slow rate freezing. Similar conclusions were found by Charoenrein & Owcharoen (2016) for mango *cv. Nam Dok Mai* after comparing freezing rates (slow freezing -20°C, medium freezing -40°C, fast freezing -80 °C) effect on frozen-thawed mango quality. All freezing conditions decreased the firmness of the mango and the sensory quality. The pectin substances analysis demonstrated that freezing did not impact the pectin content that form the cell walls, however it damages cell walls by liberating smaller pectin (water soluble pectin and ammonium oxalate soluble pectin) which contributes to the fruit texture softening. The frozen-thawed mango quality was better in the decreasing order for fast freezing (-80 °C),

medium freezing (-40 °C) and slow freezing (-20 °C). Antonia Marín et al. (1992) studied freezing at -18 °C during 4 months of storage for four *cv.* Smith, Lippens, Palmer, Davis-Haden mangoes. They observed good preservation of moisture content, soluble solids and a slowdown of polyphenoloxydase and peroxidase activities during storage. During the freezing process, mango acidity decreased by half, there was a decrease of acid ascorbic and beta carotene and its pH increased, however these parameters did not change during the frozen storage step. Overall, these studies showed that fresh mango would still be superior in quality to frozen or frozen-thawed mangoes. However, with the objective to have a continuous mango supply throughout the year, freezing could be interesting as a preservation method.

Traditional freezing methods, on the other hand, have the disadvantages of low freezing efficiency and generation of large ice crystals, leading to possible damage of food quality (Zhang et al., 2018), and as well, the need for storage and transportation cold chain at controlled freezing temperatures leaves this technology for application mostly in developed countries.

Freezing is also often used as a pretreatment prior to drying to enhance drying kinetics (Lewicki, 2006) in process such as air drying and osmotic dehydration. Freezing pretreatment was able to enhance drying rates and shorten drying time for various fruits like blueberries, carrots, pumpkins (-20 °C) (Ando et al., 2016; Zielinska et al., 2015). A 187% increase of water diffusivity was recorded after freezing at -34 °C of banana followed by drying of banana (Dandamrongrak et al., 2002). Freezing led to 27% less energy consumption in frozen blueberries drying than in untreated ones. This ability of freezing is due to its modification of tissue structure leading to ease in water removal during drying (Vallespir et al., 2018). Studies with mango freezing as pretreatment has been as well conducted prior to air drying and osmotic dehydration. For instance, Zhao et al. (2017) carried out experiments on osmotic dehydrated mango and untreated mango frozen at -18°C and -55°C to evaluate the effects on mango quality. They found out change in color for both freezing temperatures and less color change for the osmotic dehydrated mangoes due to protection against enzymatic browning and faster rate of freezing due to initial lower water content. A decrease in hardness was recorded after freezing along with up to 89.8-90.2% of vitamin C loss after 3 months of

storage, due to drip loss. Khuwijitjaru et al. (2022) recently studied freezing effect on osmotic dehydration and hot air-drying kinetics of mango *cv. Kaew Kamin.* The freezing conditions were -40°C (fast freezing) and -18°C (slow freezing) and the storage durations were 0, 1 and 2 months. Osmotic dehydration was carried out in a 38 °Brix sucrose solution on the frozen mangoes (without thawing). This experimentation set up showed that fresh mango (control) had higher water loss than frozen mangoes after osmotic dehydration. The ice state of water in the frozen mango may have reduced the rate of water loss compared to its liquid form in the fresh mango (Khuwijitjaru et al., 2022). However, sugar gain was increased due to the freezing pretreatment. No difference was recorded for the water loss and sugar as a function of the storage duration (0, 1 and 2 months) and similar conclusion for the freezing rate was found. This may be due to the relatively short period of storage used in their study. After hot air drying that followed up osmotic dehydration, water loss rate was increased for frozen mango compared to fresh mango. Frozen mango showed lower moisture content, and better color retention due to lower polyphenoloxydase activity, an advantage of freezing as shown in Antonia Marín et al. (1992). Floury et al. (2008) observed increase of water loss rate after freezing at -18 °C followed by osmotic dehydration of frozen mango in 40-50°Brix sucrose solution. In contrary to the previous study, they found no effect on the sugar gain which is quite surprising compared to the results of Khuwijitjaru et al. (2022) and Lazarides & Mavroudis (1995), which showed an increase of sugar gain after freeze-thawing of mango and apples respectively. Fruit type, maturity and conditions of the experiments may explain these differences.

Among others, the results of Antonia Marín et al. (1992) and Zhao et al. (2017) demonstrated freezing limits in maintaining good nutritive and organoleptic qualities of mango over a long period of storage. Therefore, when drying is required after freezing, choosing an appropriate method to enhance the nutrients is a better choice than drying directly at high temperatures such as hot air drying which can lower more the nutrients content. Therefore, it is beneficial to enhance the quality of the mangoes through osmotic dehydration which through the composition (nutrients, sugars) of the osmotic solution can help improve the overall quality of the final product.

1.6 Osmotic dehydration

Since research on osmotic dehydration (OD) has been pioneered by Ponting et al. (1966), the interest of scientific and industrial on the field are ongoing (Spiazzi & Mascheroni, 1997). Osmotic dehydration (Figure 1.4) represents the immersion of a water-rich cellular food, into a high osmotic pressure solution called 'osmotic solution' (Beaudry et al. 2004). Common solutes for osmotic solutions are sugars (sucrose, fructose, glucose…) or brines (NaCl), but recently, other solutes have been proposed as natural alternatives (xylitol, sorbitol, D-allulose, fructo-oligosaccharides, maple syrup, maltodextrin, corn syrup, fruit juice concentrates…) (Klewicki & Uczciwek, 2008; Lech et al., 2017).

In OD, water activity is reduced through a counter current fluxes: water outflow and solutes entrance to the solid, along with a third (more negligeable) flux from the plant material own soluble solutes (vitamins, minerals…) leaching out together with the water (Bui, 2009). OD can reduce the water content up to a final value of about 50-60% wet basis while increasing soluble solid contents (Spiazzi & Mascheroni, 1997). The intermediate dehydrated food obtained is of good quality, and can be further preserved with other methods such as, freeze-drying, vacuum drying or air-drying with benefits of energy saving, among others, due to partial dewatering through OD treatment (Angilelli et al., 2015; Spiazzi & Mascheroni, 1997).

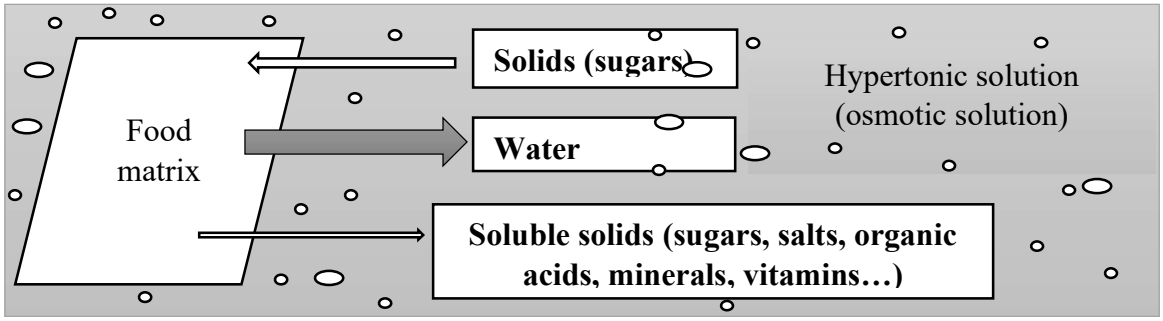

Figure 1.4: Mass transfer representation during osmotic dehydration, adapted from (Torreggiani, 1993)

1.6.1 Pathways of water movement in plant cell during osmotic dehydration

1.6.1.1 Structure of plant cell

Regarding osmotic dehydration of plant-based food such as fruits and vegetables, it is a known fact that mass transfer in such system depends on the structural properties of the plant tissue which is formed of cells (Muñiz-Becerá et al., 2017). Thus, a comprehensive description of the plant cell components is necessary to describe the different pathways of water and solutes during osmotic dehydration. Figure 1.5 illustrates plant cell structure. In general, a plant cell is composed of a cell wall and a protoplast (Lenart & Lewicki, 2006). The cell wall is perforated with small channels called plasmodesmata that connects the neighboring cells with each other. The cell wall is not a barrier for water and low molecular solute and allows their movement from and to the cells (Lenart & Lewicki, 2006). The protoplast is constituted of protoplasm, vacuoles, nucleus, plastid, etc. The protoplasm is enclosed by a membrane named the cell membrane (historically known as plasmalemma). The vacuoles are enclosed in a membrane called tonoplast and contains soluble solutes such as minerals, sugars, and organic compounds; both are located inside the protoplasm (Lenart & Lewicki, 2006). The cell membrane that encloses the protoplasm is permeable to water and selectively permeable to other solutes because of its ability to discriminate through their ionic, size and electrochemical characteristics (Muñiz-Becerá et al., 2017).

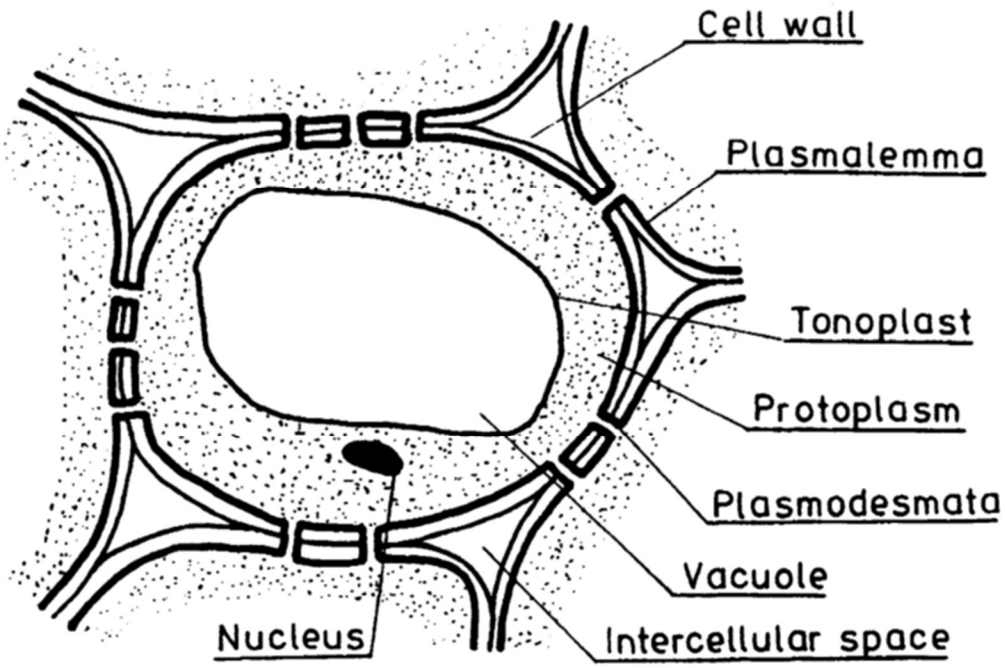

Figure 1.5: Basic plant cell structure, from (Lewicki, 2006)

1.6.1.2 Pathways of mass transfer in plant cell during osmotic dehydration

Numerous authors agreed that there are three potential accepted pathways of mass transfer in plant cells during osmotic dehydration: apoplasmatic, symplasmatic and transmembrane (Chiralt & Fito, 2003; González-Pérez et al., 2021; Nahimana et al., 2011; Shi & Le Maguer, 2002). The apoplasmatic transport is the diffusion of water and solutes through cell wall and intercellular spaces (González-Pérez et al., 2021). Symplasmatic transport represents the cell-to-cell transport through the plasmodesmata (Shi & Le Maguer, 2002). Transmembrane transport describes cellular interior (cytoplasm and vacuole) and exterior (cell wall and interspaces) exchanges across the cell membrane (González-Pérez et al., 2021). Cell wall, plasmalemma, and tonoplast, allow in normal conditions, osmotically regulated movement

of water and solutes throughout the plant cell interior (Spiess et al., 2002). However, when plant tissues are subjected to high osmotic pressure due to immersion in a hypertonic solution, they begin to lose water to the outside leading to shrinkage and detachment of the plasmalemma from the cell wall (Bui, 2009).

1.6.1.3 Driving force of mass exchange in plant tissue during osmotic dehydration

In a solution, solvent (water) builds interactions with solute. These interactions characterize the water thermodynamic state in a solution and requires energy state of each substance (water, solute) (Lenart & Lewicki, 2006). This energy state referred to one mole of the substance is called the chemical potential (Lenart & Lewicki, 2006).When a plant cell is immersed in a hypertonic solution, the driving force responsible for solute and water transport across the cell membrane is the chemical potential gradient between the solutions in the intracellular and extracellular volumes (Floury et al., 2008). The chemical potential depends on concentration, temperature, and pressure (Lenart & Lewicki, 2006). During interaction of two systems with different energy states, there is an energy exchange until the equilibrium state is attained. In equilibrium state, chemical potentials of the two systems involved are the same. Under isothermal conditions, equilibrium state is reached by changing concentration or pressure. The excess pressure needed to reach the state of equilibrium is called osmotic pressure (Lenart & Lewicki, 2006). Increase in solution concentration and low molar mass of solute increase the osmotic pressure (Rastogi et al., 2002). The smaller the molar weight of the solute, the higher will be the osmotic pressure at the same concentration (Lenart & Lewicki, 2006).

1.6.2 Osmotic dehydration kinetics

Kinetics of osmotic dehydration are mostly studied in terms of water loss and solids gain (Bchir et al., 2011), which depend on the operating conditions, cellular tissue type and eventual pretreatments (Spiazzi & Mascheroni, 1997). However, water loss is predominant (50-60%) compared to sugar gain (Pan et al., 2003). Performance of osmotic dehydration can be obtained then through the ratio of water loss to the solids gain (Khin et al., 2005).

In addition to experimental determination, mathematical modeling contributes to characterize osmotic dehydration kinetics of food materials (Yildiz et al., 2016). Empirical modelling is based on fitting experimental data with a mathematical expression of adequate tendency to predict the mass transfer. In this category, one of the most used models to represent osmotic dehydration kinetics is that described by Azuara et al. (1992), where the rates of water loss (*WL*) and solute gain (*SG*) as a function of time are represented with Equations (1.1) and (1.2) having two fitting parameters:

$$WL = \frac{S_1\ t\ WL_\infty}{1 + S_1\ t} \tag{1.1}$$

$$SG = \frac{S_2\ t\ SG_\infty}{1 + S_2\ t} \tag{1.2}$$

where *WL* is the water loss fraction at time *t*; WL_∞ is the water loss fraction at equilibrium; S_1 is a constant related to the water loss. Similarly, *SG* represents the solids gain by the food at time *t*; SG_∞ is the solid gain by the food at equilibrium; and S_2 is a constant related to the rate of solid diffusion into the foodstuff. *WL* and *SG* values determined from the experimental data at different times can be used to estimate equilibrium water loss (WL_∞) and solid gain (SG_∞) from the slope and intercept of the plot of (*t/WL*) and (*t/SG*) versus *t* (Assis et al., 2016).

Azuara model has the advantage to predict the equilibrium values. It has been applied to represent osmotic dehydration kinetics of various foodstuffs to predict the water loss and solid gain at equilibrium (Kaymak-Ertekin & Sultanoğlu, 2000). Azuara model was fitted successfully for water loss and solids gain prediction of carrots (Singh et al., 2008), apples (Kaymak-Ertekin & Sultanoğlu, 2000), onions (Sutar & Gupta, 2007), sweet potatoes (Junqueira et al., 2017) and green figs (de Mello Jr et al., 2019). However, this model does not take into account the product characteristics such as shape, size, foodstuff structure and the process conditions (temperature, concentration…) (Ochoa-Martinez et al., 2007).

Other researchers used theoretical mathematical models with the following conditions: isotropic material, constant humidity inside the food, unidirectional mass transfer, high solution/product ratio, semi-infinite geometry, limited geometric shapes (cylinder, cube,

spheres, rings) and negligible effects of shrinkage and external resistance to mass transfer (Muñiz-Becerá et al., 2017). Under these conditions of pure internal diffusion, the differential equations of the Fick's laws are used to calculate diffusion coefficients of water and solutes (Shi & Xue, 2008). Fick's first law originally formulated for diffusion of gases in one phase is based on the principles of molecular diffusion where the concentration gradient is the driving force of the diffusion. However, diffusion of compounds through porous materials such as plant tissues is different from gas diffusion in an ideal model. Indeed, the diffusion of compounds in plant tissues depends on the characteristics of the solutes but also on the plant tissues properties (Shi & Xue, 2008). The following Equation (1.3) represents the effective Fick's law for mass transfer:

$$J_i = -D_{effi}\frac{\partial C_i}{\partial x} \qquad (1.3)$$

where J_i (g/m^2/s) is the water or solute flux; D_{effi} (m^2/s,) is the effective diffusion coefficient; C_i (kg/m^3) is concentration of compound i in the solid and x (m) represents the distance of diffusion. The negative sign in Equation 1.3 indicates the direction of the diffusion which is on the opposite side to the concentration gradient (Crank, 1975).

The effective diffusion coefficient depends on tissue structure and process temperature as shown in the Equation (1.4):

$$D_{effi} = \frac{D.\ \varepsilon}{\tau} + f(T) \qquad (1.4)$$

where D stands for the pure diffusion coefficient; ε is the porosity; τ is the tortuosity and T represents the temperature.

The so-called Fick's second law (Equation 1.5) is a mass balance derived from the Fick's law (Equation 1.3), which is the most common used to study the kinetics of osmotic dehydration:

$$\frac{\partial Ci}{\partial t} = D_{effi}\frac{\partial^2 Ci}{\partial x^2} \qquad (1.5)$$

The solutions of Fick's equations are given in Crank (1975) so as to determine the effective diffusion coefficient of water and solutes.

Fick's law has been applied in numerous investigations on osmotic dehydration to estimate the effective diffusivity of water as well as solute in accordance with the experimental values.

Such were the cases for paprika (Ade-Omowaye et al., 2002), carrots (Singh et al., 2007), apples (Jalaee et al., 2011), lemon (Rubio-Arraez et al., 2015) and mango slices (Giraldo et al., 2003). However, in practice, mass transfer depends not solely on diffusion and other factors need to be considered for accurate description. These factors are properties of the biological material, boundary layer formed at the tissue interface, shrinkage, properties of osmotic solutions that may lead to a high external resistance which could impact the mass transfer. Therefore, to accurately model the mass transfer, some authors instead of using Fick's laws in their original forms, introduce important variables. Also, in the case of non-negligible external resistance (for instance high viscosity of osmotic solution) to the mass transfer, Fick's law may not be suitable to represent the mass transfer. Indeed, osmotic dehydration may be externally controlled in some conditions: high solution viscosity, low or absence of agitation, low temperature, thick samples, or shrinkage. For instance, Bui et al. (2009) considered two additional factors, shrinkage and the boundary layer formed at the solute-tissue interface for modelling the mass transfer kinetics during tomato osmotic dehydration. Other conditions were experimented by Mavroudis et al. (1998) for apple in a sucrose solution of 50% at 20 °C with laminar and turbulent flow. They found water loss was higher in the turbulent flow than the laminar flow, confirming that external mass resistance was an important factor to control the osmotic dehydration in apples. Finding similar conclusions, Garrote et al. (1992) experimented conditions for external mass transfer resistance with high concentrated osmotic solution (67.5% sucrose) at low temperatures (5 °C and 25 °C) with and without agitation for apples, strawberry and pear halves.

1.6.3 Relationship between internal and external mass transfer

During osmotic dehydration, two resistances to the mass transfer may occurred simultaneously, internal and external (Spiazzi & Mascheroni, 1997). The osmotic solution properties such as viscosity, concentration, temperature, agitation, osmotic agent (size, polar or ionic) and product/solution ratio govern the external resistance. Whereas the internal resistance is related to the tissue structure (ripeness index, variety, porosity, tortuosity, geometry, size…) (Derossi et al., 2011). Convective mass transfer coefficient, internal diffusion coefficient, sample thickness and the equilibrium relationship at the interface are

parameters used to characterize the relationship between internal and external resistance for mass transfer (Bui, 2009).

In the literature, most authors assume that the external resistance is negligible, considering the control is by purely diffusional internal resistance (Angilelli et al., 2015). However, according to the conditions of the osmotic dehydration (concentration, viscosity, solutes size…), external resistance may be important to the mass transfer. Thus, it might be necessary to consider both internal and external resistances to the mass transfer or determine at least which one is predominant. The dimensionless Biot number (Bi_m) represents the ratio of internal mass transfer resistance (food matrix) to external resistance (osmotic solution) according to Equation 1.6 (Ratti, 1994) :

$$Bi_m = \frac{K_C\, L_0}{P_1\, \rho_S\, Deff_i} = \frac{Internal\ resistance}{External\ \ resistance} \qquad (1.6)$$

where K_c = Mass transfer coefficient (kg water)/(m^2 s kPa), L_o = characteristic length (m), P_1 = equilibrium relation at the interface (kg water/kg dry matter)/kPa, ρ_s = Dry matter concentration (kg dry matter/m^3)

From the above equation, the range of the Biot number dictates the nature of the mass transfer control. It is assumed that when Biot number is >100, mass transfer is controlled by internal resistance; for a Biot number <0.1, external resistance is the controlling factor; lastly, when both resistances are equal, the control of the mass transfer is 'mixed' through internal and external resistances. Conditions of the osmotic dehydration process related to the food material and the osmotic solution properties indicate which kind of control could be predominant for the mass transfer. Mass transfer in thick food samples would be controlled through internal resistance, whereas thin samples would be controlled by external resistance (Pakowski & Mujumdar, 2020). When the sample thickness is small, the Biot number is reduced, and external conditions are likely to be predominant. Additionally, high concentration and viscosity of solution or absence of agitation favor smaller Biot number.

As the majority of research on osmotic dehydration assumed Fickian diffusion with negligible external resistance, only a limited number of studies have reported an estimation of Biot number in osmotic dehydration of food materials. Pacheco-Angulo et al. (2016) assumed for the osmotic dehydration of carrot slices in sodium chloride solutions, diffusion-

controlled process with high stirring and convective controlled process without stirring and with product/solution ratios of 1:4 and 1:20. They reported high Biot number for water (87.9) and for solute (126.7) in the case of non-stirred conditions. The values of Biot numbers without stirring were lower in the range of 21.3-28.3 and 14.8-19.7 for water and solute respectively. In this case, despite the absence of agitation, Biot number showed the process of carrot slices osmotic dehydration was mainly controlled by internal resistance. On the contrary, da Silva et al. (2013) examined coconut osmotic dehydration in sucrose solution (35 °Brix, 40 °C) in negligible and non-negligible external resistances conditions and reported Biot number of water and solute equals to 7.75 and 5 respectively showing an external resistance to the mass transfer. The same authors obtained Biot number values of 1.5-2.3 and 0.62-0.95 for water and solute respectively for pineapple (da Silva et al., 2014) and found the mass transfer was mixed with internal flux at the boundary and external convective flux control. Angilelli et al. (2015) considered an existence of a film formation that form a barrier to the mass transfer during osmotic dehydration of melon pieces in fructo-oligosaccharides. Their model confirmed that external resistance must be considered to represent accurately the experimental results and found the Biot number determined for water equals to 14.87.

1.6.4 Factors affecting osmotic dehydration mass transfer and quality of product

Two groups of parameters affect osmotic dehydration mass transfer and quality of product: process conditions (temperature, concentration, time, …) and the product parameters (tissue structure, thickness/size, pretreatments) (Lazarides, 2001). Usually, the objective being to dehydrate the food material, water loss is the most desired transfer, however in other cases it could be as well important to reduce solids uptake, therefore specific conditions of the factors can be chosen to favor one transfer or the other.

1.6.4.1 Osmotic dehydration time

During osmotic dehydration, water loss and solid gain increase with time although maximum rates are registered during the first two hours of osmotic dehydration (Allali, 2008a; Tortoe,

2010). Mass transfer in osmotic dehydration happens in two distinct stages. From 30 min to 2 hours, water loss rate is fast, then it starts to slow down, favoring solids uptake (Lenart & Lewicki, 1987). Lazarides (1994) reported water loss rate of apple slices dropped to 20% from the initial flow rate during the first hour. Then within three hours, the product lost half of its initial moisture and doubled its initial total solids content by gaining sugars and the mass transfer leveled off. During osmotic dehydration, a solute layer may form at the surface of the food material which could be responsible of reducing water diffusion rate. In addition, dehydration led to cell shrinkage and damage, leading to loss of selectivity and impregnation of solutes (Ferrando & Spiess, 2001; Mavroudis et al. (1998). Thus, to reduce sugar gain, osmotic dehydration must be stopped as soon as possible (Tortoe, 2010). The treatment time can be reduced depending on the conditions such as the concentration and the temperature of the osmotic solution, the pre-treatments (pulsed electric field, ultrasound, high hydrostatic pressure, etc.) and the presence of agitation (Phisut, 2012).

1.6.4.2 Osmotic dehydration temperature

Temperature is a critical factor affecting mass transfer during osmotic dehydration (Azuara et al., 1992). High temperatures increase water loss, solid gain, and weight reduction. This is related to high temperature effect on increasing cell permeability through tissue swelling and plasticizing and by releasing the trapped air in the cellular structure freeing space for water removal and the entry of solutes into the fruit (Lazarides et al., 1999). High temperature promotes osmotic pressure increase (Falade & Igbeka, 2007) and a reduction of solution viscosity, which represents an external resistance to the mass transfer. Lazarides (1994) reported up to 55% of solid gain in apples osmotically dehydrated at 30-50°C which was above value under room temperature osmotically dehydrated apples. Kaymak-Ertekin & Sultanoğlu (2000) reported after osmotic dehydration of apples in a 60°Brix solution at 20°C and 50°C, water loss and sugar gain increase at 50 ºC. Similar results were found by Falade & Igbeka (2007) for African star apple. Although increasing temperature favors mass transfer, obviously aside for the energy consumption required for heating the osmotic solution, it may lead to undesirable changes to the plant material at temperatures above 50°C in terms of color, flavor, aroma and nutrients degradation, in addition to enhancing sugar gain

which is nowadays unfavored by consumers (Del Valle et al., 1998; Shi & Xue, 2008). Many authors have demonstrated a significant drop of nutrients as temperature rises. Red bell peppers in sucrose and salt osmotic solutions were treated at temperatures range of 25-55°C with the aim of evaluating effect on vitamin C and carotenoid content (Ade-Omowaye et al., 2002). They found that temperature had an improvement effect on water loss and sugar gain, but to the expense of vitamin C which drops at 20%-4% of initial value and 80%-55% for carotenoids. Similarly, Devic et al. (2010) estimated 80% loss of vitamin C at 45°C and 100% loss at 60°C. As for the polyphenol content, they estimated 74-85% retention of the initial content polyphenol compounds and vitamin C after osmotic dehydration of apples at 45-60°C. In the work of Almeida et al. (2015), temperatures above 45° C during osmotic dehydration of bananas in sucrose, led to a positive effect on mass transfer while a drop of 70% of phenolic compounds along with darkening and a decrease of antioxidant activity. As for Kucner et al. (2013), they observed after osmotic dehydration of blueberry in sucrose solutions at 30-70°C, undesirable softening of texture and higher polyphenol loss at 70°C compared to 30°C. Augmenting temperature above 40°C during osmotic dehydration affects the tissue structure through cells wall rupture and collapse, reducing membrane selectivity and enhancing the leaching of nutrients (vitamins, phenolic content) through diffusion, in addition to chemical degradation (Vial et al., 1991). Based on the examples and discussion above and the statement of Ponting (1966), it can be concluded that medium temperature (<50 ºC) can sufficiently promote water loss, weight reduction and sugar gain while maintaining minimum solids uptake along with nutritional and organoleptic integrity of the final product.

1.6.4.3 Osmotic solution properties

1.6.4.3.1 Type of solute

Factors such as cost, taste and flavors compatibility with the food as well as the preservation properties determine the choice of the osmotic solute (Torreggiani, 1993). **Sucrose** is the most common solute for fruits, because it is cheap and easily available (Chen et al., 2007). Other common solutes are **glucose** (Chenlo et al., 2002; Karathanos et al., 1995; Lerici et al., 1985; Nieto et al., 2004; Panagiotou et al., 1999), **fructose** (Barman & Badwaik, 2017;

Giangiacomo et al., 1987; Klewicki & Uczciwek, 2008; Kotovicz et al., 2014; Leahu et al., 2020), **high corn syrup** (Beaudry et al., 2003; Bolin et al., 1983), **maltose** (Chottamom et al., 2012; Mastrantonio et al., 2009; Torreggiani et al., 1988; Vicentea et al.), **starch or corn syrup** (Argaiz et al., 1994; Contreras & Smyrl, 1981; Giangiacomo et al., 1987; Lazarides et al., 1995), **maltodextrin** (Azuara et al., 2002; Li et al., 2017; Shinde & Ramaswamy, 2021) and **glycerol** (Barman & Badwaik, 2017; Brochier et al., 2015; Moreira et al., 2007).

Osmotic solutes with low molecular weight diffuse more easily into plant cells compared to those with high molecular weight (Tortoe, 2010). Brine solutions (NaCl, KCl and $CaCl_2$) were found to enhance water loss and solid gain compared to sucrose solution which has a higher molecular weight (Chandra & Kumari, 2015). The cell membrane resists to the diffusion of polar molecules such as sugars and thus, solutes such as sucrose may form a surface layer on the tissues, which slows down solute diffusion and water loss compared to salts (Chandra & Kumari, 2015). Formation of chemical bonds between ionic solutes (salts) and the plant tissue matrix alters the resistance of the cell wall and membrane, facilitating the diffusion of the ionic solute (Muñiz-Becerá et al., 2017). A small proportion of salts (1 to 15%) is usually added to sugar solutions and helps to reduce the formation of layer and increase the kinetics during osmotic dehydration (Bekele & Ramaswamy, 2010; Eren & Kaymak-Ertekin, 2007).

Nowadays, factors such as low calorie and health benefits (i.e., prebiotic) are considered as criteria in choosing osmotic solutes to incorporate the best properties into the final product. Replacement of common solutes (sucrose) with alternatives to improve texture, color, and reduce calories of the dried fruits is nowadays part of novel research in osmotic dehydration of fruits (Dermesonlouoglou & Giannakourou, 2018; Katsoufi et al., 2017). Different solutes are also mixed to form multicomponent solutions to control solute uptake and enrich final product in nutrients and functional components.

Table 1.5 presents examples of osmotic dehydration studies using alternative solutes in single or multicomponent osmotic solutions. These solutes are **polyols** (xylitol, maltitol, sorbitol, erythritol), **carbohydrates** (polydextrose) and **polysaccharides** (maltodextrin, fructo-oligosaccharides, inulin). Multicomponent osmotic solutions are obtained by substituting sucrose partially (Angilelli et al., 2015; Jiménez-Hernández et al., 2017a; Maldonado et al.,

2020) or totally (Brochier et al., 2015; Dermesonlouoglou et al., 2020; Komes et al., 2007; Mendonça et al., 2017; Oliveira et al., 2012) by novel solutes. In most cases, the novel solutes bring interesting functional properties such as prebiotic (sorbitol, inulin, fructo-oligosaccharides), low calorie and better organoleptic quality (trehalose) and led to sufficient water loss. This points out the possibilities in replacing high calorie solute such as sucrose with novel solutes targeting less calorie and producing functional food through osmotic dehydration.

Other novel osmotic solutions could also be nutritious natural sweeteners such as honey, maple syrup or agave syrup. The literature does not provide much information about the use of agave syrup as an osmotic solution in the osmotic dehydration of fruits. However, studies have been conducted with success for the osmotic dehydration of fruits with maple syrup and honey (Chauhan et al., 2011; Rupasinghe et al., 2010).

Table 1.5: Examples of alternative osmotic solutes in osmotic dehydration

Fruits or vegetables	Osmotic solution °Brix (g solute/100g solution)	Conditions of osmotic dehydration (OD)	Results on OD mass transfer and product quality	References
Apples **Shape: slices** **Thickness: N/A**	25 ° Brix Stevia 50 ° Brix Stevia	Pretreatments: ultrasound, Temperature: 30° C, Time: 1 h Agitation: N/A, Product/solution ratio: N/A	Replacement of individual sugars sucrose, glucose, fructose of apples by low calorie sweetener from stevia (stevioside and rebaudioside).	(Oliveira et al., 2012)
Mango **Shape: slices** **Thickness: 5mm**	60 °Brix sucrose 60 °Brix of mixture (Inulin + Piquin-pepper oleoresin+ Tween 80 +sucrose)	Pretreatments: no, Temperature: 30,40,50 °C, Time: 2 h, Agitation: N/A Product/solution ratio: 1:30	Lower sugar uptake and higher water loss after treatment in inulin solution piquin-pepper oleoresin solution than with sucrose solution. Impregnation of functional ingredients inulin and oleoresin into the mango.	(Jiménez-Hernández et al., 2017a)
Melon **Shape: Pyramids** **Thickness: N/A**	60 °Brix (42% sucrose and 18% fructo-oligosaccharides FOS)	Pretreatments: no, Temperature: 20 °C. Time: 1 to 28 h, Agitation: N/A Product/solution ratio: 1:25	Fructo-ologosaccharides (FOS) had lowest effective diffusion coefficient, pointing out the possibility to enrich melon with prebiotic and low calorie fructo-oligosaccharides.	(Angilelli et al., 2015)
Pears **Shape: cube** **Thickness: 5mm**	25 °Brix sucrose, 25 °Brix trehalose	Pretreatments: 1% acid ascorbic, Temperature: room temperature, Time: 1 h Agitation: yes. Product/solution ratio: N/A	Higher retention of aroma with trehalose (46%) compared to sucrose (38%). Rehydration ratio was higher with trehalose (3.8%) than sucrose (3.4%).	(Komes et al., 2007)
Pineapples **Shape: cube** **Thickness: 5mm**	60% of multicomponent solutions with different level of fructo-oligosaccharides (FOS), sucrose and inulin	Pretreatments: no, Temperature: 60°C, Time: 2 h Agitation: N/A, 60°C, 2 h Product/solution ratio: 1:4	Increasing FOS concentration into the osmotic solution promoted higher water loss than sucrose and inulin. Optimum condition for higher water loss (40%) was found to be 33.4%FOS +23.3% sucrose +3.3% inulin.	(Maldonado et al., 2020)

Table 1.5: *Continued*

Fruits or vegetables	Osmotic solution °Brix (g solute/100g solution)	Conditions of osmotic dehydration (OD)	Results on OD mass transfer and product quality	References
Pumpkin **Shape: slices** **Thickness: 5mm**	40% glycerol +10% trehalose+ 10% galacto-oligosaccharides +ascorbic acid + sodium + calcium chloride mixed with: strained yoghurt whey or pure water	Pretreatments: no Temperature: 35°C-55°C Time: 10-240 min Agitation: N/A Product/solution ratio: N/A	Using strained yoghurt whey as solvent for the osmotic solution gave higher solid gain to the pumpkin microbial stability and a slightly better organoleptic and nutritive quality than pure water solvent.	(Dermesonlouoglou et al., 2020)
Strawberries **Shape: slices** **Thickness: 5mm**	50% sucrose, 20-30% mannitol, 20-40% sorbitol	Pretreatments: no, Temperature: 30°C, Time: 3 h, Agitation: Yes, Product/solution ratio: 1:4	Sorbitol solution of 30% and 40% showed better retention of bioactive compounds and color compared to sucrose. Partial replacement of strawberries natural sugars (sucrose, fructose, and glucose) with sorbitol (prebiotic). Low solubility and crystallization of mannitol from 40% concentration limits its use as an osmotic agent	(Wiktor et al., 2022)
Yacon **Shape: discs** **50 mm diameter** **Thickness: 5mm**	30.2 °Brix glycerol, 34.1°Brix maltodextrin, 34°Brix polydextrose, 37.2°Brix sorbitol	Pretreatments: no, Temperature: 23°C, Time: 6 h, Agitation: Yes, Product/solution Ratio: 1:12	Higher water loss and sugar gain for glycerol, sorbitol than for polydextrose. Low osmotic agent capability for maltodextrin due to its large molecules hindering both water loss and solute uptake.	(Brochier et al., 2015)
Yacon **Shape: slices** **Thickness: 5mm**	40°Brix xylitol, or maltitol, or erythritol, or isomalt, or sorbitol	Pretreatments: 1% citric acid, Temperature: 25°C, Time: 2 h, Agitation: N/A, Product/solution ratio : 1:10	Isomalt and maltitol large molecular weight led to lower sugar uptake. Erythritol gave higher sugar uptake. Sorbitol and xyltitol gave better water loss values and were found to be most suitable as osmotic agents.	(Mendonça et al., 2017)

Maple syrup is a multicomponent natural sweetener which contains mainly sucrose, but also glucose, fructose, vitamins and mineral salts. Rupasinghe et al. (2010) produced a patented method from vacuum osmotic dehydration of apples in maple syrup before vacuum dehydration. They reported better texture and lower water activity of apples in maple syrup compared to sucrose solutions. The osmotic treatment in maple syrup produces a nutritionally enriched apple which contained vitamins, minerals, and biological active compound such as phenolic acids and flavonoids.

Honey is made up of multiple sugars (fructose, glucose, maltose, sucrose) which can be labelled as multicomponent osmotic solution. Chauhan et al. (2011) have made a comparative study with honey, sucrose, glucose, fructose, sorbitol, and maltose as osmotic agents for apple slices. Honey solution showed better osmotic dehydration efficiency than the other solutions except for some similarity with maltose solution. However, a leathery texture and darker color of dehydrated apples was obtained with honey.

Agave syrup comes from a plant of the *Agavacae* family from the semi-desert zones of Mexico where it contributes to soil stabilization and fight against desertification (Garcia-Moya et al., 2011). Some known species of Agave are *A. Tequilana, A. Salmania*, A. *Mapisaga*, A. *Ferox* and A. *Atrovirens* (Diana et al., 2015; Ortiz-Basurto et al., 2008). Agave plant is used to produce syrup, which is recognized as nutritious sweetener due to its varied content in carbohydrates, mainly fructose and lower contents of glucose and sucrose, but also rich in inulin, fructo-oligosaccharides, vitamins, antibacterials (methyglyoxal), polyphenols, and minerals (calcium, potassium, magnesium, iron) (Willems & Low, 2012). Inulin and fructo-oligosaccharides are abundant in agave (Martinez-Gutierrez et al., 2018). Several studies have reported agave syrup functional properties due to their inulin, fructans, amino acid and sugar content (Ortiz-Basurto et al., 2008; Roberfroid, 2005). Fructans (oligomers or polymers) are composed with fructofuranosyl units linked to the fructose residue of a sucrose molecule through β (2→1) and/or β (2→6) linkages. The degree of polymerization (DP) is around 3 to 60 and is used to classify fructans into 2 classes. Fructo-oligosaccarides (FOS) have DP below 10 and inulins have DP higher than 10 (Corradini et al., 2004). Inulin and FOS are food ingredients used to replace fat or sugars in dairy products (de Paula et al., 2020; Meyer et al., 2011). In addition, their prebiotic effect enhanced proliferation of beneficial gut

microbiota (*Bifidobacteria*) (Corradini et al., 2004; Roberfroid, 2005). As in the case of maple syrup and honey, the use of agave syrup as an osmotic solution can also be possible in optimized conditions and could improve the nutritional quality and the microbiological stability of fruits processed through osmotic dehydration.

1.6.4.3.2 Osmotic solution concentration

The rate of osmotic dehydration increases with the solution concentration because osmotic pressure and chemical potential are proportional to concentration (Phisut, 2012). Panagiotou et al. (1999) studied sucrose and glucose solutions concentrations effect on osmotic dehydration kinetics of apples, kiwis, and bananas. They reported an increase of water loss and solids gain with increasing the osmotic solution concentration from 30 to 50 ºBrix. Falade et al. (2003) found similar results with watermelons for sucrose concentrations of 40, 50 and 60 ºBrix. İspir & Toğrul, (2009), Lazarides, (2001) and Mundada et al. (2011) arrived to similar conclusions with apples, apricots and pomegranate seeds respectively.

Too high concentration in osmotic solutions may decrease the rate of osmotic dehydration mass transfer (Lenart & Lewicki, 2006), particularly in the case of sucrose as an osmotic agent. This is due to the large polar molecules of sucrose that form a boundary layer at the product solution-interface and create a barrier for the water removal and solids uptake (Araya-Farias et al., 2014). Some authors have reported this phenomenon with sucrose in their works (Azoubel & Murr, 2004; Mayor et al., 2007; Saurel et al., 1994). Thus, concentration of osmotic solution is often limited to 50-60°Brix to not hinder water removal (Torreggiani, 1993).

1.6.4.3.3 Osmotic solution viscosity

Diffusion coefficient in the solution is inversely proportional to viscosity (Rastogi et al., 2002). High viscosity leads to higher external resistance to the mass transfer (Rastogi et al., 2002) which may lower water loss and sugar gain. Solution viscosity is related to the properties of the solute (size, molecular weight) and other parameters such as concentration and temperature. Some studies have attempted to control the solids uptake through increasing

osmotic solution viscosity by using high molecular solutes such as corn syrup solids (El-Aouar et al., 2006; Lazarides et al., 1995; Lazarides & Mavroudis, 1996) or thickening agents such as xanthan gum (Emam-Djomeh et al., 2001). For instance, when studying osmotic dehydration of apples cubes with sucrose and sorbitol at 60°Brix and 25, 40 and 60°C, Assis et al. (2017) reported that water loss and solid gain of the apple cubes were higher when sorbitol was added to the sucrose solution due to sorbitol smaller molecular weight and thus, the lower viscosity of the solution. Similar conclusions were made by El-Aouar et al. (2006) who reported lower water loss and sugar gain with corn syrup solids compared to sucrose. This difference was attributed to the presence of high molecular weight polysaccharides and the high viscosity of corn syrup solids solution.

Heating up the osmotic solution leads to a decrease of viscosity. Mundada et al. (2011) evaluated osmotic dehydration of pomegranate arils at temperatures of 35, 45 and 55°C. Increasing the temperature led to higher water loss and solids gain, attributed to the decrease of viscosity and increase of diffusion coefficient due to the heating effect. It could be possible to choose the optimum solute and concentration to adjust the viscosity according to the final product desired characteristics in terms of water or sugar contents. To increase the viscosity of the osmotic solution it is possible to use either higher osmotic solution concentration or use thickening agents such as xanthan gum (Emam-Djomeh et al., 2001) or polydextrose (Brochier et al., 2015) among others. The advantage of the thickening agents is that they necessitate only a small quantity to increase the viscosity. Xanthan gum is a polysaccharide produced by the bacteria *Xanthomonas Campestris* (Embuscado & Huber, 2009), which is effective in increasing viscosity of solutions at low concentrations (Emam-Djomeh et al., 2001). In their study, Emam-Djomeh et al. (2001) after addition of 0.5 g/L xanthan gum in sucrose-salt solutions, reported a decrease of solids gain during osmotic dehydration of the model food agar gel.

As shown previously, internal and external resistance to mass transfer during osmotic dehydration can be compared through the Biot number (Ratti, 1994). In the case of thicker products, mass transfer would be controlled internally through diffusion, for which viscosity does not have any impact. However, for thinner samples, kinetics would be influenced by convection. Mass transfer coefficient representing convection depends on solution viscosity. Therefore, high viscosity could be an important parameter to obtain low solids gain in the

case of thinner samples. Overall, viscosity is an important factor for the mass transfer control and would have an important impact in terms of water loss and solid uptake.

1.6.4.3.4 Osmotic solution acidity and pH

The pH of the osmotic solution influences the process (Ramya & Jain, 2017). Studies have shown that optimum temperature and concentration of the osmotic solution is pH dependent (Lewicki, 2006). Contreras & Smyrl (1981) studied osmotic dehydration of apples at low pH (pH=3) and found that acidification increases the rate of water removal from the fruit. Acidification of the osmotic solution at (pH=2), promoted softer texture in apples, this change was attributed to depolymerization and hydrolysis of pectin. In addition, it contributes to the microbiological stabilization of the final product. Ascorbic acids and citric acids are commonly used to lower acidity of osmotic solution (Chavan et al., 2010). Low acidity also improves color by preventing enzymatic browning (Chavan et al., 2010). However, the chosen pH for osmotic dehydration must consider the taste of the final product (Ahmed et al., 2016), because softer texture leads to more uptake of solid (Khin et al., 2005).

1.6.4.3.4 Movement of solution

The agitation of the osmotic solution is used to improve the mass transfer and avoid local dilution of the osmotic solution around the samples (Goula et al., 2017). In addition, it keeps the temperature and concentration homogeneous in the osmotic solution (Eren & Kaymak-Ertekin, 2007). The greater the agitation of the osmotic solution, the greater the water loss during osmotic dehydration (Rastogi et al., 2002). Agitation reduces the external resistance caused by the viscosity of the osmotic solution on the tissue surface and which slows water loss rate (Tortoe, 2010). Agitation has no effect on the gain in solutes when the duration of osmotic dehydration is short, but for a longer period, agitation reduces the gain in sugars (Mavroudis et al., 1998; Raoult et al., 1989). The decrease in the gain in sugars is explained by the high-water loss which influences the concentration gradient of solutes in the plant tissue. However, Ponting (1966) suggested agitation should be designed carefully to avoid fragile fruits disintegration. Bath with agitations, or oscillatory system (most recently

ultrasound) can be used to create movement of the osmotic solution during osmotic dehydration (Bui, 2009; Goula et al., 2017; Saurel et al., 1994).

1.6.4.4 Food material properties

1.6.4.4.1 Intrinsic properties

Fruits have heterogeneous tissue structure related to cell characteristics, void fraction in the cell wall, membrane permeability, tortuosity, skin, porosity, chemical composition (protein, fat, carbohydrates, minerals, etc.) among others (Porciuncula et al., 2013). Heterogeneity in fruits tissue is also related to the varieties, species (even differences among species), maturity level, region, conditions of production (Lazarides, 2001). These intrinsic properties affect mass transfer phenomena during osmotic dehydration (Porciuncula et al., 2013; Rahman, 2007). Mavroudis et al. (1998) reported solid gain and water loss of outer and inner parenchyma of Granny Smith apple were different at similar conditions of osmotic dehydration, probably due to different pathways of mass transfer. Similar results were observed for five apricot varieties (Singh & Heldman, 2001) and two mango varieties (Tiwari & Jalali, 2004). Hartel (1967) obtained a difference of nearly 25% (water loss) under the same osmotic dehydration conditions with potatoes of different varieties. Cell membrane of biological materials is often cited as an important factor in mass transfer during drying process of plant tissues (Rastogi et al., 2002) because cell membranes are semi-permeable and act as a barrier to water removal and solids uptake. As well, Equation (1.4) shows the importance of tissue structure (porosity and tortuosity) in diffusion coefficients in a cellular matrix. Thus, modifying cell membranes microstructural properties can help reduce the internal resistance to mass transfer. This can be achieved through pretreatment (PEF, ultrasound, freeze-thawing, blanching, etc.) that may create more openings to the tissue such as pores and degassing the tissue to facilitate mass transfer during dehydration process.

In addition, the state of the cell membranes is related to the ripening stage of the fruit. Biochemical changes occur during fruit ripening and lead to decrease in firmness (Sulistyawati et al., 2018) consequently leading to low internal resistance for water removal and solids gain.

1.6.4.4.2 Fruit size, geometry, and thickness

The shape and size of the fruits have an impact on the rate of water removal in osmotic dehydration because of the variation in the surface area to thickness ratio (Tortoe, 2010). Generally, fruit thickness and geometry for osmotic dehydration may vary from application to application (Rastogi et al., 2002). Large food pieces lose water more slowly than small ones (Rastogi et al., 2002). Lerici et al. (1985) studied the impact of geometry on the mass transfer during osmotic dehydration of apple in corn syrup solids solution. Apples were cut into sticks, slices, cubes, and rings. Water loss was maximum for apple rings and the minimum was obtained with cubic shape. The authors suggested that increasing surface area to thickness ratio leads to higher solids gain. Indeed, Biot number described in Equation 1.6 is the ratio of internal to external mass transfer resistances and includes the characteristic length (L_o) which is the half thickness in a parallelepiped shape. Hence, an increase of the food material thickness increases towards internal resistance with higher Biot number values. In such cases, mixed or internal mass transfer may become predominant and parameters to control osmotic dehydration may change.

1.6.4.5 Fruit/osmotic solution Ratio

The ratio between fruit and osmotic solution is important for mass transfer kinetics. High ratios of fruit/ osmotic solution at the range of 1:10 to 1:60 are used to avoid dilution of the osmotic solution and avoid undesired effects such as decreasing of osmotic pressure strength and lower osmotic dehydration rate (Tortoe, 2010).

1.6.5 Pretreatments on mass transfer during osmotic dehydration

One cause of resistance to mass transfer during osmotic dehydration is the cellular membrane of the fruit tissue (Dermesonlouoglou et al., 2016). Pretreatments have been developed to improve intrinsic characteristic of the material, leading to permeabilization of cell membrane, and better mass transfer rates. Methods such as coating, ultrasound, high hydrostatic pressure, freezing, freeze-thawing and pulsed electric field are often used as pretreatments (Ahmed et al., 2016). Many of those techniques represent innovative intensification technologies for

improvement of traditional processes by increasing production yields, reductions in equipment size, energy use and waste, and increasing product quality (Nguyen et al., 2021). The following section will focus on suitable methods, such as freezing and freeze-thawing, application of coatings, ultrasound (US) and pulsed electric field (PEF), to improve water loss and possibly minimize solids uptake during osmotic dehydration.

1.6.5.1 Freezing and freeze-thawing on mass transfer during osmotic dehydration

Fruit production in tropical countries takes place only during few months per year, thus a common practice is to freeze the fruits during the short period when they are harvested and later process them by other transformation processes such as osmotic dehydration. **Freezing** was reported to enhance mass transfer during drying and osmotic dehydration. However, possible tissue destruction due to thawing could increase solids uptake, turning the final product into a less healthy high-sugar products. Formation of ice during freezing pretreatment, modifies the tissue structure by mechanisms involving depolymerization of cell walls, cell membranes breakage and osmotic pressures alteration (Li et al., 2018) in addition to degassing. Numerous studies have explored freezing as a pretreatment prior to osmotic dehydration of frozen material. Mundada et al. (2011) proceeded to freeze pomegranate arils at -18 °C before osmotic dehydration to increase the outer layer permeability for a better mass transfer. As well, Bchir et al. (2012) froze pomegranate seeds up to -50 °C before osmotic dehydration in 55°Brix sucrose solution. They reported increment of water loss and solids gain of the frozen samples compared to the untreated. Similarly, after a freezing pretreatment of pumpkin, Kowalska et al. (2008) observed higher water loss (*WL)* and solids gain (*SG)*, but better *WL/SG* ratio for non-treated pumpkin.

As well, **freeze-thawing** has been reported as one of the methods to increase mass transfer rate during foodstuff dehydration (Xin et al., 2021). Freeze-thawing consists of freezing foodstuff to their freezing point, followed by thawing at a higher temperature (Wu et al., 2017). Studies have shown the effectiveness of freeze-thawing pretreatment in improving drying processes (air drying, microwave drying, microwave-vacuum drying) (Feng et al., 2020; Magdalena Zielinska & Markowski, 2018). Feng et al. (2020) submitted garlic to freezing at -25 °C then thawing at +25 °C, followed by vacuum freeze drying at 0.518 mbar.

Results showed reduction of drying time (22.22%–33.33%) and the energy consumption (14.25%–15.50%). Results showed that frozen-thawed carrots (-20 °C and thawed at room temperature before air drying at 60 °C for 10 h) gave highest drying rate compared to untreated (Ando et al., 2016). Xin et al. (2021) were able to improve okra vacuum freeze-drying rates by freezing okra at -20°C and then thawing through 4 methods (water, ultrasound or air thawing, and air thawing + ultrasound). However, freeze-thawing as a pretreatment for osmotic dehydration is scarcely studied, notwithstanding Lazarides & Mavroudis (1995) study on frozen-thawed apple slices partially dehydrated with a sucrose osmotic solution. They did not find improvement of water loss after the freeze-thawing, but solids gain was promoted.

1.6.5.2 Coating on mass transfer during osmotic dehydration

Edible coatings are thin layers applied on food to protect and improve their quality, which are derived from bio-based materials such as polysaccharides, proteins, lipids or their composites (Kowalska et al., 2021). In osmotic dehydration, edible coating pretreatments could be used as a barrier to limit solids uptake while maintaining sufficient water removal (Khin et al., 2005). Coating is usually conducted by dipping the food material in the coating solution for about 30 seconds followed by a dipping into calcium-based solution (often $CaCl_2$ solution) as a stabilizer of the gel before drying between 5-15 min at ambient or high temperature. It is possible to repeat this procedure twice for a double coating result that may give better osmotic dehydration efficiency as was the case in the study on strawberries by Matuska et al. (2006). Some authors have used coatings on the food product prior to osmotic dehydration, such as those for apples (Azam et al., 2013; Emam-Djomeh et al., 2006; Jalaee et al., 2011), for strawberries (Matuska et al., 2006) together with mangoes (Rahman et al., 2020), potatoes (Lević et al., 2008), carrots (García et al., 2010) and papaya (Etemadi et al., 2020). These studies showed various interesting results depending on the type of coating agent used in minimizing solids uptake through edible coating pretreatment.

Emam-Djomeh et al. (2006) precoated apples with carboxyl-methyl cellulose (CMC) at different concentrations (0.5%,1%,1.5%,3%) in ternary glucose syrup solutions (30%, 40%, 50%) with sodium chloride (2%, 4%, 6%). Though water loss was less affected by coating, a better performance ratio of osmotic dehydration (*WL/SG*) for coated apples than uncoated

ones was observed. Coating agent of 1% CMC was thus proposed as the optimal condition for minimal solute uptake. Another study was conducted on apples rings coated with low-methoxyl pectinate (LMP), carboxyl-methyl cellulose (CMC) and corn starch, before dehydration in sucrose solution (50% and 60%) (Jalaee et al., 2011). They reported mainly higher *WL/SG* ratio with improvement of water loss for higher concentration of the coating agents 2% CMC, 3% LMP and 3% starch in 60% sucrose. Mango cubes of different maturities were pretreated with novel-gluten based coating before osmotic dehydration in 45, 55, and 65°Brix sucrose solutions (Azam et al., 2013). At concentration of 55 °Brix water loss was significantly higher for coated mangoes than uncoated ones, probably due to the use of the novel hydrophilic coating. Additionally, increasing chemical potential from 45 ° Brix to 55 °Brix, increased the chemical potential leading to higher water loss. While above 65 ° Brix, there is a case hardening that slows the water loss.

Coating pretreatment of fresh-cut fruits was reported to help prevent enzymatic browning, loss of natural solutes, microbiological growth and provide better texture (Khin et al., 2005; Kowalska et al., 2021). Coating is a promising technology for production of low-calorie products through osmotic dehydration. However, challenges occurred in terms of difficulty to form stable gel with polysaccharides, necessitating further addition of additives to the coating agent preparation, such as $CaCl_2$ for cross linking (Khin et al., 2005). It also may necessitate multiple dipping processes followed by drying of the fruit, which can lead to high cost and low acceptability from consumers who are looking for clean-label products with the least additives possible.

1.6.5.3 Ultrasound on mass transfer during osmotic dehydration

Ultrasound (US) represents a mechanical wave ranging from 20 kHz to 100 MHz frequency (Nowacka et al., 2014). It can be applied both on liquid and solid material and is divided into low and high frequency ultrasound (Rodrigues & Fernandes, 2007). High frequency (>100 kHz) provides low energy that does not damage the material and is applied in food quality monitoring (Awad et al., 2012). Whereas low frequency (20-100 kHz) discharges high energy which causes disruption of the material to which it is applied, and is used to enhance

processing such as enzyme inactivation, freezing, extraction processes, drying and osmotic dehydration (Povey & Mason, 1998; Witrowa-Rajchert et al., 2014).

Particularly in drying and osmotic dehydration processes, high frequency ultrasound is used as a pretreatment or during the process to enhance mass transfer. The mechanism involves alternative compression and expansion of the medium leading to bubbles formation that collapse rapidly causing localized heating and pressure (Witrowa-Rajchert et al., 2014). When bubbles collapse near a solid material, they released microjet at a speed of 200 m/s (Leonelli & Mason, 2010), consequently leading to microscopic channels that constitute new pathways for moisture transfer, thus improving drying of foodstuff (Nowacka et al., 2012).

Ultrasound pretreatment consists of immersing the foodstuff into distilled water in a ultrasound bath or applying a ultrasound probe into the bath for a short period of time (10-30 minutes (Fernandes & Rodrigues, 2009). Figure 1.6 depicts these two types of operating modes.

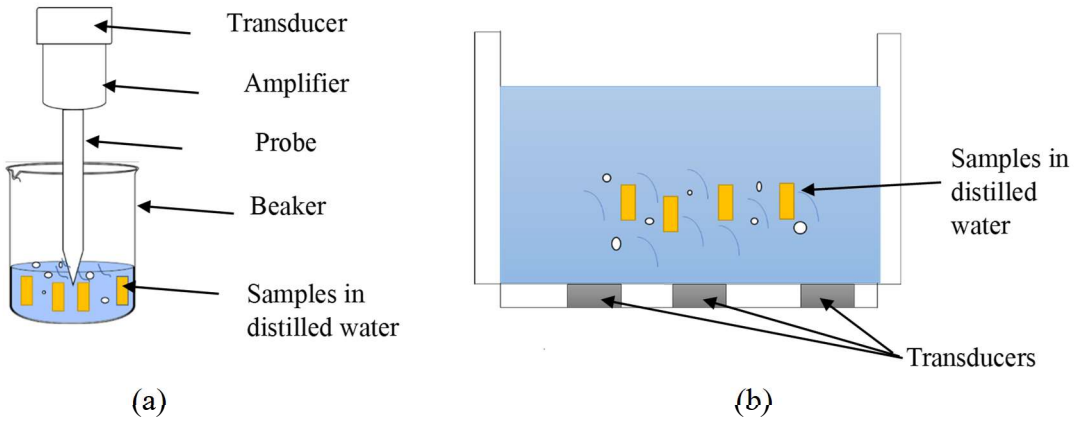

Figure 1.6: Schematic ultrasound application on foodstuff

(a) ultrasound probe; (b) ultrasound bath. Adapted from (Strieder et al., 2019)

Regarding osmotic dehydration, ultrasound pretreatment showed promising results. In a work by Goula et al. (2017) potatoes were subjected to four treatments in a combined maltodextrin and sodium chloride solution (30-70%): osmotic dehydration with and without agitation, osmotic dehydration with ultrasound assistance or ultrasound pretreatment. As a result, ultrasound pretreatment gave higher water loss and solids uptake than osmotic dehydration

with or without agitation. In another study by Nowacka et al. (2014), kiwifruit was pretreated by ultrasound at 35 kHz for up to 30 min before osmotic dehydration in 61.5% sucrose solution. Water loss and solids uptake increased with ultrasound pretreatment above 10 min treatment time. Additional microscopic analysis of kiwifruit microstructures showed the formation of microchannels responsible for facilitating the water outflow and increase in water diffusivity during osmotic dehydration. Moreover, similar mass transfer improvement due to ultrasound was reported by Bozkir et al. (2019) for persimmon fruit, Prithani & Dash (2020) for kiwifruit and Simal et al. (1998) for apples. Other authors have reported ultrasound positive effect on better preservation of color and bioactive compounds and texture as for ginger (Osae et al., 2019), for kiwifruits (Vallespir et al., 2019), and tomato (Corrêa et al., 2015).

1.6.5.4 Pulsed electric field effect on mass transfer during osmotic dehydration

PEF consists of the application of a non-stationary current regime with alternating pulse and pause periods during a fixed time (Cifuentes-Araya et al., 2011; Dufton et al., 2020) usually in the range of microseconds (Toepfl & Knorr, 2006). PEF is often applied to biological tissue (Gürsul et al., 2016) for modification of cells permeabilization and is recognized as one of the most popular among novel technologies in food processing (Ahmed & Alam, 2011). Tissue permeabilization occurred through electroporation phenomenon. When an external electric field is applied on a tissue up to the transmembrane voltage critical value, pores formation which is called electroporation, is induced consequently compromising its semi permeability (Balasa, 2016; Vorobiev & Lebovka, 2009). Figure 1.7 illustrates application of PEF on a foodstuff.

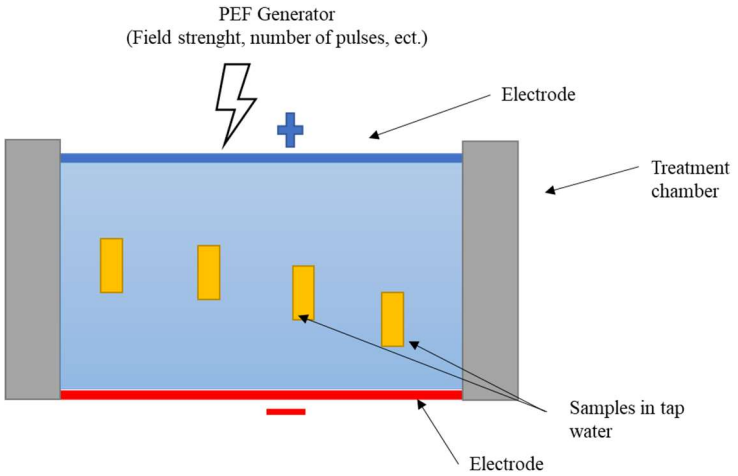

Figure 1.7: Pulsed electric field applied to foodstuff, adapted from (Baldi et al., 2021)

As shown in Figure 1.7, the food material is placed inside a treatment chamber consisting of 2 electrodes. Electrodes can be parallel plates, or cylindrical with coaxial and co-linear configurations. A pulsed power modulator provides required voltage through exponential decay or rectangular pulses forms (Toepfl & Knorr, 2006).

Numerous factors can determine the extent of PEF effects on the tissue : treatment intensity (field strength, pulse number, pulse duration or width, pulse shape and the total treatment time); tissue properties (cell size, cytoplasm conductivity), extracellular media, orientation in the electrical field, etc.. (Evranuz, 2011; Tylewicz, 2020). In terms of electric field strength (E), PEF treatment is classified as low (E = 100-200 V/cm), moderate (E = 300-1500 V/cm) and high (E > 1500 V/cm) (Bazhal & Vorobiev, 2000). The combination of the different parameters determine presence of temporary pores (low and moderate intensity) or irreversible pores (high intensity) in the tissue (Tylewicz, 2020).

Cell permeabilization after PEF treatment can be estimated from electrical conductivity and texture, or by microscopy analysis. The disintegration index (Z) can make use of the different measurements by the following calculation:

$$Z_m = \frac{m_t - m_i}{m_d - m_i} \tag{1.7}$$

where *m* is the type of measure (electrical conductivity, firmness, or other), and subindexes *t*, *i*, and *d* mean ‘at time *t*’, initial, and 'totally destroyed tissue’, respectively. Thermal treatments (freeze-thawing or blanching) are used as reference method to obtain ‘totally disintegrated tissue’. *Z* values vary from 0 to 1, an intact tissue would have a *Z* value of 0 while a totally disintegrated tissue, a value of 1 (Lebovka et al. 2002).

Cell permeabilization effect of PEF is used as a pretreatment to improve mass transfer rates during process such as extraction, pressing, drying and osmotic dehydration (Luengo et al., 2013; Ostermeier et al., 2018; Toepfl & Knorr, 2006; Zhu et al., 2015). Regarding the quality of PEF treated products prior to drying, it is generally of better quality compared to the untreated samples or other pretreatments such as thermal (blanching, freezing), enzymatic and mechanical (Vorobiev & Lebovka, 2009). As well, pulsed electric field was found to be less invasive than other pretreatments (Huang & Wang, 2009). This fact was proven in Taiwo et al. (2001), where mango tissue pretreated by the highest intensity of field was six times less damaged than with freeze-thawing pretreatment. For instance, red pepper pretreated by PEF (1.0–2.5 kV/cm, 1-4s) reached final moisture content 1 hour faster than untreated ones, consequently leading to better color retention (Won et al., 2015). Moreover, texture preservation was enhanced in carrots and parsnips after PEF pretreatment prior to drying due to the reduced drying time (about 21-28%) (Alam et al., 2018) .

Drying processes such as osmotic dehydration are limited by external and internal resistances. The internal resistance is linked to the cell matrix permeability (porosity) (Wiktor & Witrowa-Rajchert, 2012). Novel technologies such as PEF are sought for to create perforation in the cell structure reducing the internal resistance to the mass transfer and enhancing diffusivity during osmotic dehydration (Toepfl & Knorr, 2006).

Some PEF and osmotic dehydration conditions are shown in Table 1.6 indicating that in general, when PEF pre-treatment is applied on the plant tissue, improvement of mass transfer could occur during osmotic dehydration. Generally, PEF pretreatment is conducted at ambient temperature and most of the studies have reported only a slight increase of temperature due to PEF (less than 5-7 °C) (Ade-Omowaye et al., 2002; Amami et al., 2014; Dermesonlouoglou et al., 2016; Rastogi et al., 1999), which makes it a non-thermal method

good for nutrients and bio compound preservation. PEF compared to other pretreatments presented in Table 1.6 (high pressure, supercritical carbon dioxide) did not necessitate heating (Taiwo et al., 2001), thus leading to less damage to product and fresh-like product. As can be seen in Table 1.6 water loss was consistently improved while solids gain behavior did not show a consistent trend. For instance, sugar gain increased for apple, bananas, carrots (Amami et al., 2014), mango (Tedjo et al., 2002) and bell peppers (Ade-Omowaye et al., 2002) compared to respective untreated samples. But few authors reported low solids gain for strawberries in trehalose solution (Tylewicz et al., 2017) and kiwifruit in sucrose solution (Traffano-Schiffo et al., 2016), while water loss was enhanced in both cases. The variability on solids gain results, emphasizes the need for more research on optimizing PEF parameters towards modulating tissue permeability for a selective mass transfer aiming at controlling solids uptake according to desire objective, low or high solids uptake, in food material after osmotic dehydration.

Table 1.6: Some examples of PEF pretreatment for osmotic dehydration of fruits and vegetables

Fruits or vegetables	PEF and other pretreatments conditions	Osmotic dehydration (OD) or other drying conditions	Main findings for mass transfer and product quality	References
Apple, banana, carrots **Shape: discs** **Thickness: 0.85 cm (apples), 1 cm (carrots and bananas)**	0.90 kV/cm, 0.75s, 750 pulses, 15kJ/kg (apples) 0.30kV/cm, 0.05s, 500 pulses, 10 kJ/kg (bananas) 0.60 kV/cm, 0.05 s, 500 pulses, 19 kJ/kg (carrots) Pulse duration = 100 µs Samples were blotted in paper and placed between 2 electrodes of 2.9 cm diameter each Temperature rises due to PEF: no less than 7°C	Temperature: 25°C Concentration: 65% sucrose (w/w) Time: 4h Product/solution ratio: 1:3 Agitation: 0; 250; 500; 1000; 1500 rpm	Agitation alone increased *WL* and *SG* compared to static osmotic dehydration. But when agitation was combined with PEF it led to further increment of *WL* and *SG* for all three fruits. Browning was observed in carrots for high agitation and apples after PEF treatment and pigments leakage led to banana more yellowness.	(Amami et al., 2014)
Bell peppers **Shape: discs** **Thickness: 0.64 cm**	2 kV/cm, 1 to 50 pulses, 0.32 kJ/kg Pulse duration = 400 µs Temperature rises due to PEF: no less than 5°C Freeze-thawing:-28°C followed by thawing at room temperature	Temperature: 40 °C Concentration: 50 °Brix sucrose Time: 5h Product/solution ratio: 1:10 Agitation: Yes Air drying following OD: 60°C, 1 m/s, 5 h	Porosity increased in the order of freeze-thawing > PEF Pulses number of 5-10 pulses significantly increased *WL* Pulses number of 5-50 pulses increased *SG* PEF increased *WL* compared to freeze-thawing both after OD and air drying; inverse results were obtained for *SG* Freeze-thawing led to better vitamin C retention than PEF Increasing pulse number decreased vitamin C content	(Ade-Omowaye et al., 2002)
Carrots **Shape: discs** **Thickness: 1 cm**	0.22 kV/cm; 0.64 kV/cm; 1.09 kV/cm; 1.60 kV/cm; 5 pulses Frequency: 1 Hz Pulse duration (µs): 322; 336; 378; 405 Energy (kJ/kg): 0.04; 0.28; 0.86; 2.25 Temperature rises due to PEF: no less than 1°C	Temperature: 40 °C Concentration:50°Brix sucrose Time: 5h Product/solution ratio: 1:25 Agitation: N/A OD followed by vacuum oven drying: 70 °C for 18 h	Carrot tissue disintegration index and tissue softening increased exponentially with PEF field strength up to 1.09 kV/cm and did not change. Furthermore *WL* and *SG* increased significantly with field strength up to 1.09 kV/cm	(Rastogi et al., 1999)

Table 1.6: *Continued*

Fruits or vegetables	PEF and other pretreatments conditions	Osmotic dehydration (OD) or other drying conditions	Main findings for mass transfer and product quality	References
Kiwifruit **Shape: Cylinders** **Thickness:10 mm length**	100 V/cm; 250 V/cm; 400 V/cm; 60 pulses Pulse duration: 100 µs Frequency: 100 Hz	Temperature: 25 °C Concentration: 61.5°Brix sucrose Time: 2h Product/solution ratio: 1:4 Agitation: N/A	*WL* was higher for PEF treated kiwifruit than untreated ones. Up to 250 V/cm, no significant difference was found for *WL* between PEF treated samples Inversely, PEF treated samples showed lower *SG* compared to untreated ones	(Traffano-Schiffo et al., 2016)
Kiwifruit **Shape: discs** **Thickness: 6.83 mm**	0.7 kV/cm; 1.1 kV/cm; 1.8 kV/cm; 250 pulses Energy (kJ/kg): 8; 16.6; 42.3 Pulse duration: 15 µs, Frequency: 300 Hz Temperature rises due to PEF: no less than 5°C *PEF was applied on whole peeled kiwifruit	Temperature: 25 °C, 35°C, 45°C Concentration: 30% glycerol + 20% high DE maltodextrin + 10% trehalose +2% ascorbic acid + 1.5%calcium chloride + 1%sodium chloride + 0.2%citric acid Time: 4h Product/solution ratio: 1:5 Agitation: N/A *OD was applied on Kiwi discs	PEF increased *WL* and *SG* of kiwifruits compared to untreated ones Firmness decreased with field strength up to 1.1 kV/cm Water activity decreased with the field strength Vitamin C was decreased with PEF treatment but remained at 77% even at the maximum field strength	(Dermesonlouoglou et al., 2016)
Mango **Shape: disk** **Thickness:8 mm**	PEF: 2.67 kV/cm, 100 pulses, 1 Hz, pulse duration: 840 µs; High Pressure (HP): 600 MPa, 90 °C Super critical carbon dioxide ($ScCO_2$): 62 MPa, 95 °C	Temperature: 40 °C Concentration: 50 °Brix sucrose Time: 5h Product/solution ratio: 1:25 Agitation: N/A	The type of pretreatment is more predominant on mass transfer than the cell permeabilization degree. PEF and HP enhanced *WL*, while it was not the case for $ScCO_2$ *SG* was increased by all pretreatments, but PEF led to less sugar uptake among the pretreatement methods	(Tedjo et al., 2002)
Strawberries **Shape: rectangular** **Thickness:2 cm**	100, 200, 400 V/cm, 100 pulses Pulse duration: 100 µs, frequency: 100 Hz, energy input: 0.123kJ/kg	Temperature: 25°C Concentration: 40 % sucrose, 40 % trehalose Time: 2h Product/solution ratio: 1:4 Agitation: Yes	PEF increased *WL*. A field strength of 400 V/cm led to highest WL (50%- *SG* of PEF sample was higher than untreated for sucrose In trehalose PEF decreased *SG* at 200-400 V/cm, SG decreased compared to the untreated	(Tylewicz et al., 2017)

1.6.6 Advantages and limitations of osmotic dehydration

Foodstuffs have in general high moisture content which require energy input during drying for removal due to the high latent heat of vaporization of water (Bekele & Ramaswamy, 2010). Osmotic dehydration presents an interesting alternative to conventional drying because water can be removed without phase changing and therefore it does not necessitate excessive heating and can be conducted at mild temperature, lower than 50°C (Ahmed & Alam, 2011). After osmotic dehydration of apples and carrots at 40 °C followed with syrup reconcentration, energy consumption was at least two times lower than convection drying at 70°C (Lenart, 1992; Lenart & Lewicki, 1988). Osmotic dehydration reduces by 30-50% the moisture content of a food material (Bekele & Ramaswamy, 2010) depending on the process conditions. Additionally, solute uptake lowers water activity. Consequently, further treatments such as air drying, or vacuum drying can be moderate to remove the partial water remained after osmotic dehydration leading to time and energy savings (Tortoe, 2010). As energy becomes more challenging in industry, processing method that leads to less consumption are sought for. According to Beedie (1995), saving 1% energy could lead to 10 % of profits in the industry. Furthermore, in developing countries where energy is expensive, osmotic dehydration could be of benefits substituting or complementing air drying or sun drying for less energy input as demonstrated by Levi et al. (2007) which successfully reduces energy needed for papaya drying after osmotic dehydration pretreatment. Osmotic dehydration has quality and economic benefits that promotes its utilization by industrial and small entrepreneurs alike throughout the world. The process implementation requires only simple equipment, therefore, its initial cost is available not only for industries but also for small production units (Bchir et al., 2011). As for its advantages on organoleptic properties, osmotically dehydrated products are reported to have better color, flavor, taste, texture and nutrients retention close to the fresh product (Ahmed et al., 2016). Decrease of color damage was observed in osmotically dehydrated apples and bananas compared to the untreated samples (Krokida et al., 2000). During osmotic dehydration, the food is immersed in an osmotic solution, which prevents the contact of oxygen, thus inhibiting enzymes responsible of browning (Ahmed et al., 2016). A study by Dermesonlouoglou et al. (2007) demonstrated the protective effect of osmotic dehydration on acid ascorbic and carotenoids retention of tomato. The solute incorporation along with the water loss, helps prevent the food structure

collapse and retain its texture. Some fruits cannot be eaten in their fresh form because of their high acidity (i.e., cranberries), thus osmotic dehydration helps to enhance their taste by reducing the acidity. It is also possible to modulate food chemical composition by incorporating high quality nutrients into the final product (Sravani & Saxena, 2021). Through osmotic dehydration of plums, Klewicki & Uczciwek (2008) have succeeded to partially replace sucrose with fructo-oligosaccharides that lowers the energy uptake to 12-37%. The possibility to re-use the osmotic solution or its further utilization in the beverage industry (Sravani & Saxena, 2021) or animal breeding makes the process a good candidate for a circular economy.

Despite many advantages of osmotic dehydration, it has limitations due to the lengthy process and the large amount of solids (usually sugars) into the food material that is undesirable most of the time (Khin et al., 2005; Lazarides, 2001). These limitations represent a challenge for industry in terms of optimization of product yields and to cater nowadays consumers' preferences for low calorie and healthier processed foods. To increase dehydration rate and reduce solute uptake, parameters related to osmotic dehydration such as food material properties and osmotic solution composition are sought for optimization. Pretreatments such as coating, pulsed electric field, ultrasound, freezing, freeze-thawing are used to improve cellular structure of food tissue before osmotic dehydration to facilitate water loss and reduce dehydration time which might lead to solute uptake decrease. Osmotic solutions compositions can be chosen in terms of solute molecular weight and solution inherent properties such as viscosity, concentration, interactions between solutes to modulate the solutes diffusivity aiming at low solids uptake. Also, the Biot number is a parameter that could be used to identify mass transfer controlling mechanisms and its value can help to choose appropriate conditions to obtain the desired final product characteristics.

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Chapitre 2: Problématique, hypothèse et objectifs

2.1. Problématique

La déshydratation osmotique est un procédé qui a démontré avoir plusieurs avantages dans la production de produits alimentaires de meilleure qualité nutritionnelle et organoleptique. Cependant, dans le processus de déshydratation osmotique, la sortie d'eau s'accompagne simultanément d'une entrée de sucres provenant de la solution osmotique et entrainée par la différence de potentiel chimique entre la solution osmotique et le tissu du matériel cellulaire à déshydrater. Cette entrée devrait être contrôlée car de plus en plus les consommateurs ne tolèrent plus les produits trop sucrés, particulièrement les produits contenant des sucres possédant un apport calorique élevé tel que le sucrose. La revue de littérature a permis d'identifier des facteurs internes et externes importants pour réduire l'entrée de sucres, tels que la structure cellulaire, l'épaisseur du produit, les caractéristiques de la solution osmotique, la température, l'agitation, les prétraitements, etc. Bien qu'il existe des travaux sur l'utilisation des solutions osmotiques alternatives au sucrose, comme le miel et le sirop d'érable, l'effet combiné du choix de la solution osmotique alternative et des paramètres de la déshydratation osmotique a été peu étudié. De plus, le sirop d'agave reste peu utilisé surtout dans la déshydratation osmotique des mangues. Il peut cependant par sa composition (prébiotique, vitamines, minéraux…) améliorer la qualité nutritionnelle et améliorer l'attractivité et la valeur commerciale du produit final. Ainsi, moduler les paramètres de la déshydratation osmotique et utiliser le sirop d'agave pourraient contribuer à produire des mangues ayant une valeur calorique moins élevée et enrichie en composés bénéfiques pour la santé des consommateurs.

2.2. Hypothèse de l'étude

La combinaison de l'épaisseur de la mangue, la composition en solutés et la viscosité de la solution osmotique, les prétraitements tels que la congélation/décongélation et le champ électrique pulsé permettent d'identifier des conditions optimales pour une diffusion sélective lors la déshydratation osmotique de la mangue.

2.3. Objectif général

L'objectif général de ce projet est d'étudier la déshydratation osmotique de la mangue en vue d'obtenir un produit ayant une faible teneur en sucre ajoutée en utilisant des ingrédients naturels pour une éventuelle contribution à la création de snacks 'santé'.

2.4. Objectifs spécifiques

Trois objectifs spécifiques ont été déterminés pour la vérification de l'hypothèse émise :

- Caractériser la rhéologie et la viscosité des solutions osmotiques composés de sucrose, fructose, glucose, modèle d'agave avec ou sans ajout d'inuline ou de *xanthan gum* et le *corn syrup solids.*
- Étudier l'impact de l'épaisseur des tranches de mangues et de la rhéologie/viscosité des solutions osmotiques sur le transfert de matières lors de la déshydratation osmotique des mangues.
- Étudier le profil de sucres individuels dans la mangue après la déshydratation osmotique dans les différentes solutions osmotiques utilisées.
- Étudier l'impact de la congélation/décongélation ainsi que le nombre d'impulsions et l'intensité du champ électrique pulsé sur l'accélération du transfert de masse lors de la déshydratation osmotique de la mangue.

Transition vers l'article 1

La revue de littérature a montré les paramètres pertinents à explorer pour avoir une réduction de la rentrée de sucres tout en maintenant une sortie d'eau significative durant la déshydratation osmotique. Dans le Chapitre 3, l'étude de **l'épaisseur de l'échantillon ainsi que de la rhéologie de la solution osmotique et l'ajout de molécules à haut poids moléculaire dans cette solution**, sera présentée. Ce chapitre a fait l'objet d'une publication au *Journal of Food Process Engineering* :

Zongo, A. P., Khalloufi, S., & Ratti, C. (2021). Effect of viscosity and rheological behavior on selective mass transfer during osmotic dehydration of mango slices in natural syrups. *Journal of Food Process Engineering*, 44(7), e13745.

Les résultats obtenus ont été présentés par poster au :

- North American Biological and Engineering Conference *(*NABEC) du 16 au 19 juin 2019. Titre: *Sugar intake reduction during osmotic dehydration of mango.*
- International Commission of Agricultural and Biosystems Engineering (CIGR) du 11 au 14 mai 2021. Titre: *Impact of rheological properties on sugar uptake during osmotic dehydration of mangoes.*

Chapter 3: Effect of viscosity and rheological behavior on selective mass transfer during osmotic dehydration of mango slices in natural syrups

3.1 Résumé

Une étude sur la déshydratation osmotique des mangues a été conduite dans le but de produire des mangues déshydratées par osmose à teneur en sucres ajoutés faibles et de tester l`utilisation de sirops naturels comme solutions osmotiques. Différentes solutions osmotiques à 60°Brix ont été utilisées (préparées avec du sucrose, du glucose, du fructose, des solides de sirop de maïs et du sirop d'agave avec ou sans ajout de gomme xanthane ou d'inuline) lors de la déshydratation osmotique à 40°C de tranches de mangue (0.4 et 1.5 cm d'épaisseur). Le comportement rhéologique et la viscosité des différentes solutions osmotiques ont été déterminés à 22°C et 40°C. Selon les résultats, l'augmentation de la viscosité et de l'épaisseur de l'échantillon a permis de réduire le gain en sucre tout en maintenant une perte en eau adéquate. Le gain en sucre le plus élevé a été enregistré pour les solutions de sucrose, de glucose, de fructose, et les solutions de sirop d'agave. Tandis que, le plus faible gain en sucres a été constaté, pour les solutions de sirop de solides de maïs et le sirop d'agave contentant la gomme xanthane. L'impact de l'augmentation de la viscosité apparente sur le gain en solides était plus prononcé pour les échantillons à faible épaisseur, indiquant l'importance du nombre de Biot sur la sélectivité du transfert de matières lors de la déshydratation osmotique. Cette étude a permis de produire des mangues osmotiquement déshydratées à faible teneur en sucre en utilisant une solution osmotique naturelle telle que le sirop d'agave enrichi avec des ingrédients. Dans cette étude, le rôle de la viscosité de la solution combinée à l'épaisseur de l'échantillon dans la réduction du gain de solides pendant la déshydratation osmotique a été élucidé. De plus, le modèle de sirop d'agave utilisé dans cette recherche, est particulièrement intéressant en raison de sa richesse en vitamines et prébiotiques (inuline) qui rehaussent les valeurs nutritives de la mangue. Cette étude aiderait les industries à offrir des collations plus saines, en particulier pour les consommateurs qui souhaitent réduire leur consommation de sucre.

3.2 Abstract

Osmotic dehydration of mangoes was investigated for the reduction of solids gain and potential use of natural syrups as osmotic solutions. Different osmotic solutions at 60°Brix were used (made with sucrose, glucose, fructose, corn syrup solids, and agave syrup with or without added xanthan gum or inulin) during osmotic dehydration at 40ºC of mango slices (0.4 and 1.5 cm thickness). Rheological behaviour and viscosity of the different osmotic solutions were determined at 22ºC and 40ºC. According to the results, increasing the viscosity and the sample thickness helped to reduce the sugar gain while maintaining an adequate water loss. The highest sugar gain was found for sucrose, glucose, fructose, agave syrup solutions, and the lowest, for corn syrup solids solutions and xanthan gum added to agave syrup. The impact of increasing apparent viscosity on solids gain was more pronounced for thin samples, indicating the importance of the Biot number on selective mass transfer during osmotic dehydration. This research aims to obtain osmotically dehydrated mangoes with low sugar content by using a natural multicomponent solution such as an agave syrup with added ingredients. In this study, the role of solution viscosity combined to sample thickness in lowering solids gain during osmotic dehydration was elucidated. As well, agave syrup used in this research as a model, is particularly interesting due to its rich content in vitamins and prebiotic (inulin) which levels up the mango nutritious values. This study would help industrials to offer healthier snacks, in particular for consumers who wish to reduce their sugar intake.

3.3 Introduction

Mango (*Mangifera indica* L.) is one of the most consumed tropical fruits, with a production increasing each year (Araya-Farias et al., 2014; Jahurul et al., 2015; Jiménez-Hernández et al., 2017b; Yao et al., 2020). This fruit has a rich profile of vitamins (A, C, E, K, B1, B2, B3, B5, B6, B12), minerals (calcium, iron, phosphorus, potassium, magnesium, zinc, manganese), dietary fibers (cellulose, hemicellulose, lignin) along with antioxidants (vitamin C, ß-carotene, dehydroascorbic acid) (Maldonado-Celis et al., 2019a; Rocha Ribeiro et al., 2007; Sudha et al., 2015). Due to its high-water content and nutrients, fresh mango is a highly perishable fruit with up to 50% wasted during post-harvest period, storage, transport, and ripening (Islam & Absar, 2013; Maldonado-Celis et al., 2019a). On the other hand, processed mango enjoys a substantial worldwide trade (Gerbaud, 2016), in particular dried mango. Asian countries such as Philippines, Thailand and India dominate the worldwide dried mango market, however their products present some quality problems that should be addressed, such as too high sugar content (if osmotically dehydrated), too dry, dark color or low in nutrients (if convectively dried) and, in the case of Indian processed mangoes, considerably high levels of pesticides (CBI, 2020). As well, nowadays consumers are concerned about the reduction of their sugar intake and thus, innovations are required in the osmotic dehydration process to reduce the sugar gain of fruits while improving its nutritive and organoleptic values.

Osmotic dehydration is the immersion of a food product (fruits, vegetables, meat) in a hypertonic solution which creates a chemical potential gradient mainly due to osmotic pressure difference (Bui et al., 2009; Paes et al., 2019) resulting in the removal of water and the gain of solutes by the product (Ketata et al., 2013). Unlike most drying processes, osmotic dehydration allows reducing the moisture content of fruits with little use of energy, however it lowers only partially the moisture content of a foodstuff and requires further processing to stabilize the product so as to preserve it for longer terms (Araya-Farias et al., 2014). Hence, this process is used in the food industry as a pre-treatment prior to freezing, freeze-drying, vacuum drying, etc. (Lakshmishri Roy, 2015). Mild temperatures and absence of oxygen during osmotic dehydration help to better preserve the nutrients and colors than in traditional drying (Bui et al., 2009; Paes et al., 2019).

Several attempts have been undertaken in the last years to tackle the problems of reducing solids gain or/and increasing product nutritive values during osmotic dehydration. To reduce the sugar intake, increasing the viscosity of the solution has been investigated for orange and mandarin peels (Cháfer et al., 2001) melon cubes (Ferrari & Hubinger, 2008), apple cubes, (Assis et al., 2017) beetroot slices (Manivannan & Rajasimman, 2011), agar gels (Emam-Djomeh et al., 2001) and mango (Giraldo et al., 2003), with ambiguous results depending on the product and particularly, the temperature range at which experiments were done. Since temperature affects in an inverse manner the viscosity and diffusion coefficients, it could be possible for lower temperatures to have reduced sugar gain as osmotic solution viscosity increases, and the opposite effect for higher temperatures (Ferrari & Hubinger, 2008; Manivannan & Rajasimman, 2011). In terms of internal mass transfer, some researchers have explored the relationship between solute diffusion coefficients within the solid and the viscosity of the external osmotic solution (Emam-Djomeh et al., 2001; Giraldo et al., 2003),which is theoretically questionable since internal diffusion coefficients should be dependent on temperature and product structure, and not on external variables. Always with the objective of reducing solids uptake, (Azam et al., 2013; Jalaee et al., 2011) investigated edible coating before osmotic dehydration as a barrier for sugar diffusion in apple and mango. Polysaccharides such as chitosan, carrageenan, pectin and gums were also used as edible coatings to reduce the sugar gain during the osmotic dehydration of several fruits (Sulistyawati et al., 2020; Vargas et al., 2008). Sulistyawati et al. (2018) studied the effects of vacuum impregnation (VI) and high pressure (HP) pretreatments and adding pectin methylesterase (PME) with calcium to the sucrose osmotic solution on the quality of osmotic dehydrated mango of different ripeness. They found out that only osmotic solutions with PME addition significantly increased OD efficiency in VI or HP pretreated ripe mangoes due to a more rapid and homogenous penetration of PME and calcium into cells, forming a calcium-pectin gel leading to soluble solid gain reduction. Another recent study from the same researchers (Sulistyawati et al., 2020) confirmed that applying VI, and to a lesser extent adding PME with calcium to the osmotic solution, decreased the sugar gain, but without increasing vitamin C loss of osmo-dehydrated mangoes. In the same line of research, Sanjinez-Argandona et al. (2018) found that using calcium chloride during osmotic dehydration of mango lower the sugar uptake due to the formation of calcium pectate which

increases the cell stiffness thus reducing the mass transfer. Other scientific reports on the same subject found out that increasing the thickness of the product may reduce the sugar uptake, as observed during such is the case of the osmotic dehydration of statistical approach research done for mangoes (Madamba & Lopez, 2002) or kiwifruit (Cao et al., 2006).

In addition, research has been done to investigate alternative healthier/natural osmotic solutions (fruit juices, honey, maple syrup, etc.) to traditional ones such as sucrose solutions in order to increase the nutritive/organoleptic value of the OD products (Chauhan et al., 2011; Gupta et al., 2012; Joshi et al., 2011; Samborska et al., 2019) . To improve nutritional quality of mangoes, an inulin pequin oleoresin emulsion was used to enrich mango with polyphenol and inulin during osmotic dehydration, with results suggesting that this emulsion made it possible to incorporate microcapsules of inulin into mango tissue (Jiménez-Hernández et al., 2017b). However, there is a scarce information about the use of agave syrup as osmotic solution in the available scientific literature. Agave syrup contains high percentage of fructose (reported to be 55% to 90%, (Willems & Low, 2012)), it also contains sucrose and glucose, but is gaining attention because of its richness in micronutrients (Edwards et al., 2016) and prebiotic inulin (>0.5% (Corrales Escobosa et al., 2014b). According to Corrales Escobosa et al. (2014b), high fructose agave syrups present as well bacteriostatic activity against *Bacillus Subtilis* and *E. coli.* A previous study made by St-Pierre et al. (2014) showed that agave syrup has similarities with maple syrup with respect to bioactive components and insulin response, hence both can be used as natural alternatives to the traditional sweeteners like pure sucrose.

Therefore, the objective of this research work is to explore the combined effect of osmotic solution viscosity and sample thickness on selective mass transfer during osmotic dehydration of mango, to reduce the solids uptake in different osmotic solutions including agave syrup and corn syrup solid solutions.

3.4 Materials and methods

3.4.1 Mango samples

Fresh and firm Tommy Atkins mangoes (purchased in a local grocery) were used for the osmotic dehydration experiments. Mangoes were stored at room temperature up to four days towards ripening at 12-14° Brix before further processing. Then, mangoes were washed, peeled, and their flesh was manually cut with cutters in cuboid shape (2.5 cm length by 2 cm width). Finally, mango cuboid samples were sliced at 0.4 and 1.5 cm thicknesses with a mechanical slicer.

Initial refraction index of mango samples was measured on mango puree with an Atago Pocket refractometer PAL-1 (Tokyo, Japan). Then, samples were stored at -60°C in a -86C Freezer Forma Scientific freezer (USA) before osmotic dehydration experiments. Some frozen mangoes were weighed and lyophilized in a Freeze Mobile (25L EL, Virtis, Gardiner, NY, USA) for 72 hours at a shelf temperature of 30°C under vacuum (4 Pa), to estimate their dry mass and thus, the initial moisture content.

3.4.2 Osmotic solutions

Eight different osmotic solutions at 60 °Brix were prepared for osmotic dehydration experiments: sucrose, glucose, fructose, corn syrup solids (CSS), agave syrup model (AS), AS with added 0.1% or 0.3% xanthan gum, and AS with added 5% inulin. Fructose, sucrose, glucose (dextrose) and corn syrup solids (Clintose 24) were bought at Farinex (Canada, Quebec), while polysaccharides (xanthan gum and inulin), at Biovea (from Rawgoods, USA). The corresponding amount of distilled water and solids (sucrose, glucose, fructose, corn syrup solids) were mixed at room temperature to obtain the osmotic solutions.

The agave syrup model (from now on it will be called just ‘agave syrup’, AS) was used to control the homogeneity of the solution. It was prepared as a mixture of sugars and distilled water to mimic the sugar composition of real agave syrup. High-performance liquid chromatography (HPLC) analysis was carried out on agave syrup (Unicornio, Jarabe de Agave de origen Orgánico, Mexico) to determine its composition. HPLC results indicated

that agave syrup contained mainly fructose (79%±0.32) and glucose (20%±0.13), with a low amount of sucrose (1% ±0.2), which are close to literature values (Corrales Escobosa et al., 2014a).

Xanthan gum was added to the agave syrup at 0.1% and 0.3% to create osmotic solutions with higher viscosity. Inulin was added at 5% to the agave syrup to study its effect on the mass transfer, but also taking into account that inulin is a prebiotic naturally present in agave syrup at concentrations higher than 0.5% (Corrales Escobosa et al., 2014a). The method used to dissolve xanthan gum and inulin in agave solutions follows. Xanthan gum was weighed and added to the monosaccharide's powders, then the mixture was mixed thoroughly before adding distilled water at ambient temperature, mixed thoroughly again, and heated up under continuously stirring to obtain a homogeneous solution. A separate solution of inulin was prepared in hot water (80 °C), then added to the osmotic solution containing the other monosaccharides. The mixture was again heated up, under constant stirring to obtain a homogeneous solution. The refractive index of the solutions was determined by using an Atago Pocket refractometer PAL-2 (Tokyo, Japan).

3.4.3 Rheological determinations

A Brookfield rheometer R/S plus (Harlow Essex, England, UK) was used to determine apparent viscosity, shear rate and shear stress of the different osmotic solutions at 22 and 40 °C. Concentric coaxial cylinder #CC25 was used for more viscous solutions, such as corn syrup solids and agave syrup with added xanthan gum, and for those solutions having lower viscosity (i.e., sucrose, glucose, fructose and agave syrup), cylinder #CC40. The temperature of the solution was controlled by connecting the Brookfield R/S concentric coaxial cylinder containing the solution to a water bath which maintained the target temperature. The data was collected with software Rheo3000 connected to the Brookfield R/S.

Shear stress (τ) was represented as a function of shear rate ($\dot{\gamma}$) by the power-law equation, which is considered a special case of the Herschel–Bulkley general model having yield stress equal zero (Steffe, 1996):

$$\tau = K\,(\dot{\gamma})^{n} \qquad (3.1)$$

where K (Pa s^{-n}) and n (--) are the consistency and flow indexes, respectively. From this equation, apparent viscosity, μ_{app}, can be estimated as:

$$\mu_{app} = K\,(\dot{\gamma})^{n-1} \qquad (3.2)$$

3.4.4 Osmotic dehydration experiments

Osmotic solution was heated up to 40°C in a Fisher Scientific bath (model Isotemp 1016 S, USA, Pittsburgh), with bath agitation corresponding to 15 L/min measured with water. Frozen mango slices (0.4cm or 1.5cm thickness) were weighed (5g ±1 or 24g ± 1, respectively) and put individually in identified metallic wire rack cages. The cages with mango samples were then immersed into the osmotic solution with slight agitation for up to 4 or 8 hours for 0.4cm and 1.5cm thickness, respectively. The ratio of samples/solution was above 1/100 to maintain the proper level of the bath, this also helped prevent dilution of the solution. During the osmotic dehydration process, individual mango samples were taken each 30 minutes, rinsed quickly with distilled water, gently tapped with paper, and then weighed.

Osmotic dehydrated samples were afterwards lyophilized in a Freeze Mobile (25L EL, Virtis) at 30°C and 30 millitorr vacuum for 72 hours. Freeze-dried samples were then weighed (Mettler Toledo AB104-S, Greifensee, Switzerland) in order to obtain their dry mass (M_d). Solids Gain (SG) and Water Loss (WL) represent respectively the water removed and the solids uptake from the mango samples after osmotic dehydration, based on initial mass of mango samples (Panagiotou et al., 1999):

$$SG\ (\%) = 100 * \frac{M_{df} - M_{do}}{P_0} \qquad (3.3)$$

$$WL\ (\%) = 100 * \frac{(P_o - M_{do}) - (P - M_{df})}{P_0} \qquad (3.4)$$

where M_{d0} is the initial dry matter (g); M_{df}, final dry matter (g); P_0, initial sample mass (g), and P, the final sample mass (g).

Equations (3.5) and (3.6) described below were used for correlation of the osmotic dehydration data kinetics of mangoes. Parameters WL_{eq} and SG_{eq} represent the maximum water loss and sugar gain respectively (an estimate of their values at equilibrium), t is the time (hours) and b_1 and b_2 are parameters related to the osmotic dehydration kinetics:

$$WL = \frac{WL_{eq}\ t}{(b_1 + t)} \tag{3.5}$$

$$SG = \frac{SG_{eq}\ t}{(b_2 + t)} \tag{3.6}$$

Experimental data was fitted to mathematical models (Equations 3.5 and 3.6) with a non-linear regression wizard function (Sigmaplot v.14, 2017). Standard error of estimate was an output result from the regression software which measures the 'goodness' of fitting (average error between predicted and experimental values) and will be provided together with Equations 3.5 and 3.6 parameters.

Maximum equilibrium values were approximated from WL_{eq} and SG_{eq} parameters, from which osmotic dehydration efficiency (*ODE*) was estimated:

$$ODE = \frac{WL_{eq}}{SG_{eq}} \tag{3.7}$$

3.4.5 Data and Statistic analysis

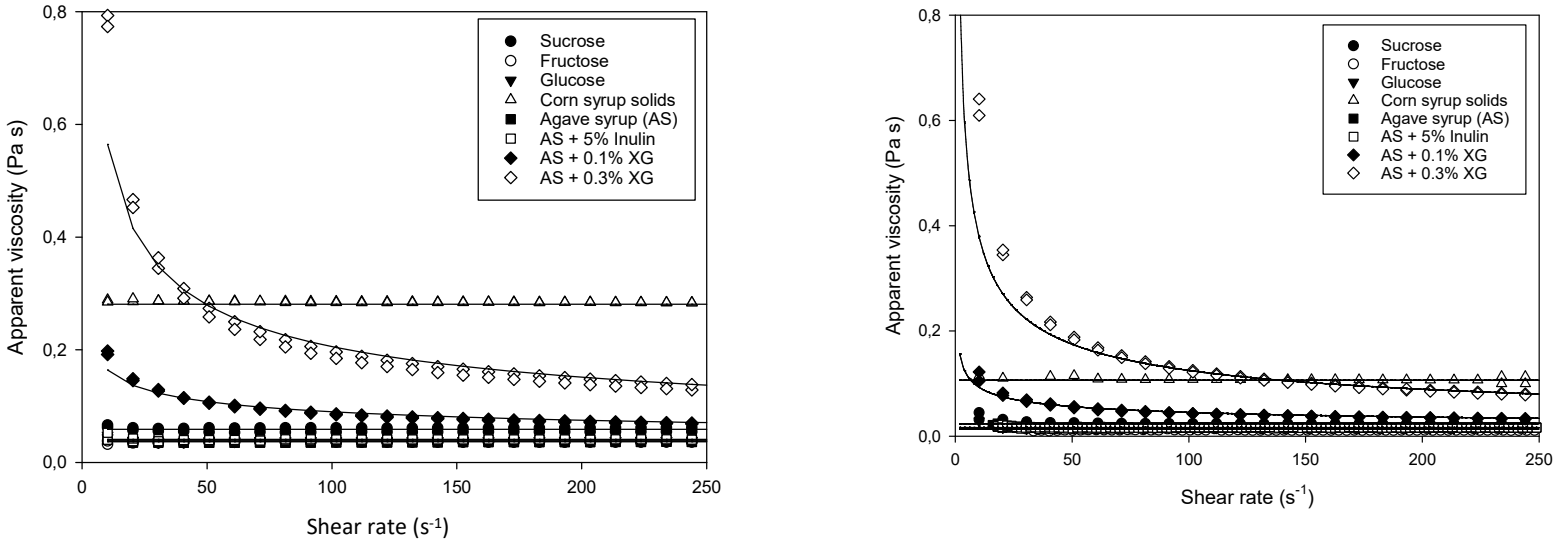
Osmotic dehydration experiments were repeated three times to estimate the mean value and the standard error used to represent the kinetic curves. The statistics analysis was made with Rstudio software (RStudio-1.2.5033). A *p*-value adjustment was made with Tukey test for comparing family of 4 estimates. The confidence level used was 95%.

3.5 Results and discussion

3.5.1 Rheology of osmotic solutions

Figure 3.1 shows the apparent viscosity of the different osmotic solutions used in this study (sucrose, glucose, fructose, corn syrup solids, and agave syrup alone or with added 0.1 or 0.3% xanthan gum (XG) or added 5% inulin (I)) at two temperatures. For most osmotic solutions (but those having added xanthan gum), viscosity remains constant with the shear rate indicating Newtonian behavior. When xanthan gum is added to agave syrup (even at minimum concentrations), solutions' behavior become Shear-thinning with a decreasing apparent viscosity as a function of shear rate (Steffe, 1996).

The information gathered in Figure 3.1 shows that different carbohydrate mixtures at same concentration (60° Brix) give osmotic solutions which are distinct not only through their rheology behavior, but also through their viscosity values. Osmotic solutions with low viscosities are those made from fructose (0.037 and 0.0136 Pa.s at 22 and 40ºC, respectively), agave syrup presenting a high percentage of fructose (0.038 and 0.0166 Pa.s at 22 and 40ºC, respectively), glucose (0.039 and 0.0164 Pa.s at 22 and 40ºC, respectively), and agave syrup with 5% inulin (0.041 and 0.016 Pa.s at 22 and 40ºC, respectively). Viscosity results of solutions made from fructose, glucose and agave syrup with or without 5% inulin were not significantly different (p>0.05) at each temperature. In comparison to fructose and glucose, sucrose solution has the highest viscosity (p <0.005) since it is composed of two molecules (fructose and glucose). The tendency and values of viscosity found for those osmotic solutions made of simple sugars are in good agreement with previously published data by Telis et al. (2007).



(a) (b)

Figure 3.1: Apparent viscosity of different osmotic solutions at (a) 22° C and (b) 40°C

The solid lines in the graphs are the regression model predictions obtained with Equation (3.2)

Solutions made from corn syrup solids are composed of long chains of maltodextrin (Helstad, 2019) and thus, have a remarkably high viscosity at both temperatures (0.28 and 0.11 Pa.s at 22 and 40°C, with $p<0.001$), which is 5 to 8 times those of sugar solutions. Agave syrup solutions containing mainly fructose and in lower proportion glucose (as determined in the present work by HPLC, please refer to Materials and Methods section) have similar viscosity to fructose solutions (0.038 and 0.0166 Pa.s at 22 and 40°C, respectively, with $p<0.05$). However, adding xanthan gum to agave syrup solutions changed the rheological behavior to Shear-thinning and increased the viscosity as well. For instance, at 22ºC and in practical absence of movement (10 s^{-1} shear rate), viscosity values of 0.1 and 0.3% XG added agave syrup were 0.133 Pa.s and 0.516 Pa.s, respectively, while at 40ºC, 0.065 and 0.381 Pa.s, representing highly significant increases in viscosity values. Even at high shear rates (up to 600 s^{-1}), agave syrups with xanthan gum present high viscosity values, 0.058 and 0.095 Pa.s for 0.1 and 0.3% XG, respectively at 22ºC, and at 40ºC, 0.027 and 0.052 Pa.s. With respect to corn syrup solids solutions viscosity curves, Figures 3.1a and 3.1b show an intercept with added 0.3%XG agave syrup solutions curves at shear rates of 50 s^{-1} and 130 s^{-1} for 22 and 40°C, respectively. This means that at low shear rates, 0.3% XG added agave syrups have higher viscosities than corn syrup solids solutions, and at high shear rates, the opposite. This characteristic of xanthan gum agave solutions makes their use in the industry beneficial only if shear rate is well monitored. In practice, depending on the power of mixing (shear rate) during osmotic dehydration, the industry has the opportunity to obtain the most appropriate viscosity desired for the targeted application. With respect to the addition of inulin, although it is also a long-chain polysaccharide, results in Figure 3.1 did not show a significant increase in the viscosity of agave syrup. In fact, inulin is known as not affecting the viscosity at low concentration, but it may enhance viscosity when its concentration exceeds 30% (Anderson-Dekkers et al. 2021).

The impact of temperature on viscosity of osmotic solutions is noticeable, with a reduction by half when the temperature changes from 22 °C to 40 °C (Figures 3.1 a, b). Similar results were found in the literature for sugar solutions at the same concentration (Telis et al., 2017).

Fitting parameters K and n of the Herschey-Buckley equation (Equation 3.1) are shown in Table 3.1. As can be observed, all the tested osmotic solutions were Newtonian ($n = 1$) but

for those formed by adding xanthan gum to agave syrup, which had $n < 1$ (Shear-thinning), confirming the experimental results shown previously. Increasing the concentration of XG, decreased the n parameter further from 1, which shows the major impact of xanthan gum (even at low concentrations) on the depart of the agave syrup from Newtonian ideality. Temperature has a marked impact in consistency parameter K, but a lesser effect on flow parameter n.

Predictions of apparent viscosity using Equation (3.2) and parameters K and n from Table 3.1, are shown in Figure 3.1 together with experimental data at 22°C and 40°C. As can be seen, there is a very good agreement between predicted and experimental values for all osmotic solutions tested, confirming the excellent coefficients of determination shown in

Table 3.1: Parameters K et n of the Herschel-Bulkley equation (Equation 3.1)

Osmotic solutions (22 °C)	**r^2**	***K* (Pa.s^n)**	***n***	**Type**
Sucrose	0.9998	0.0588	1	Newtonian
Fructose	0.9997	0.0368	1	Newtonian
Glucose	0.9996	0.0395	1	Newtonian
Corn syrup solids (CSS)	0.9999	0.2807	1	Newtonian
Agave syrup (AS)	0.9995	0.0376	1	Newtonian
AS + 5% Inulin	0.9997	0.0410	1	Newtonian
AS + 0.1% Xanthan gum	0.9993	0.3020	0.737	Shear thinning
AS + 0.3% Xanthan gum	0.9933	1.5740	0.558	Shear thinning
Osmotic solutions (40 °C)	**r^2**	***K* (Pa.s^n)**	***n***	**Type**
Sucrose	0.9991	0.0235	1	Newtonian
Fructose	0.9931	0.0136	1	Newtonian
Glucose	0.9989	0.0164	1	Newtonian
Corn syrup solids (CSS)	0.9999	0.1062	1	Newtonian
Agave syrup (AS)	0.9949	0.0166	1	Newtonian
AS + 5% Inulin	0.9984	0.0169	1	Newtonian
AS + 0.1% Xanthan gum	0.9997	0.1920	0.686	Shear thinning
AS + 0.3% Xanthan gum	0.9955	1.1650	0.515	Shear thinning

3.5.2 Water loss and solids gain during osmotic dehydration

Figure 3.2 presents the experimental data of water loss and solids gain during dehydration of mango samples in 60° Brix osmotic solutions made from sucrose, fructose, glucose, and agave syrup.

Curves of water loss (*WL*) and solids gain (*SG*) show the typical tendency of osmotic dehydration over time with a steep slope in the first hour for 0.4 cm thickness (Figure 3.2 a, b) and in the first 2 hours for 1.5 cm thickness (Figure 3.2 c, d). The increase rate slows down after the initial raise followed by a 'plateau' indicating that equilibrium has been achieved and no net mass transfer is possible. The equilibrium times happened after 2 and 8 hours (*WL*), and after 2.5 and 5 hours (*SG*), for samples having 0.4 and 1.5 cm thickness, respectively. Floury et al. (2008), during osmotic dehydration of mango cubes (1 cm) in a sucrose solution (40 Brix) at 40ºC, found that equilibrium arrived at longer times (11 hours for *SG* and more than 14 h for *WL*). While the temperatures are similar, sucrose concentration of this study (60 °Brix) is higher than the one used by Floury et al. (2008). This difference in Brix could explain the long equilibrium times observed by Floury et al. (2008). Closer equilibrium times to those presented in this study (1.5 hours for *SG* and 2.5 hours for *WL*) were provided by Jiménez-Hernández et al. (2017b) after osmotic dehydration of mango slices (0.5 cm) in a 60 °Brix sucrose solution at 40ºC. No significant difference ($p>0.05$) for *WL* and *SG* were found for osmotic solutions made of agave syrup with or without added inulin (5%) for 0.4 cm thickness (Figure 3.2 a, b), as for *SG* in mangoes having 1.5 cm thickness (Figure 3.2 d). However, *WL* in 1.5 cm mangoes dehydrated in agave syrup solutions with added inulin (5%) (Figure 3.2 c) was found significantly higher than in simple agave syrup solutions ($p = 0.0327$).

Increasing sample thickness, lowered solids gain and water loss. Nevertheless, this effect was more pronounced for *SG* (reductions of the order of 65%, Figure 3.2 b, d) than *WL* (reductions of the order of 18%, Figure 3.2 a, c). For mango slices having 0.4 cm thickness, the different osmotic solutions made of sugars, did not provide a significant impact on *WL* or *SG* ($p>0.05$) with final average values of 55% and 45%, respectively (Figure 3.2 a, b). For samples with 1.5 cm thickness, however, sucrose solutions significantly lowered *SG* to 12% ($p< 0.05$), while for fructose, glucose and agave syrup, the gain was between 15 to 20% (Figure 3.2 c, d). This could be due to the high molecular weight of sucrose, which is a disaccharide, while the other solutions are made of monosaccharides. Indeed, high molecular weight may decrease diffusion coefficients in liquid and solids for this type of sugar. According to previous research, small molecules such as fructose and glucose with molecular weight of

180 g/mol, diffuse more easily in the fruit tissue than larger molecules such as sucrose (346 g/mol, Panagiotou et al. (1999).

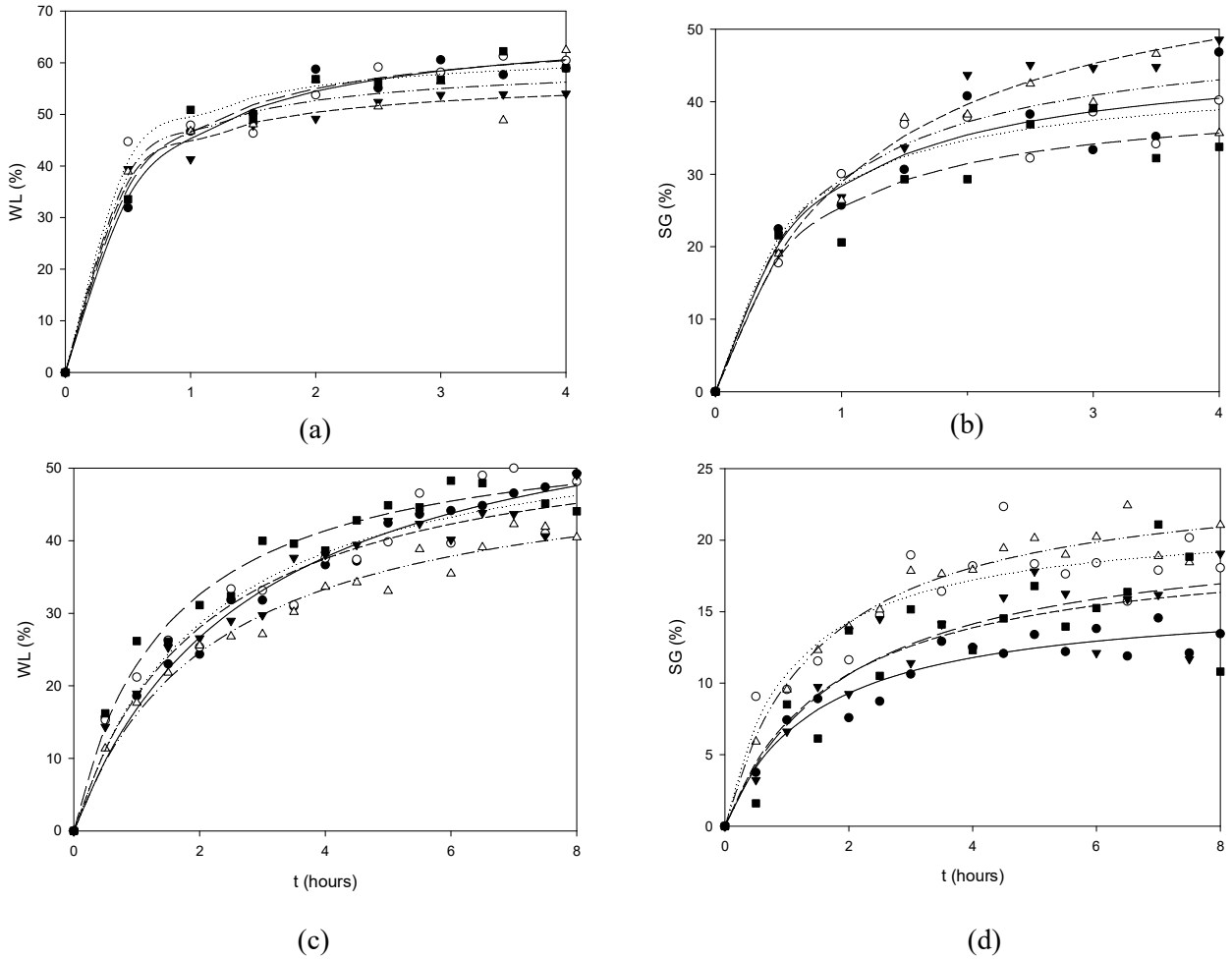


Figure 3.2: Water loss (a, c) and solids gain (b, d) during osmotic dehydration in syrup solutions of mango samples of 0.4 cm thickness during 4 hours (a, b) and 1.5 cm thickness during 8 hours (c, d).

Symbols (experimental data): ● Sucrose, ○ Fructose, ▼ Glucose, △ Agave syrup (AS), ■ AS + 5% Inulin. **Lines** (regression model predictions): ——— Sucrose, ····· Fructose, - - - - - - Glucose, – · – · – Agave syrup (AS), – – – – – – AS + 5% Inulin

Figure 3.3 presents the experimental data of water loss and solids gain during dehydration of mango samples in 60° Brix osmotic solutions made from agave syrup (with or without added xanthan gum) and corn syrup solids. Corn syrup solids solutions provided the lowest solids

gain (less than 5% at the end of the process) for 1.5 cm samples, while agave syrup, the highest one ($p< 0.05$). Osmotic solutions containing xanthan gum show a drastic decrease in sugar gain, by a half and a third respectively, when 0.1% or 0.3% xanthan gum was added to agave syrup (Figures 3.3 b, d).

Regarding water loss, for samples having 0.4 cm thickness, only corn syrup solids solution had a slight impact on decreasing water loss (48% compared to an average of 55% for the other three solutions), while for those having 1.5 cm, both agave syrup with added 0.3% XG and corn syrup solids solutions provided the lowest water loss (Figure 3.3). This behavior can be explained by the high viscosity of the corn syrup solids and agave syrup with added XG solutions (Figure 3.1).

Previous described results showed that sugar gain reduction depends largely on the viscosity of the solution rather than on the solute molecular weight. Both xanthan gum and inulin are large polysaccharides, however xanthan gum largely affects the solution viscosity compared to inulin and thus, helps to decrease markedly the solids gain as well.

As found previously for sugar osmotic solutions (Figure 3.2), increasing sample thickness also significantly lowered the solids gain ($p<0.05$) and less markedly, the water loss during osmotic dehydration in agave syrup solutions (Figure 3.3).

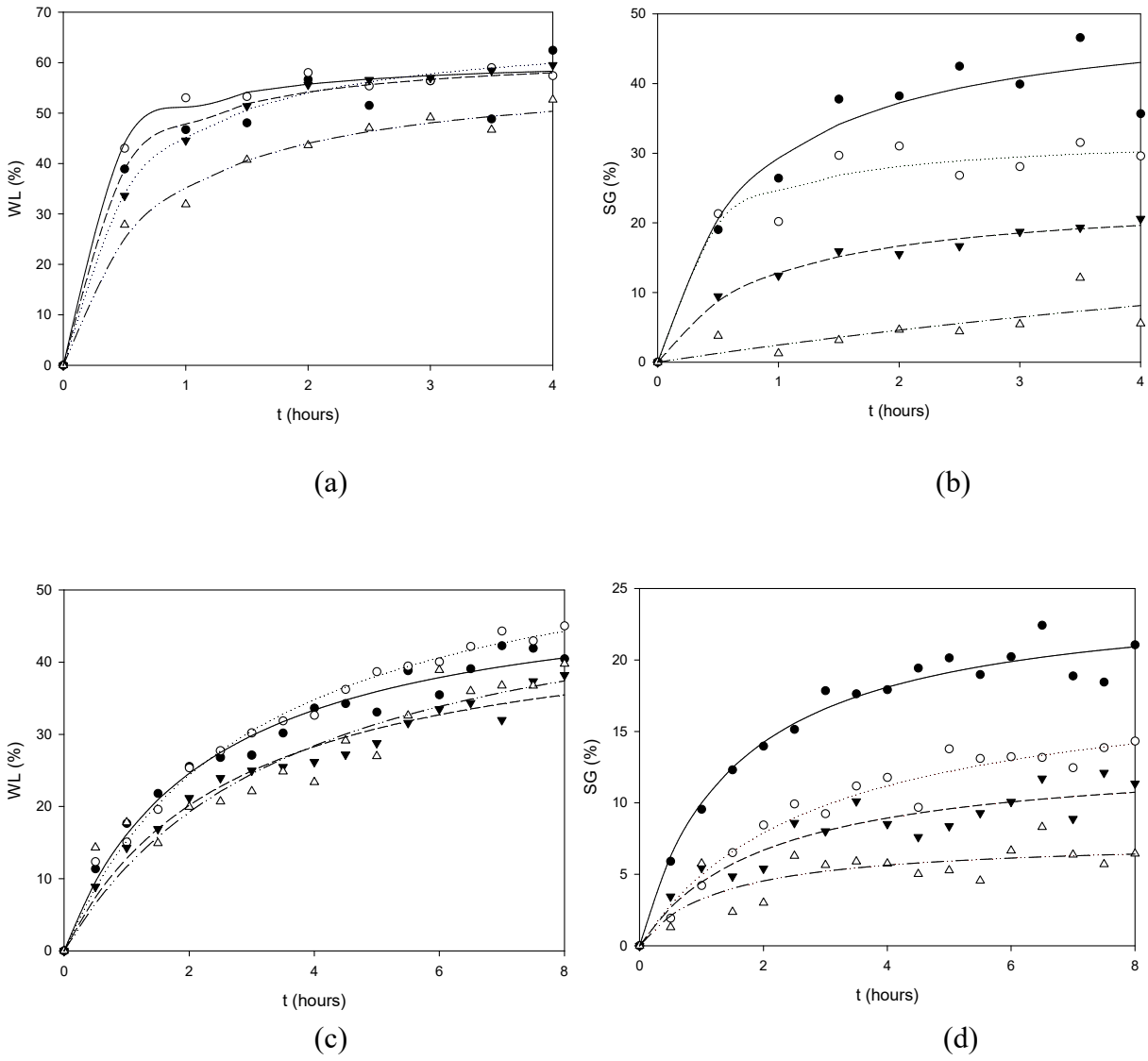


Figure 3.3: Water loss (a, c) and solids gain (b, d) during osmotic dehydration in syrup solutions of mango samples of 0.4 cm thickness during 4 hours (a, b) and 1.5 cm thickness during 8 hours (c, d).

Symbols (experimental data):●Agave syrup, ○AS+0.1%XG, ▼AS+0.3%XG, △Corn syrup solids.

Lines (regression model predictions): —— Agave syrup, ... AS+0.1%XG, - - - - - - - AS+0.3%XG, -·-·-·- Corn syrup solids

Tables 3.2 and 3.3 show the fitting parameters of Equations (3.5) and (3.6), WL_{eq}, SG_{eq}, b_1, b_2, for water loss and solids gain during osmotic dehydration of mango slices of 0.4 cm and 1.5 cm thickness, respectively, as well as the standard error for the fitting which was found to be less than 5% in all the correlations. In Figures 3.2 and 3.3, predictions of water loss and solids gain made from Equations (3.5) and (3.6) using parameters from Tables 3.2 and 3.3 are shown, which are in good agreement with experimental data.

As found already experimentally, the impact of different solutions and thickness was more pronounced for SG_{eq} than for WL_{eq} (Tables 3.2 and 3.3). For a thickness of 0.4 cm (Table 3.2), the maximal sugar gain was higher than 45% for all sugar osmotic solutions with similar viscosities (Table 3.1). However, adding xanthan gum to agave syrup, or working with corn syrup solids solutions, makes SG_{eq} to decrease to values lower than 35% probably due to the important impact of solution viscosity on solids uptake during osmotic dehydration. SG_{eq} dropped with increasing thickness as shown in Table 3.3, where corn syrup (7.4%) and agave syrup + 0.3% XG (13.5%) gave the lowest maximum sugar gain values. Osmotic solutions with similar molecular weight could not give significant differences for samples having 0.4 cm thickness, but it showed a difference for 1.5 cm samples in sucrose solutions, as found experimentally.

Osmotic dehydration efficiency (*ODE*) was calculated from Equation (3.7) (ratio of maximum water loss to maximum sugar gain) and presented in Tables 3.2 and 3.3 for 0.4 cm and 1.5 cm, respectively. Typically for osmotic dehydration, high efficiency ratio indicates high water loss values, which are most wanted. Higher values of osmotic dehydration efficiency were obtained with osmotic solutions containing polysaccharides for both sample thickness (except for 1.5 cm samples in sucrose solutions). For 0.4 cm samples, solutions containing XG showed the highest efficiency values (1.88 and 2.81 for 0.1% XG and 0.3% XG, respectively), followed by CSS solutions and AS+5% Inulin (1.68 and 1.63, respectively). For 1.5 cm samples, CSS solutions presented the highest osmotic dehydration efficiency value (7.39), followed by sucrose solutions (4.00), and then, XG solutions (3.17 and 3.52 for 0.3% and 0.1% XG solutions, respectively). Thus, in general, adding polysaccharides to simple osmotic solutions was found to be beneficial not only for reducing solids gain but increasing dehydration efficiency as well.

Table 3.2: Fitting parameters (Equations (3.5) and (3.6) for 0.4 cm mango samples (*Standard error*$_1$ is for water loss modelling, and *Standard error*$_2$, for solids gain modelling), and estimated dehydration efficiency *ODE* (Equation (3.7))

Solution/Solute type	WL_{eq}	b_1	*Standard error*$_1$	SG_{eq}	b_2	*Standard error*$_2$	*ODE*
Sucrose	68.3	0.51	2.36	47.2	0.66	4.37	1.45
Fructose	63.0	0.27	3.48	44.1	0.54	3.37	1.43
Glucose	57.5	0.28	1.85	63.1	1.18	2.22	0.91
Agave Syrup (AS)	60.3	0.29	4.08	51.0	0.74	3.97	1.18
AS + 5% Inulin	67.1	0.44	2.57	41.2	0.62	3.57	1.63
AS + 0.1% XG	61.2	0.19	1.47	32.6	0.32	2.63	1.88
AS + 0.3% XG	67.2	0.49	0.82	23.9	0.86	0.82	2.81
Corn syrup solids	58.9	0.67	2.15	35.1	13.27	2.38	1.68

Table 3.3: Fitting parameters (Equations (3.5) and (3.6)) for 1.5 cm mango samples (*Standard error*$_1$ is for water loss modelling, and *Standard error*$_2$, for solids gain modelling), and estimated dehydration efficiency *ODE* (Equation (3.7))

Solution/Solute type	WL_{eq}	b_1	*Standard error*$_1$	SG_{eq}	b_2	*Standard error*$_2$	*ODE*
Sucrose	64.4	2.83	2.37	16.1	1.46	1.04	4.00
Fructose	58.2	2.07	3.48	21.7	1.03	1.98	2.68
Glucose	56.7	2.04	2.34	19.9	1.74	2.12	2.85
Agave Syrup (AS)	51.8	2.21	1.78	24.8	1.48	1.06	2.09
AS + 5% Inulin	56.6	1.47	2.52	21.2	1.99	2.68	2.67
AS + 0.1% XG	60.9	2.98	1.29	19.2	2.87	0.90	3.17
AS + 0.3% XG	47.5	2.71	1.69	13.5	2.02	1.15	3.52
Corn syrup solids	54.7	3.69	3.56	7.4	1.28	1.22	7.39

The previous results indicate that solids gain and to less extent water loss, is affected by sample thickness but mainly by the viscosity of the osmotic solution. Thus, maximum solids gain $(SG)_{eq}$ (Tables 3.2 and 3.3) was represented in Figure 3.4 as a function of consistence index K at 40ºC (Table 3.1), and results were then correlated through a power function.

The results of the power fitting for 0.4 cm thickness (Equation 3.7) and 1.5 cm thickness (Equation 3.8) were found to be:

$$\left(SG_{eq}\right)_{0.4cm} = 24.565\ K_{40C}{}^{-0.168} \tag{3.7}$$

$$\left(SG_{eq}\right)_{1.5cm} = 11.633\ K_{40}{}^{-0.126} \tag{3.8}$$

The standard error of estimation was 0.74% for the 0.4 cm thickness correlation (Equation 3.7), and 5.52% for 1.5cm thickness (Equation 3.8). As can be seen from Figure 3.4, maximum solids gain is dependent on viscosity (through consistency parameter K), well represented by a power decay function (Equations 3.7 and 3.8). Thus, for osmotic solution concentrations above those providing a positive driving force for an intake of solids gain and when comparing different osmotic solutions having same concentration but different viscosities, the viscosity was shown to negatively affect the sugar gain, probably by increasing the mass transfer resistance for the external diffusion as pointed out in the literature (Contreras & Smyrl (1981); Hawkes & Flink (1978); Khoyi & Hesari (2007). Higher viscosity increases the difficulty for the solutes to move through the solution towards the mango surface, creating an external resistance which can modify the control for mass transfer (from internal to mixed one), and decreasing the interphase sugar concentration. This in turn will decrease the driving force for internal diffusion. Therefore, high viscosity solutions like corn syrup solids and those containing xanthan gum would lower the solids gain more than simple sugar solutions. It was reported in previous studies that high molecular solutes like corn syrup mixed with sucrose lowered the sugar gain because of the presence of higher molecules with low dextrose equivalent in corn syrup (Allali, 2008b); Bolin et al.

(1983). As well, in an article by Giraldo et al. (2003), it was pointed out that internal 'liquid' diffusion coefficients in mango samples during osmotic dehydration decrease by the increase in external osmotic solution viscosity.

In addition, a high thickness (1.5cm) led to low solids gain for all the osmotic solutes used. From Figure 3.4, it can be observed that the impact of solution rheology is also more evident for low thickness, and also the power decay function for 0.4 cm thickness presents a higher scaling factor (24.565) and exponent (-0.168) than for 1.5 cm thickness samples (11.633 and -0.126, respectively), as shown in Equations (3.7) and (3.8). This different behaviour for different thicknesses could be probably explained by the change in mass transfer control from mixed (internal and external) to internal control as thickness increases, and thus pointing out the importance that the Biot number for mass transfer could have in selective osmotic dehydration.

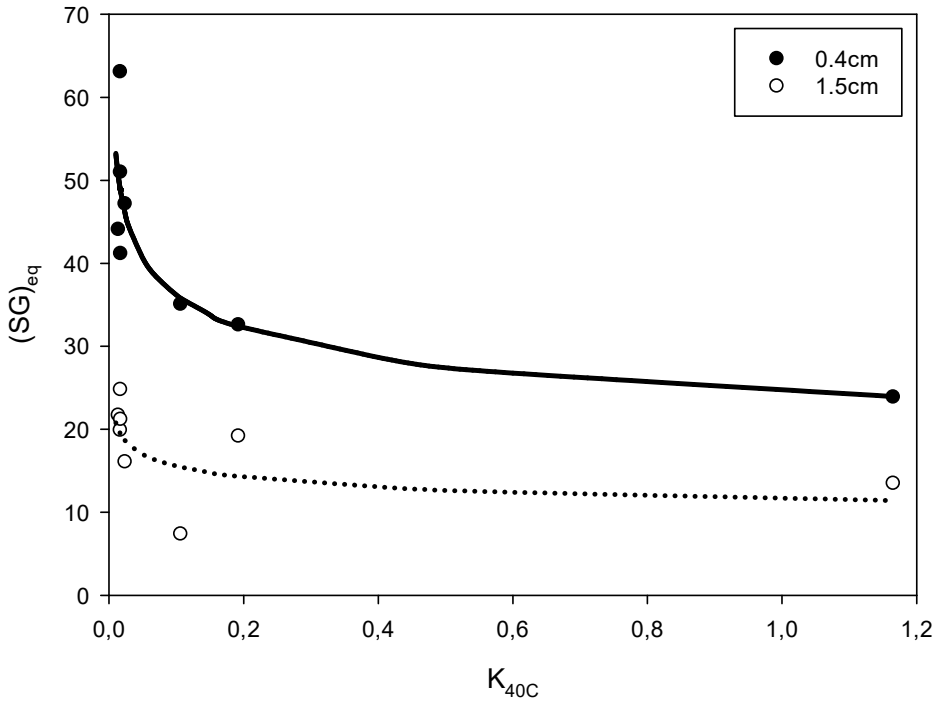


Figure 3.4: Effect of solutions apparent viscosity on sugar gain according to the sample thickness

3.6 Conclusions

This study focused on the optimization of solution viscosity and sample thickness to lower solids uptake during osmotic dehydration of mango. Agave syrup with added inulin shows similar solids uptake as agave syrup, fructose or glucose solutions. However, solutions with high viscosity, such as corn syrup solids and xanthan-gum added agave syrup solutions presented the lowest solids uptake for both 1.5 cm and 0.4 cm thick samples, indicating that the choice of the polysaccharide should be determined by the effect on the increase of solution viscosity if the aim is to lower the solids gain. In addition, high thickness (1.5 cm) led to low solids gain for all the osmotic solutes assessed. The impact of increasing apparent viscosity on solids gain was found to be more pronounced for low thickness, showing the importance of choosing a high solution viscosity combined with thicker mango samples to succeed in lowering the solids uptake. To conclude, healthier mango snacks could be produced through osmotic dehydration in agave syrup solutions with added xanthan gum.

3.7 Bibliography

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Transition vers l'article 2

Dans le chapitre précédent, une caractérisation de huits solutions osmotiques de compositions différentes a été réalisée en analysant la rhéologie et la viscosité. Ensuite l'effet des solutions osmotiques et de l'épaisseur des mangues congelées sur l'évolution de la perte en eau et du gain en sucres (**totaux**) au cours de la déshydratation osmotique a été suivi. Les résultats ont montré que l'augmentation de la viscosité de la solution osmotique et de l'épaisseur de la mangue permettent de réduire le gain en sucres tout en maintenant une quantité suffisante de perte en eau. Le chapitre suivant (4) est une étude de la modulation du profil en sucres **individuels** de la mangue après la déshydratation osmotique dans les solutions osmotiques avec mono et multi-solutés utilisées dans le chapitre antérieur, ainsi que de la détermination de la présence d'inuline dans la mangue quand la déshydratation osmotique a été faite avec des solutions enrichies avec cet ingrédient. Une évaluation de la formulation du produit final a complété ce chapitre.

Ce chapitre a été soumis pour publication au *Journal of Food Science* (en attente d'acceptation final après corrections) :

Zongo, A. P., Khalloufi, S., & Ratti, C. (2022). Sugar profiles modulation of mango during osmotic dehydration in agave syrup solutions. *Journal of Food Science* (corrections envoyées).

Les résultats obtenus ont fait l'objet d'une communication par poster au Green Food Technology (GFT) le 28 Avril 2021. Titre: *HPLC sugar profiles evolution in mango slices during osmotic dehydration in agave syrups.*

Chapter 4: Sugar profiles modulation of mangoes during osmotic dehydration in agave syrup solutions

4.1 Résumé

L'interaction chimique et la compétition de plusieurs composés ont été étudiées sur le gain en solides et l'évolution des profils de sucres lors de la déshydratation osmotique des mangues. Des tranches de mangue Tommy Atkins (0.4 cm et 1.5 cm d'épaisseur) ont été traitées par osmose à 40°C pendant 4 heures et 8 heures, respectivement. Deux catégories de solutions osmotiques à 60 °Brix ont été utilisées: les solutions contenant un seul soluté (sucrose, glucose, fructose) et plusieurs solutés (sirop d'agave, seul ou additionné de 5 % d'inuline ou de 0,1 % à 0,3 % de gomme xanthane). Une analyse par chromatographie liquide à haute performance (HPLC) a été réalisée sur la mangue traitée pour déterminer l'évolution des profils de sucres au cours de la déshydratation osmotique et les concentrations du produit final. Les résultats ont montré que la composition de la solution osmotique peut moduler les profils de sucres de la mangue en favorisant l'absorption ou la perte de sucres selon différents phénomènes : le gradient de potentiel chimique, la lixiviation, le control prédominant du transfert de matières, la formation d'une barrière de sucres et l'augmentation de la viscosité de la solution. Dans la solution à un soluté, la mangue s'est enrichie du soluté présent, tout en perdant ses propres sucres natifs qui étaient absents de la solution osmotique. L'augmentation de l'épaisseur de l'échantillon a réduit l'absorption ou la perte de sucres individuel dans la mangue traitée avec des solutions à un ou plusieurs solutés. Des différences significatives dans le comportement de la solution à soluté unique ont été rapportées pour le sucrose en raison de sa capacité à former une couche de sucrose à l'extérieur de la surface des échantillons plus épais, ce qui a été montré par des images de microscopie électronique à balayage (SEM), une barrière entravant nettement l'absorption ou la perte de sucrose. L'ajout de polysaccharides (en particulier la gomme xanthane) s'est avéré avoir un impact sur la diminution de l'absorption individuelle de sucre de la mangue (18 à 30%). Ces résultats sont utiles pour comprendre les mécanismes par lesquels le gain de sucres individuels pourrait être réduit et la composition pourrait être modulée lors de la déshydratation osmotique des fruits. Ainsi, les résultats de ce travail pourraient conduire à produire des

collations à base de mangue déshydratées par osmose ayant une faible teneur en sucre ajoutés et enrichies en inuline, améliorant ainsi leur valeur alimentaire et commerciale.

4.2 Abstract

Chemical interaction and multicompound competition were investigated on solids gain and carbohydrate profiles evolution during osmotic dehydration of mangoes. Tommy Atkins mango slices (0.4 cm and 1.5 cm thickness) were osmotically processed at 40 °C for up to 4 hours and 8 hours, respectively. Osmotic solutions (60 °Brix) were separated in two categories: single solute (sucrose, glucose, fructose) and multi-solute (agave syrup, alone or with additions of 5% inulin or 0.1%-0.3% xanthan gum) solutions. High Performance Liquid Chromatography (HPLC) analysis was carried out on treated mango to determine sugar profiles evolution during osmotic dehydration and final product concentrations. Findings pointed out that composition of osmotic solution may modulate mango sugar profiles by triggering uptake or loss of sugar according to different phenomena: chemical potential gradient, lixiviation, predominant control for mass transfer, formation of carbohydrate barrier, and the increment of solution viscosity. Mango was enriched with the solute present in single solute osmotic solution, while lost its own native sugars which were absent in the osmotic solution. Increasing sample thickness reduces individual sugar uptake or loss in mango treated with both single and multi-solute solutions. Significant differences in mono solute solution behavior were found for sucrose due to its capability to form a sugar layer outside the surface of thicker samples, which was shown by scanning electron microscopy (SEM) images, a barrier hindering markedly sucrose uptake or loss. Addition of polysaccharides (particularly xanthan gum) was found to have an impact of lowering mango individual sugar uptake (18 to 30%). These results will help to understand the mechanisms by which gain of individual sugars could be reduced and composition could be modulated during osmotic dehydration of fruits. Thus, the findings in this work could lead to the production of low sugar content osmotically processed mango snacks, enriched with inulin, enhancing their dietary and marketable value.

4.3 Introduction

Mango is one of the most important tropical fruit crops in the world (Yadav & Singh, 2014). In addition to pleasant flavor and sweet taste, mangoes contain vitamins E and C, beta-carotene and phytochemicals (Ntsoane et al., 2019). However, water content (≥80%) and nutrients availability facilitates microorganisms' growth leading to mangoes deterioration. Hence the necessity to develop appropriate preservation methods for mangoes to retain its nutritive and organoleptic qualities. Hot air-drying, for example, is an easy and efficient method to preserve fruits and vegetables (Izmir & Arif, 2020). However, it requires operation at high temperatures that usually leads to nutritional and sensorial damages (Lenart, 1996; Lin et al., 1998; Torreggiani, 1993). Osmotic dehydration is a mild-temperature method consisting of immerging cellular foods in a hypertonic solution (usual solutes are sugars or salt) (Shi, 2009). The chemical potential gradient between the tissue cells and the solution generated by an osmotic pressure difference, favors on one hand the transfer of moisture towards the solution and as well, the diffusion of solute into the plant tissue together with the leakage of soluble compounds from the tissue (Akharume et al., 2019). Osmotic dehydration is a multicompound mass transfer phenomenon, where water loss should be maximized but solids intake and biocompounds leakage, minimized. Osmotic dehydration requires simple equipment and preserves shelf life, favors organoleptic and nutrients retention due to use of mild temperatures and an osmotic solution free of oxygen which prevent oxidation and enzymatic browning (Chavan & Amarowicz, 2012; Yadav & Singh, 2014).

Mass transfer mechanisms in osmotic dehydration are mainly dictated by effective diffusion in the product and convection in the solution. While diffusion coefficients are an intrinsic function of the microstructure of the product (porosity, tortuosity, etc.) and temperature, external mass transfer coefficients are especially dependent on solution characteristics such as movement and viscosity. The Biot number for mass transfer (Ratti, 1994) helps to establish which mechanism could be controlling the mass transfer, and thus which parameters are more important to modulate water loss or solids gain. In this sense, thicker samples are prone to be controlled by diffusion (internal mass transfer) while thinner, by external convection.

Nowadays, reducing total solids gain and manipulating the carbohydrate composition in osmotically dehydrated fruits is a growing research interest (Jiménez-Hernández et al., 2017;

Turkiewicz et al., 2020) following the consumers' quest for healthier snacks and natural products. Edible coatings have been explored to reduce solute gain through creating a physical barrier for mass transfer (Azam et al., 2013; Jalaee et al., 2011). As well, increasing the viscosity of the solution was investigated for several fruits and vegetables including mango (Zongo et al., 2021; Assis et al., 2017; Manivannan & Rajasimman, 2011) with ambiguous results on solids gain reduction depending on the product and particularly, the temperature. Other scientific reports on the same subject found out that increasing the thickness of the product may also reduce the sugar uptake (Zongo et al., 2021; Cao et al., 2006). However, these previous studies focused on total solids gain and water loss but did not investigate the impact of the osmotic dehydration conditions (viscosity, sample thickness, solution composition) on the modulation of sugar profiles of the final product.

In addition, agave syrup is scarcely reported in the literature in relation to osmotic dehydration, despite its rich nutritive composition (vitamins, antioxidant, antibacterial, inulin) (Martinez-Gutierrez et al., 2018; Mellado-Mojica & López, 2015). It is a natural sweetener that is already used for culinary and medical purposes (Corrales Escobosa et al., 2014) and it could be interesting to investigate its usage as an alternative osmotic solution. Agave syrup exhibits low glycemic index due to its high content in fructose (>70%) (Willems & Low, 2012). The other sugars present in agave syrup are glucose and sucrose although in lower percentages (Mellado-Mojica & López, 2015). The presence of inulin in agave syrup may lead to production of functional products because of its prebiotic effect contributing to a healthy gut microbiota. Therefore, the use of agave syrup osmotic solution can help to create attractive products with added value for consumers. Regarding agave syrup's composition, it is by itself a multicomponent solution with thermodynamic characteristics driven by the interaction of its compounds in solution. These inter-solute interactions could be weak or strong showing diverse deviations to the Roos model for multicompound water activity (Sone et al., 2015) and thus, they might impact at various levels the speed of relative movement of individual solutes towards the solid.

The objective of the present work aims to investigate how single solute osmotic solutions (sucrose, glucose, fructose) and multi-solute solutions (agave syrup with or without addition of polysaccharides) may modulate sugar profiles of osmotically dehydrated mangoes

depending on the control for mass transfer for the process, and as well minimize individual sugars uptake.

4.4 Materials and Methods

4.4.1 Materials

Fresh Tommy Atkins mangoes were purchased in a local supermarket and stored at ambient temperature until mango reached a soluble sugar content of 12 ± 2 °Brix. Fructose, sucrose and glucose were purchased from Farinex (Quebec, Canada), while xanthan gum and agave inulin were bought at Biovea (USA). Agave syrup (Jarabe de ágave orgánico) was purchased at the market in Guadalajara (Mexico) and its sugar composition was analyzed through HPLC with Refractive Index detector to later reproduce a 'lab-made' syrup that is close in composition to the real agave syrup.

Standards for HPLC determinations were analytical grade (99% purity). Fructose, glucose and sucrose standards were purchased at Fisher Scientific (Ottawa, Canada) and for chicory inulin, at Sigma Aldrich (Steinheim, Germany). MilliQ water (Elga Purelab Ultra, High Wycombe, UK) was used as solvent in HPLC determinations.

4.4.2 Osmotic Solutions

A total of 7 osmotic solutions at 60 °Brix were prepared for the osmotic treatments. Distilled water at a temperature of 80°C was used to facilitate dissolution of carbohydrates. A Fisher Thermix stirring hot plate (Model 210T, Ottawa, Canada) was used for stirring and heating the solutions. Solutions were made by mixing the corresponding amounts of carbohydrates with distilled water, followed by heating up the mixture (100 °C) for 30 min until a syrup is obtained. Three (3) solutions (later called single-solute solutions) were composed by just one monosaccharide, i.e., sucrose (S), glucose (G), or fructose (F), and the other four (4) solutions (multi-solute solutions), by a mixture of carbohydrates as follows. The 'lab-made' simulated agave syrup (AS) was prepared with 79% fructose, 20% glucose, and 1% sucrose in distilled

water at 60 °Brix, to which 5 % inulin (I) or 0.1% or 0.3% xanthan gum (XG) were added. To easily dissolve inulin in the agave syrup, it was first separately mixed with hot water (80°C) and strongly stirred with the Fisher Thermix stirring hot plate, before integrating it to the syrup. For xanthan gum, it was mixed well with the other sugars in powder form, then water (ambient temperature) was added before heating it up and stirring to obtain a homogenous solution. A pocket refractometer PAL-2 (Atago, Tokyo, Japan) with a range of 45-93% was used to verify the solutions final total soluble solids content (°Brix). Solutions obtained were stored at 4 °C.

4.4.3 Mango samples for osmotic dehydration

Mangoes were stored at ambient temperature (20°C) for up to 4 days to reach 12-14 °Brix, before further processing. An Atago Pocket refractometer PAL-1 (0-53%) was used to measure the total soluble solids (TSS) of the samples. Then, they were washed, rinsed, peeled, and cut manually with a knife into rectangular slices of 2.5 cm width, 5 cm length. Two different sample thickness were studied, 0.4 cm and 1.5 cm. The samples weighed approximatively 5 g for the 0.4 cm thickness slice and 24 g, for 1.5 cm thickness. Samples were kept in a freezer (Forma Scientific freezer, USA) at -60°C for further use in osmotic dehydration experiments.

4.4.4 Osmotic dehydration

The osmotic solution was heated up to 40 °C in a model Isotemp 1016 S water bath (Fischer scientific, Pittsburgh, USA), before immersing mango samples placed in individual cages. Temperature was automatically maintained in the bath at 40 °C with a precision of ±0.2°C. The solution to sample ratio was 1:100 (w:w). Osmotic dehydration was carried out separately for both sample thicknesses in a model Isotemp 1016 S water bath at 40°C for up to 4 hours (0.4 cm thick, 5 g per sample, total of 8 samples in the bath) and 8 hours (1.5 cm thick, 24 g per sample, total of 16 samples in the bath). One piece of sample was taken out of the solution every 30 min, rinsed quickly, and blotted with paper before weighing and kept

at -60 °C (Forma Scientific freezer, USA) in aluminium dishes wrapped in Ziploc bag during at least 24 hours prior to further processing.

Osmosed mangoes were freeze-dried in a Freeze Mobile 25L EL (SP Virtis, Pennsylvania, USA) at 30°C shelf temperature and 30 millitorr vacuum for 72 hours. The lyophilized samples were weighed to obtain the dry mass (m_s), packaged in Ziploc bags, and stored at -30 °C for further HPLC analysis.

Solid's gain (G) or loss (L) in (g carbohydrate i/100 g fresh mango) for individual carbohydrates (i) was calculated from HPLC concentration results (methodology will be described later), with the following equation:

$$G_i \text{ or } L_i = \frac{(c_{it}\, m_t - c_{io}\, m_o)}{m_o}\, 100 \qquad (4.1)$$

where c_{it} and c_{io} are the concentrations of carbohydrate i at times t and initial (g i/g product), respectively (measured from HPLC determinations), m_t and m_o, are the mass of product (g) at time t and initial, respectively. The individual carbohydrate (sucrose, fructose, glucose, inulin) gain or loss curves were represented as a function of osmotic dehydration time.

A two parameter hyperbola lines were fitted using SigmaPlot version 14.0 (Systat Software, Inc., San Jose California USA) and added to the graphical representations for visual clarity purposes. Equilibrium sugar gain/loss values for individual carbohydrates were estimated from these curves as the corresponding ordinate value when the curves become almost parallel to the abscissa.

Final water and total solids contents after 4 and 8 hours osmotic dehydration (for 0.4 and 1.5 cm thickness samples, respectively) were reported in g /100 g final product (%). Values of total solids content were estimated at a fixed water content of 17.3% (recommended by USDA, 2021, for mango snacks) for comparison purposes.

4.4.5 HPLC analysis for sugar profiles

Lyophilized mango samples were individually crushed in a mortar and particle obtained was used to extract soluble sugars and carbohydrates according to Petkova (2014) with slight modifications. Fresh mango samples were also lyophilized as described previously and used as control to determine the initial sugar composition of mangoes. Half gram (0.5 g) of mango particle was weighed in a 15 mL falcon tube, to which 12 mL of Millipore milliQ water was added. The tubes were shaken with a vortex for better dispersion in the liquid, and heated up to 80 °C in a water bath (Grant JB Nova, Cambridge, UK) for 20 min. The solution was then vortexed and kept at 4 °C for 24 hours so as to obtain a better extraction of the sugars and carbohydrates. After extraction, samples were homogenized in a shaker, followed by centrifugation at 4696 g at 22 °C for 10 min (Thermo Scientific, Sorvall Legend X1R centrifuge, Germany, Am Kalkberg). Then, 1 mL of the supernatant was transferred into 1.5 mL microtube. Dilution was made to 1/100 in a 1 mL HPLC vial for injection.

Fifty (50) uL volume samples were injected on an Agilent 1100 series (Hewlett Packard, Waldbrown, Germany) equipped with a refractive index detector (Agilent 1260 Infinity II). Chromatographic separation was performed on a (Shodex, Tokyo, Japan) sugar SP0810 column (300 mm×80 mm i.d) with Pb^{2+} equipped with a column guard (50×9.2 mm i.d) according to the method developed by Petkova (2014) with slight modifications. The chromatographic conditions of the HPLC analysis were 85 °C column temperature, 0.6 mL/min flow, 35 min analysis time, 18-23 bar pressure. The solvent used was HPLC grade water (MilliQ water). Before injection of samples, column was rinsed with methanol 10% during 30 min to avoid contamination, then rinse with HPLC grade water to remove methanol residue.

Seven-point calibration curves were made by mixing sucrose, fructose, glucose standards in distilled water and a six-point calibration curve, for inulin. A correlation of 0.99 was accepted. Calibration curves were obtained by injecting different concentrations of standard sugar solutions (50, 100, 200, 500, 700, 1000, 1500 ug/mL) and standard inulin solutions (10, 20, 50, 100, 250, 500 ug/mL).

Final carbohydrate concentrations of osmotically dehydrated mango, c_{ti}, were estimated from HPLC determinations as a function of time in (g of carbohydrate *i*/g product).

4.4.6 Scanning electron microscopy

Fresh and osmotic dehydrated mango surfaces were analyzed by scanning electron microscopy (SEM). With the supposition that solutes deposit on the surface during osmotic dehydration would be independent of sample thickness (and to solve technical difficulties), SEM analysis was performed on mango cubes (1 cm thickness) dehydrated in different osmotic solutions for 4 hours. The osmotic solutions used in this part of the work were the same as described earlier, but also a 60% pure inulin osmotic solution was used as osmotic agent for comparison and discussion.

To prepare samples for SEM, both fresh and osmotically dehydrated samples were freeze-dried in a Freeze Mobile 25L EL at 30°C shelf temperature and 30 millitorr vacuum for 72 hours. Freeze-dried samples were mounted on a metal disc using carbon double-sided conductive tape. Then, samples were gilded using an EMS950x vacuum evaporator (Electron Microscopy Sciences, PA, USA). Silver paint was used to ensure permanent bonding. Then, samples were scanned in a JSM-6363LV (JEOL, Tokyo, Japan) SEM operated at 3 and 15 KV with magnifications of 50, 200 and 800 times.

4.4.7 Statistics

HPLC analysis were made on two different osmotically dehydrated samples coming from two randomly chosen repetitions, so as to obtain duplicates for statistical purposes. The statistical analysis was made with Rstudio software (RStudio-1.2.5033). A *p*-value adjustment was made with Tukey test for comparing family of estimates. The confidence level used was 95%.

4.5 Results and Discussion

4.5.1 HPLC profiles determination

Figures 4.1(a, b) shows HPLC chromatograms for inulin (I), and a mixture of sucrose (S), glucose (G) and fructose (F) from which retention times were determined as 8.57 min, 12.48 min, 14.46 min and 21.09 min, respectively. Figure 4.1c presents an example of the chromatogram for sugar profiles of fresh mango, where sucrose, glucose and fructose eluted at times according to those previously found for the standards (Figure 4.1b). Figure 4.1d shows the HPLC carbohydrate profile of osmotic dehydrated mango in agave syrup, where sucrose, fructose and glucose peaks were detected. Peak's heights of fructose and glucose in Figure 4.1d showed an increase (and sucrose a decrease) compared to those of Figure 4.1c due to osmotic dehydration. Finally, the HPLC profile of osmosed mango in a solution of simulated agave syrup with 5% added inulin (Figure 4.1e), showed the presence of the three sugars and a significant peak of inulin at 8.57 min, clearly indicating that is possible to impregnate samples with inulin through osmotic dehydration. (Jiménez-Hernández et al., 2017) also revealed inulin impregnation in mango samples with inulin-oleoresin microcapsules, which was shown by SEM.

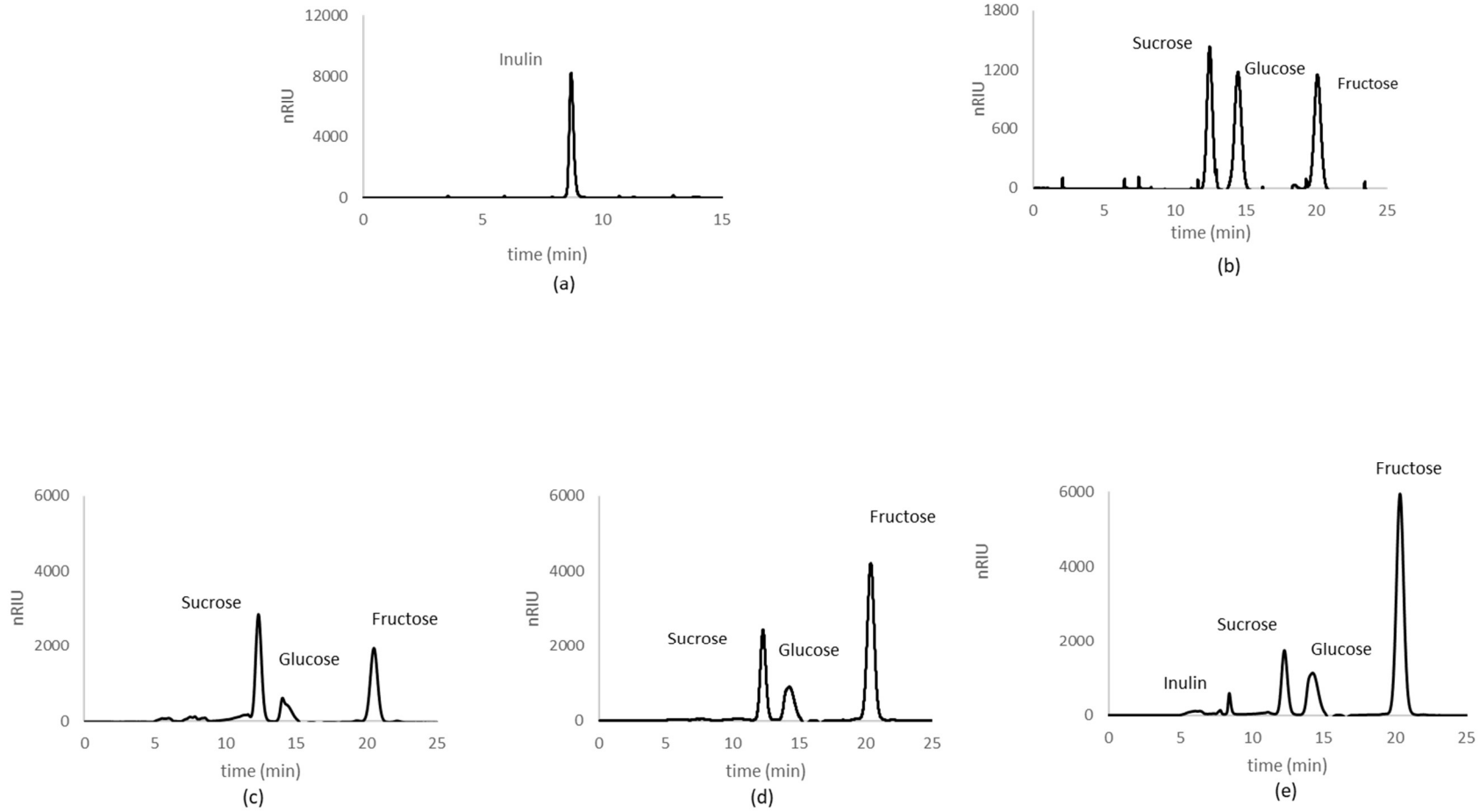

Figure 4.1: HPLC profiles of inulin standard (a), mixture of glucose, fructose and sucrose standards (b), fresh mango (c), osmotic dehydrated mango in agave syrup (d), and osmotic dehydrated mango in agave syrup + 5% inulin (e)

4.5.2 Carbohydrate content in fresh mango and agave syrup

Carbohydrate composition of fresh mango and agave syrup is shown in Table 4.1. As shown in this table, fresh mango contains sucrose, fructose and glucose in decreasing levels. Although somehow lower, these results follow the same tendency as the ones reported by United States Department of Agriculture (USDA, 2018) for ripe Tommy Atkins mangoes, where sucrose is pointed out as the main carbohydrate, followed by fructose and finally glucose (6.97, 4.68 and 2.01 g/100 g fresh product, respectively). Lower carbohydrate amounts found in the present study could be due mainly to different maturity states between mangoes used for the research, and the fact that mangoes in this study were imported for which conditions of handling and storage were unknown. Bello-Pérez et al. (2007) analyzed sugars profiles in mangoes of different varieties during ripening and stated that sucrose and fructose increase during ripening, while glucose decreases, which was attributed to enzymatic reactions. Similar remarks were found in (Maldonado-Celis et al., 2019b). To end, the proportion of the different sugars in the dry solids of fresh mango (calculated from Table 4.1) were found to be 46.59%, 35.93% and 17.49% for sucrose, fructose and glucose, in accordance to the literature (USDA, 2018).

Table 4.1: Carbohydrate composition in mango and natural agave syrup (g/100 g product)

Carbohydrate → **Product ↓**	**Sucrose**	**Fructose**	**Glucose**	**Inulin**
Fresh mango	3.89 ± 0.53	3.00 ± 0.20	1.46 ± 0.30	---
Agave syrup	0.55 ± 0.05	62.63 ± 0.09	23.77 ± 0.08	0.34 ± 00

As seen in Table 4.1, the major carbohydrate present in agave syrup was fructose, followed in lesser extent by glucose, and the smaller components, sucrose and inulin. These results correspond to the general knowledge that agave syrups are mainly composed by fructose (more than 60% of the total soluble solids), followed by glucose and with traces of sucrose (Mellado-Mojica & López, 2015). As well, values shown in Table 4.1 correspond to

proportions in dry solids of 1.89% (sucrose), 71.72% (fructose), 25.92% (glucose) and 0.45% (inulin), which are in agreement to those indicated by the Mexican Official Norm for agave syrup (Corrales Escobosa et al., 2014a). However, Willems & Low (2012) reported that the Mexican standard for agave syrup indicates that blue and *salmania* agave syrups should contain in total weight a minimum of 80 and 70% fructose and a maximum of 15 and 25% glucose, respectively. The results shown in Table 4.1 do not follow the Mexican standard for agave syrup in terms of composition by total weight, and thus, the 'lab-made' syrup to be used in the present study was otherwise formulated with 79% fructose, 20% glucose, and 1% sucrose (please refer to Materials and Methods description).

4.5.3 Osmotic dehydration kinetics

Figures 4.2 to 4.4 represent solids gain or loss of individual carbohydrates during osmotic dehydration in single or multi solutes osmotic solutions. The horizontal line added to these figures at zero ordinate value was taken as a reference separating positive and negative values, indicating solids gain or loss, respectively.

4.5.3.1 Single solute solutions

The effect of mono-solute osmotic solutions (sucrose, fructose, glucose) on solids gain/loss kinetics is illustrated in Figure 4.2 (a-f) for both mango sample thicknesses. As expected, analysis of Figure 4.2 indicated that solids gain/loss depend on the type of sugar in the osmotic solution. Mango was significantly enriched with the sugar present in the osmotic solution but lost some of its initial sugars (the ones absent in the osmotic solution). As an example, during osmotic dehydration in sucrose solution, there is a marked sucrose increase in the mango sample, but individual curves of fructose and glucose were below the horizontal line, pointing out that these sugars were lost during osmotic dehydration. This lixiviation phenomenon is caused by a negative chemical potential gradient between the osmotic solution and the mango tissue

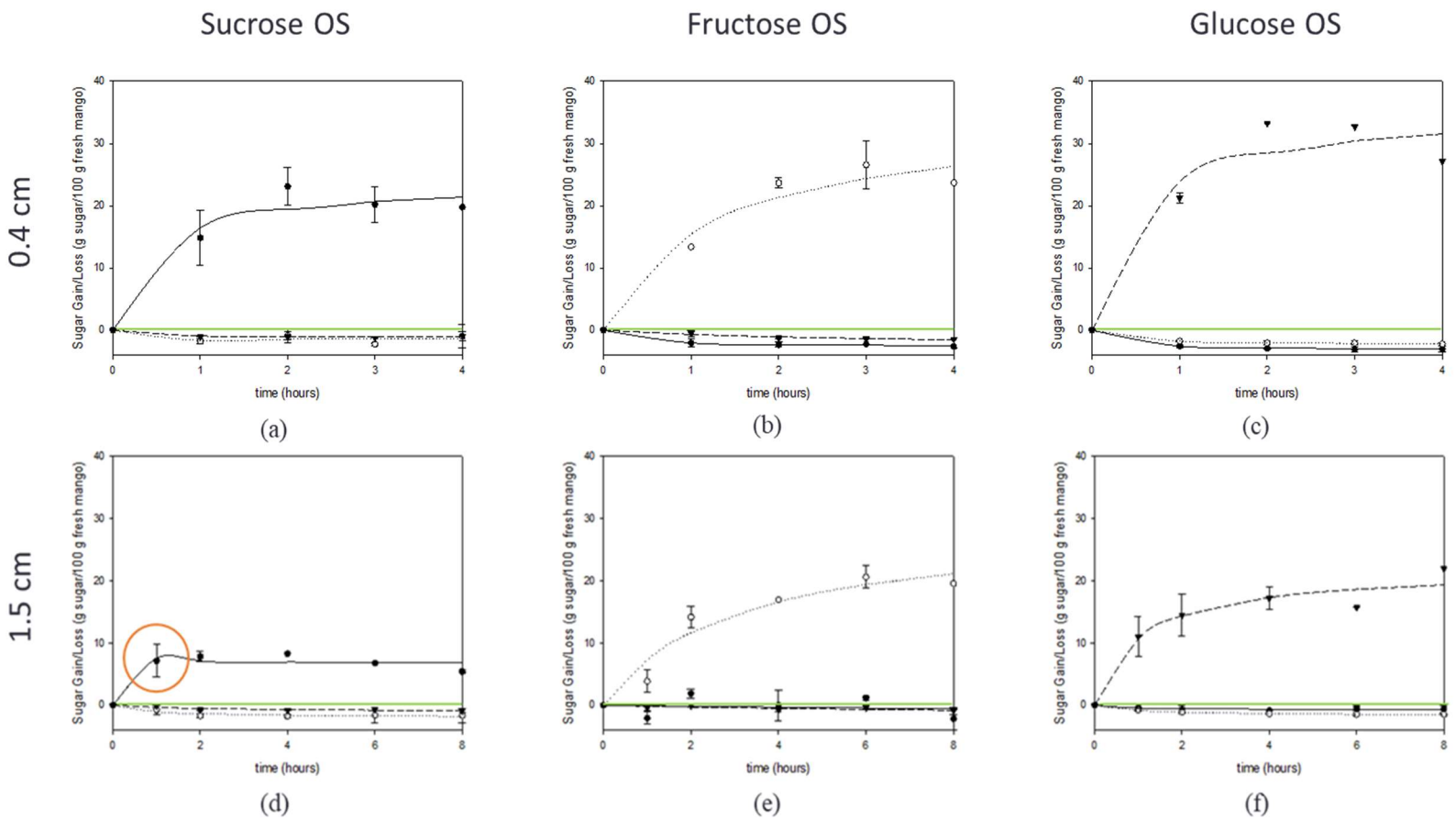

Figure 4.2: Kinetics of individual sugar gain/loss in mango during OD in mono-solute osmotic solutions (OS) of sucrose (a, d), fructose (b, e), glucose (c, f) for samples of 0.4 cm (a, b, c) or 1.5 cm (d, e, f) thickness

Symbols (experimental data): individual sugars are sucrose (●), fructose (○) and glucose (▼). **Lines** (predictions): — sucrose, …. fructose, ---- glucose,— reference.

Individual sugar gain increased over time and in general attained an equilibrium after 2 and 4 hours, respectively for 0.4cm and 1.5cm thickness (Figure 4.2 (a-f)). However, during osmotic dehydration in sucrose solution for 1.5cm thickness, mango reached equilibrium earlier than 2 hours. This different behavior of sucrose could be explained by the rapid formation of a sugar layer at the mango surface creating a resistance for sugar uptake (Bui et al., 2009). Sucrose layer formation during osmotic dehydration has been long reported in the literature when working with osmotic solutions at higher sucrose concentration (Hawkes, 1978; Raoult Wack, 1994). Please note a small 'bump' in the sucrose gain curve of 1.5cm thickness mango slice (Figure 4.2d, indicated by a circle) which may indicate the saturation of the mango surface by the creation of a sucrose layer.

As can be seen in Figure 4.2, increasing sample thickness decreased predominantly ($p<0.05$) the sugar gain and slightly the loss values ($p>0.05$). The individual sugar uptake is inversely proportional to the specific surface which is defined as total surface/half thickness ratio (Lazarides et al., 1995), and thus, higher thickness sample would lower the sugar gain. Zongo et al. (2021) showed similar tendency between maximum total solids gain and thickness during osmotic dehydration of mango slices.

As shown in Figure 4.2 (a, b, c), at lower sample thickness when external mass transfer by convection is predominant, maximum gain values for predominant sugars are in the range from 20 to 30 g/ 100 g fresh mango, although it is the highest ($p<0.05$) for glucose which is the least soluble sugar in aqueous solution. Glucose solubility is much lower than sucrose and fructose, with values of 1.04, 2.07 and 4 g/g water, respectively at 25°C (Hanover & White, 1993). However, at higher thickness (1.5 cm) when internal diffusion prevails, fructose and glucose showed similar maximum values at equilibrium ($p>0.05$) (Figure 4.2 (e, f)), while sucrose presented a lower value for maximum sugar gain ($p<0.05$) (Figure 4.2d). This can be explained by several factors, to start fructose and glucose are monosaccharides having similar molecular weight, and sucrose is a disaccharide having a slower diffusion. Self-diffusion coefficient for sucrose being a heavier molecule, was found to be significantly lower than those for glucose and fructose in aqueous solutions (Aroulmoji et al., 2012). Thus, this difference in diffusion rate is expected to be more pronounced when the matrix is denser than an aqueous solution, such as the mango tissue. As well, as explained earlier, a possible

sugar layer was formed at the surface of thicker mango samples, when sucrose was the predominant sugar in the osmotic solution.

4.5.3.2 Multi-solute solutions

Figures 4.3 and 4.4 present the kinetics of individual sugar evolution in mango slices during osmotic dehydration in multi-solute solutions made from agave syrup with or without added polysaccharides (xanthan gum or inulin).

As explained in Materials and Methods, 'lab-made' agave syrup used in this study is mainly composed of fructose (79%), followed by glucose (20%) and sucrose (1%), and fresh mango is slightly richer in sucrose than fructose (Table 4.1). Thus, predictably, fructose was the most predominant individual sugar uptake in osmosed mango for all four different osmotic solutions (AS, AS+5%I, AS+0.1%XG, AS+0.3%XG) and for both sample thickness (Figure 4.3 and 4.4).

For samples having 0.4 cm thickness (Figure 4.3), glucose and fructose were gained by mango slices as expected due to individual sugar positive driving force for mass transfer. Sucrose, on the other hand, was slightly lost during osmotic dehydration in simple agave syrup or agave syrup with added inulin (Figure 4.3 (a, b)), but slightly gained in osmotic solutions with added xanthan gum (Figure 4.3 (c, d)). Sucrose minor loss for 0.4 cm thickness samples could be explained by the lower sucrose concentration in the osmotic solution, which creates a negative chemical potential taking native sucrose out of mangoes (Figure 4.3 (a, b)). Addition of xanthan gum to agave syrup created a higher external resistance through an increase in viscosity (Zongo et al., 2021), hindering the loss of sucrose towards the osmotic solution, especially for small thickness conditions favorizing external control for mass transfer (low Biot numbers for mass transfer), as shown in Figure 4.3 (c, d). Inulin addition to agave syrup, on the other hand, do not markedly increase solution viscosity (Zongo et al., 2021) and thus, sucrose is still lost (Figure 4.3b).

As sample thickness increased to 1.5 cm, lower overall gains (p<0.05) of individual sugars were achieved in different agave syrup osmotic solutions (Figure 4.4 compared to Figure 4.3). Sample thickness increase has been previously found to reduce solids gain in osmotic

dehydration of mangoes (Zongo et al., 2021), so the same impact for individual sugar gain was found in this work. Despite previous results for 0.4 cm thickness samples, no sucrose loss was observed for 1.5 cm samples regardless of the osmotic solution used. This could be explained by sucrose saturation at the interface, since diffusion controlling process would slow mass transfer providing the necessary time to create a sucrose layer, and thus reverting the chemical potential of sucrose. Lenart & Flink, (1984) have already pointed out that for an osmotic treatment in a 60% sucrose solution, the osmotic penetration depth was limited by the formation of a compacted surface layer with resultant limited water removal. The formation of such a layer has a major effect on the control of mass transfer during OD, favoring water loss, limiting solute impregnations and reducing the loss of water-soluble solutes, such as ascorbic acid or fructose (Raoult-Wack, 1994). Bui et al. (2009) presented a mathematical model for osmotic dehydration of tomato in sucrose solutions where a solute saturation layer at the interface could be revealed from water and solids profiles. Please note that in Figures 4.4 (b, d) the sucrose gain curve presented a 'bump' around 1 hour of osmotic dehydration probably indicating saturation or a barrier at the surface (as previously pointed out for Figure 4.2d).

In general, individual sugar equilibrium gain/loss values for osmotic dehydration in multi-solute solutions were attained after 2 hours (0.4cm) and 4 hours (1.5cm), as shown in Figures 4.3 and 4.4. Equilibrium value was reached earlier for 0.4cm because of the higher surface contact which enables rapid mass transfer during externally controlled process (water loss and sugar gain).

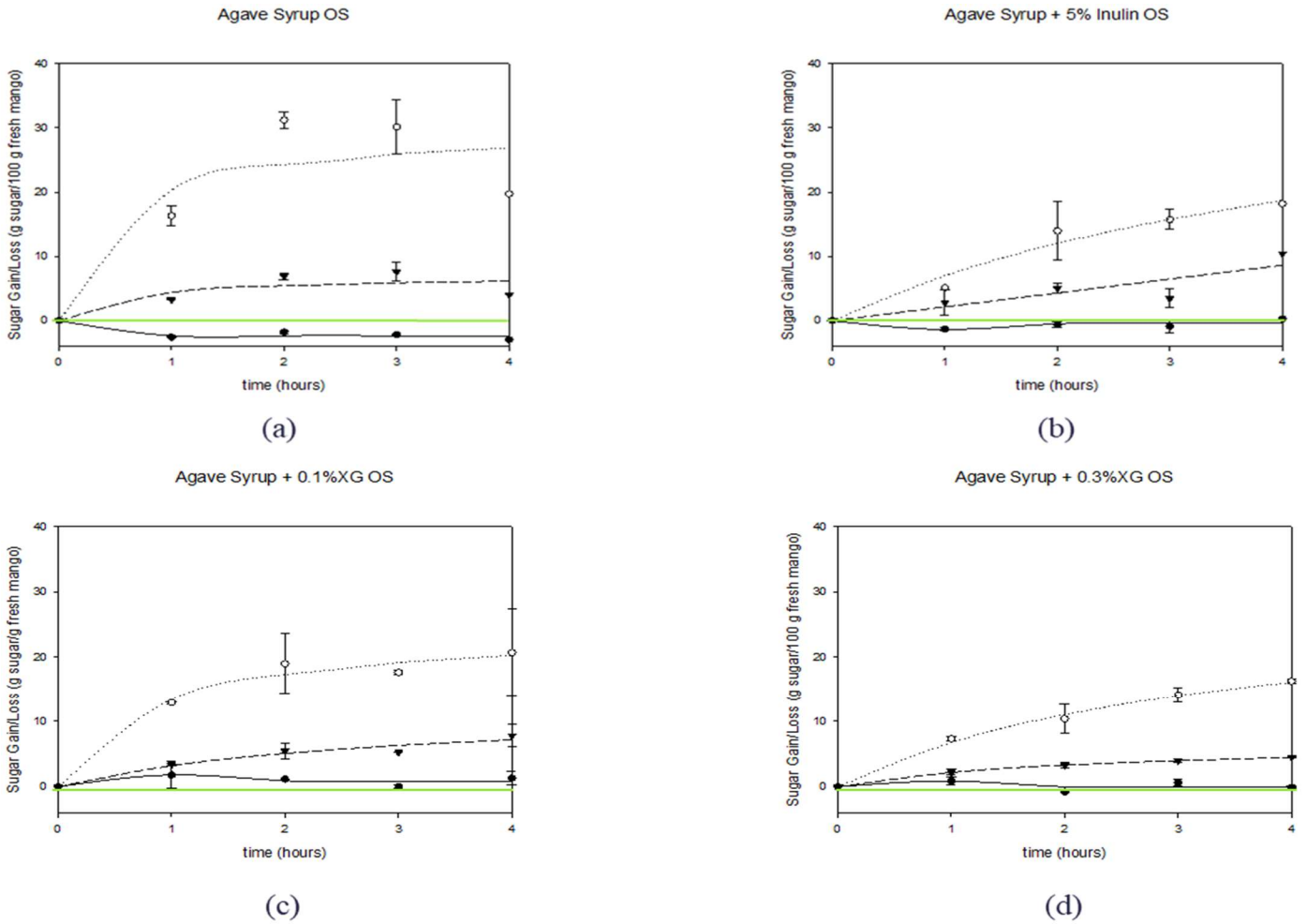

Figure 4.3: Individual sugar gain/loss during osmotic dehydration of 0.4 cm thickness mango slices at 40°C. Osmotic solutions (OS) are agave syrup (a), agave syrup with 5% inulin (b), agave syrup with 0.1% xanthan gum (c) and agave syrup with 0.3% xanthan gum (d). **Symbols** (experimental data): individual sugars are sucrose (●), fructose (○) and glucose (▼). **Lines** (predictions): — sucrose, …. fructose, ---- glucose, — reference.

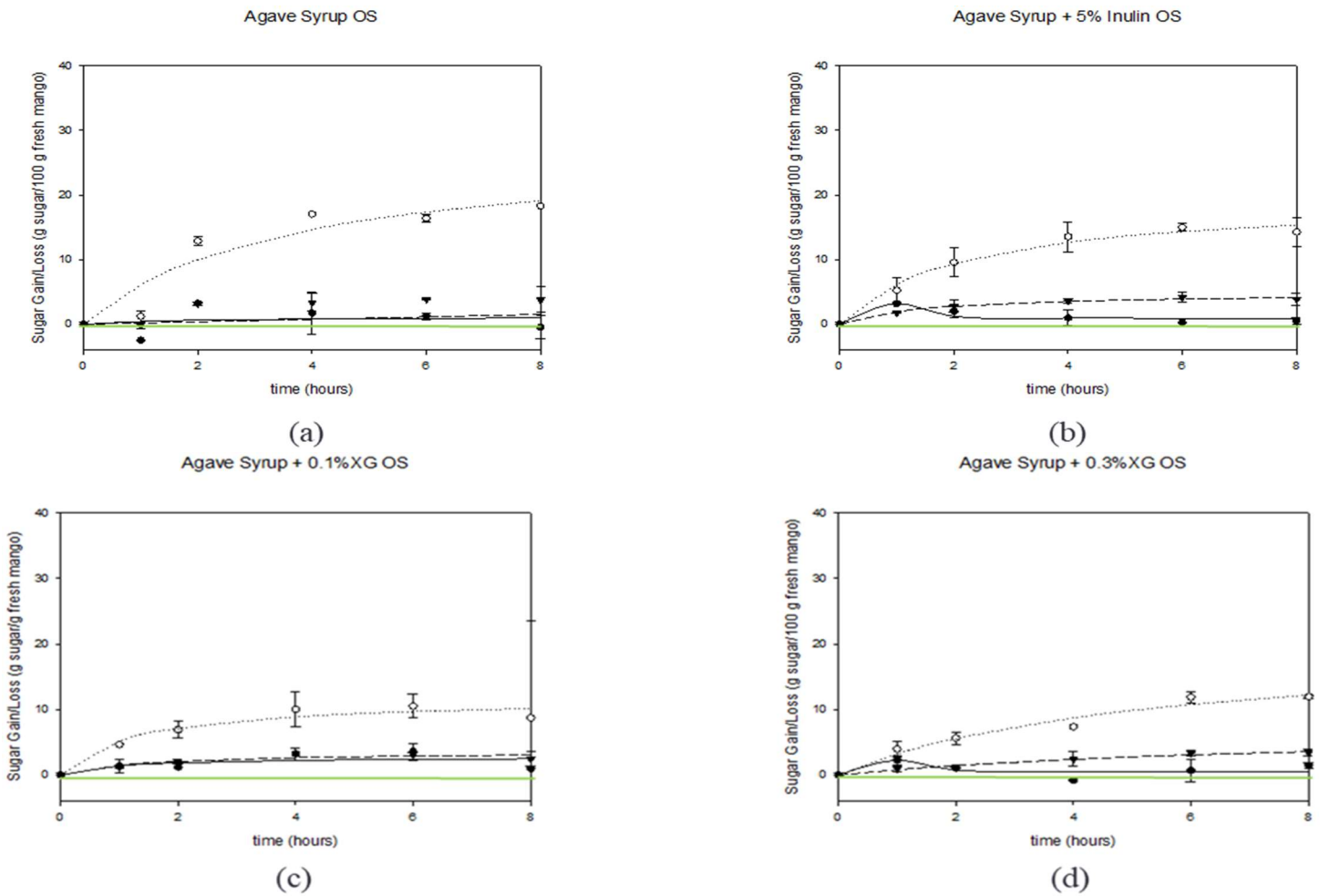

Figure 4.4: Individual sugar gain/loss during osmotic dehydration of 1.5 cm thickness mango slices at 40°C. Osmotic solutions (OS) are agave syrup (a), agave syrup with 5% inulin (b), agave syrup with 0.1% xanthan gum (c) and agave syrup with 0.3% xanthan gum (d). **Symbols** (experimental data): individual sugars are sucrose (●), fructose (○) and glucose (▼). **Lines** (predictions): — sucrose, …. fructose, ---- glucose, — reference.

4.5.3.3 Equilibrium sugar gain/loss

Equilibrium sugar gain or loss values were estimated from data on Figures 4.2 to 4.4 and are presented in Tables 4.2 and 4.3 for 0.4 and 1.5 cm thickness mango samples, respectively. From these Tables, equilibrium values confirmed that glucose had the highest sugar gain ($p<0.05$) for 0.4 cm (31.56±1.64 g/100g fresh mango) while it had 7% ($p<0.05$, calculated as the percentage ratio of glucose to total sugars) less uptake than fructose in 1.5 cm sample thicknesses during osmotic dehydration in single solute solutions. A loss of sugars was recorded for sugars which were not present in the osmotic solution for both thicknesses. For example, fructose and glucose losses were -1.36±0.40 and -1.15±0.09 g/100 g fresh mango when dehydrated in a sucrose solution (0.4 cm thickness slices). These results agree with those found by Ramallo & Mascheroni (2005), where it was explained that solute loss could be due to the water loss carrying away the minor solutes from the fruit tissue to the solution.

Table 4.2: Equilibrium values of individual sugar gain/loss achieved after osmotic dehydration of 0.4 cm thickness mango slices

Osmotic solutions	Equilibrium values (g sugar /100 g fresh mango)			
	Sucrose	Fructose	Glucose	Inulin
Sucrose	**21.03 ±0.39^{a}**	-1.36 ±0.40^{d}	-1.15 ±0.09^{c}	-
Fructose	-2.38 ±0.12^{d}	**24.63 ±1.01^{ab}**	-1.35 ±0.02^{c}	-
Glucose	-2.99 ±0.23^{d}	-2.11 ±0.08^{d}	**31.56 ±1.64^{a}**	**-**
AS	-1.96 ±0.15^{cd}	**30.67 ±2.76^{a}**	7.25 ±1.01^{b}	
AS+5%I	-0.45 ±0.42^{bc}	**18.96 ±1.05^{bc}**	5.32 ±0.75^{b}	**1.22±0.55**
AS+0.1%XG	0.84 ±0.26^{b}	**18.32 ±2.19^{bc}**	6.17 ±0.14^{b}	-
AS+0.3%XG	-0.08 ±0.17^{b}	**13.58 ±0.23^{c}**	3.91 ±0.02^{b}	-

Note: ^{a,b,c} Means in a column with different superscripts are significantly different ($p < 0.05$)

Table 4.3: Equilibrium values of individual sugar gain/loss achieved after osmotic dehydration of 1.5 cm thickness mango slices

Osmotic solutions	Equilibrium values (g sugar /100 g fresh mango)			
	Sucrose	**Fructose**	**Glucose**	**Inulin**
Sucrose	**6.07 ±1.27^{a}**	-1.64 ±0.87^{c}	-0.37 ±0.18^{c}	-
Fructose	-0.33 ±1.32^{b}	**20.47 ±2.01^{a}**	-0.65 ±0.36^{c}	-
Glucose	-0.46 ±0.49^{b}	-1.43 ±0.05^{c}	**17.56 ±0.17^{a}**	-
AS	0.82 ±1.84^{ab}	**17.03 ±0.02^{ab}**	3.69 ±1.15^{b}	-
AS+5%I	0.75 ±0.37 ^{ab}	**14.27 ±0.16^{ab}**	3.86 ±0.47^{b}	**0.85±0.06**
AS+0.1%XG	3.53 ±1.46 ^{ab}	**10.68 ±2.60^{b}**	2.96 ±0.76^{b}	-
AS+0.3%XG	1.41 ±0.51 ^{ab}	**11.26 ±1.27^{b}**	3.07 ±0.69^{b}	-

Note: ^{a,b,c} Means in a column with different superscripts are significantly different ($p < 0.05$)

Diffusion laws dictate that uptake of larger molecules should be lower than for smaller molecules (Cichowska et al., 2018). Thus, it was expected that in mono-solute solutions, sucrose gain (being a disaccharide) would be less than fructose and glucose (monosaccharides). This was observed in particular for mango slab thickness of 1.5 cm (Table 4.3), when diffusion controls the mass transfer.

As can be seen in Tables 4.2 and 4.3, dominant sugars in agave syrup solution with or without added polysaccharides was fructose. Impact of inulin or xanthan gum addition was a decrease in individual sugar gain or loss which was mostly significant (p<0.05) for the predominant fructose gain. Inulin addition led to more than 45% decrease of fructose gain in 0.4 cm thickness (Table 4.2) and a 20% decrease in 1.5 cm sample, compared to simple AS solution. In Table 4.2, for 0.4 cm thickness, sugar uptake was reduced from 30.67% to 18.32% and 13.58% in AS+0.1%XG and AS+0.3%XG, respectively. Similar decreasing effect was found for 1.5 cm thickness in Table 4.3. These results confirmed again the roles of large molecules (inulin) and thickening agents (XG) as an additional resistance to sugar transport towards the

mango (Zongo et al., 2021). Also, a gain of inulin (1.22±0.55 and 0.85±0.06 g/100g fresh mango) for 0.4cm and 1.5cm respectively was recorded in both sample thicknesses. Other equilibrium values shown in Tables 4.2 and 4.3 reflect the same conclusions explained previously for Figures 4.2 to 4.4: the importance of viscosity in lowering solute gain for smaller thicknesses, and the change in control for mass transfer from external to diffusion when thickness is increased. Sucrose is a minor component of AS solution (only present at 1%), while it is the dominant sugar in mango (please refer to Table 4.1). Therefore, according to the chemical potential gradient, it would normally leak out of the cell as seen in 0.4 cm thickness for AS+5%I and AS+0.3%XG providing negative gain values. However, in thicker samples, internal resistance to mass transfer increases, osmotic dehydration times are longer, and a sucrose barrier may form at the surface, therefore leading to less leakage and even leading to solute gain as seen for 1.5 cm thickness samples.

4.5.3.4 Inulin

Figure 4.5 shows the evolution of inulin gain of 0.4 and 1.5 cm mango slices during osmotic dehydration in solutions of agave syrup with 5% added inulin, which follows the same tendency of increase shown for other solutes (Figures 4.2-4.4). There is a rapid increase in inulin in the beginning of the dehydration followed by equilibrium gain after approximately 2 hours for both thicknesses. Thickness has a marked effect on final equilibrium values, which were estimated as 0.85 ± 0.07 and 1.22 ± 0.13 g inulin/100 g fresh mango, for 1.5 and 0.4 cm thickness slices, respectively (Table 4.3). As found previously for other individual solute gains (Figures 4.2-4.4) and for total gain of solids during osmotic dehydration (Zongo et al., 2021), increasing thickness produces a lower inulin uptake.

The present data on inulin uptake clearly shows that it is possible to impregnate fruits samples with inulin through osmotic dehydration by adding some inulin to the osmotic solution.

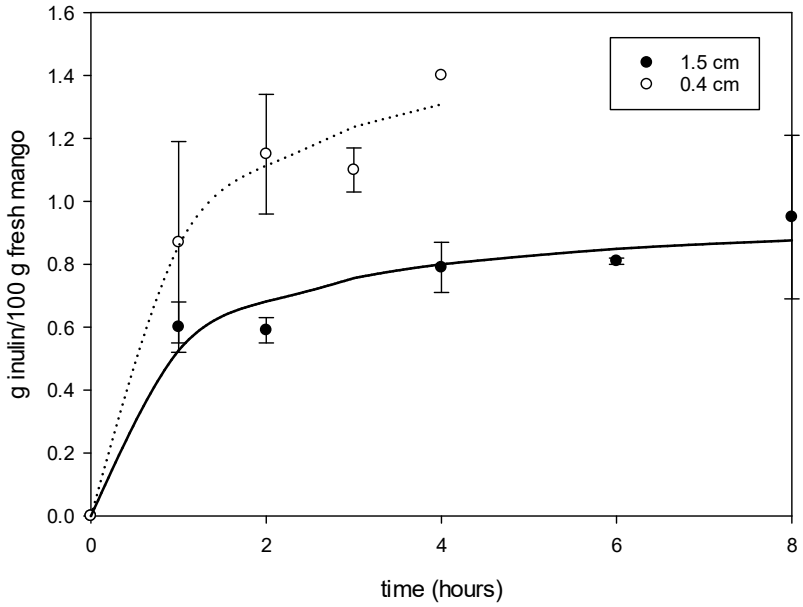

Figure 4.5: Inulin evolution in mango during osmotic dehydration

1.5 cm (●) and 0.4 cm (○) slices in solutions of agave syrup with 5% inulin

4.5.4 Microstructural changes of mango surface

4.5.4.1 Single-solute solutions

Figure 4.6 shows the scanning electron microscopy images of mango surface tissues after OD in different osmotic single-solute solutions, together with initial mango surface. Differences between fresh (Figure 4.6a) and dehydrated mango surfaces (Figures 4.6 (b-e)) were noted according to the type of osmotic solution used. Two types of openings were observed in the surfaces, pores (holes) and cracks. Fresh mango (Figure 4.6a) showed a uniform distribution of medium size pores (>100/image) throughout the surface, the rest of the surface being interconnected and mildly rough.

Surfaces of mango samples after osmotic dehydration in sucrose (Figure 4.6b), were found to have a thick compact layer with no pores but presenting cracks. These cracks at the surface might have been caused by differential shrinkage between the cellular vegetable tissue versus the carbohydrate layer during osmotic dehydration, or another more plausible possibility

could have been that these cracks were produced later by the extensive water loss during freeze-drying (used to prepare the sample for SEM analysis, please refer to Materials and Methods). During freeze-drying, differential retraction of the cell membrane may have occurred producing cracks of the sucrose layer on the surface of the mango. Thus, in general, after osmotic dehydration in a sucrose solution, the surface showed a thick layer, probably a barrier for water/solute exchange. Many previous reports indicated the formation of an impermeable sucrose layer on osmotically dehydrated fruit tissues treated with high sucrose concentration osmotic solutions (Bchir et al., 2012).

For treatments with inulin osmotic solution (Figure 4.6c), the surface of dehydrated mango appeared to have a thick layer of inulin with a good number of reduced size pores, when compared to the surface of fresh mango (Figure 4.6a). Inulin large molecules seem to mainly rest at the surface during osmotic dehydration, blocking the pores and thus, reducing their size, and affecting negatively mass transfer. Adding inulin to agave syrup do not significantly increase the solution viscosity as other ingredients, such as xanthan gum (Zongo et al., 2021), so the behavior of inulin creating a surface layer of reduced porosity (Figure 4.6b) could probably explain the lower sugar uptake during osmotic dehydration of mango in agave solutions with added inulin (Figures 4.3b and 4.4b).

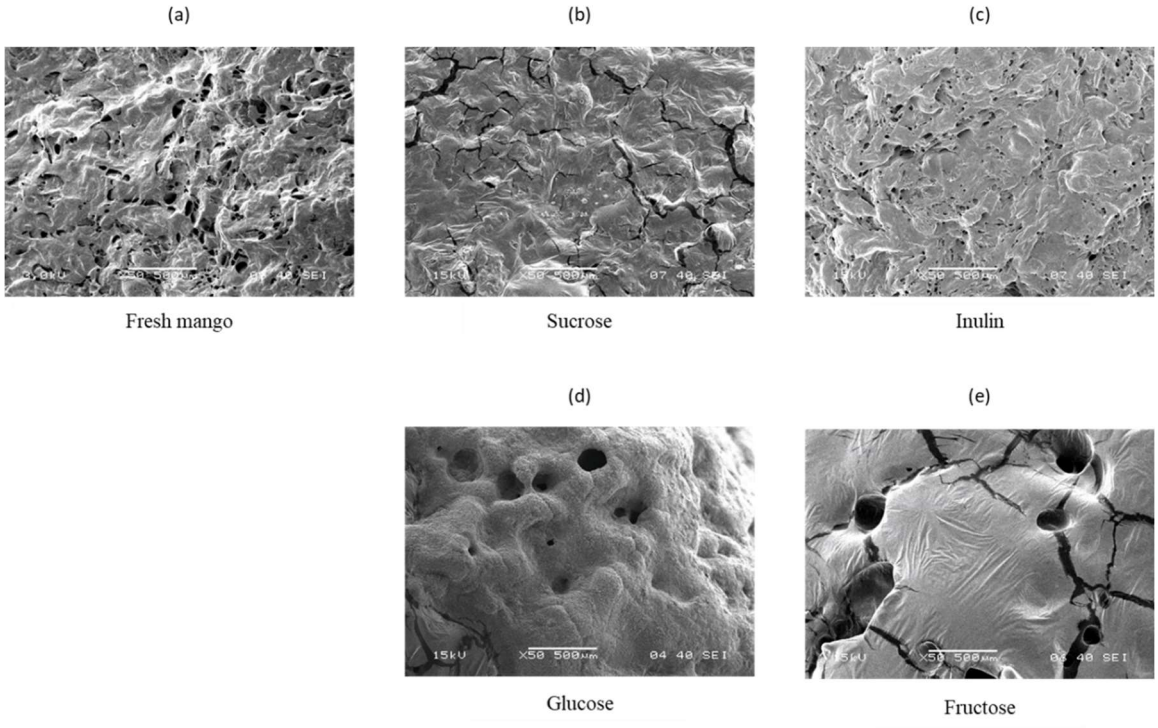

Figure 4.6: Surface microstructure of (a) fresh mango, and osmotically dehydrated mango with magnifications of 50 times, in (b) sucrose, (c) inulin, (d) glucose and (e) fructose, osmotic solutions.

Finally, Figures 4.6 (d, e) shows overall views of mango surfaces after osmotic dehydration in glucose or fructose solutions, respectively, where drastically reduced numbers of pores (≤10/image) are portrayed although they have significantly higher sizes than those for fresh mango (Figure 4.6a). The rest of the surface of treated mangoes present rugosity for glucose (Figure 4.6d) and smoothness for fructose (Figure 6e). This can be clearly observed in Figure 4.7, where SEM pictures with higher magnification reveal different topographies of mango surfaces after osmotic treatment with fructose (Figure 4.7a) and glucose (Figure 4.7b) solutions. Fructose deposits on the mango surface are observed as smooth thin sheets (Figure 4.7a), while glucose accumulates as ridges with many small peaks leaving a non-uniform rugged surface. This shows that glucose has crystallized on the surface, most probably during the sample preparation for SEM (i.e., during the freezing step prior to freeze-drying). It is known that glucose has lower solubility than fructose, and thus it crystallizes easier as the

temperature is lowered, characteristic that has been reported in the literature to separate fructose and glucose (Silva et al., 2010).

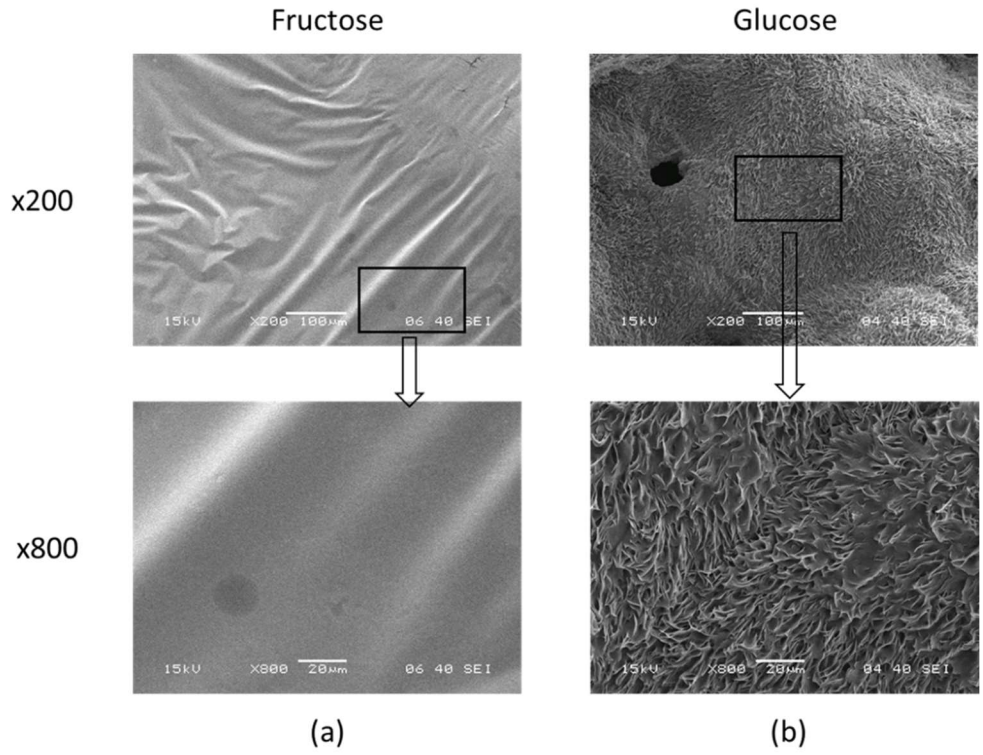

Figure 4.7: Surface microstructure of osmotically dehydrated mango with magnifications of 200 and 800 times, in (a) fructose and (b) glucose osmotic solutions.

4.5.4.2 Multi-solute solutions

Figure 4.8 shows the SEM images of mango surfaces treated with osmotic solutions made of agave syrup with and without additives together with those immersed in fructose osmotic solution (for comparison purposes). As explained in the previous section, mango treated in fructose solutions present smooth surfaces with big pores and cracks, as shown in Figure 4.8a. As well, Figures 4.8 (b-e) show that, for most cases, mango treated in multi-solute solutions show smooth surfaces with cracks, which seem less deep than in the case of pure fructose solution (Figure 4.8a) and covered by thin/transparent sheets. Mango surface treated with agave syrup with added 0.3% xanthan gum (Figure 4.8e) was an exception to the previous statement, since the surface appears rougher, the cracks deeper and the superficial carbohydrate layer, thicker. As explained earlier, cracks could be due to differential shrinkage during osmotic dehydration or freeze-drying after osmotic dehydration (please refer to the previous section). Contrary to what is presented in surfaces after OD in pure fructose solution (Figure 4.8a), those treated with multi-solute solutions revealed no pores on the mango surfaces (Figures 4.8(b-e)) but, in some cases, pores seem covered by thin layers (i.e., Figure 4.8c). Absence of pores could be explained by the incorporation of solutes during osmotic, or by cellular shrinkage (Bui et al., 2009).

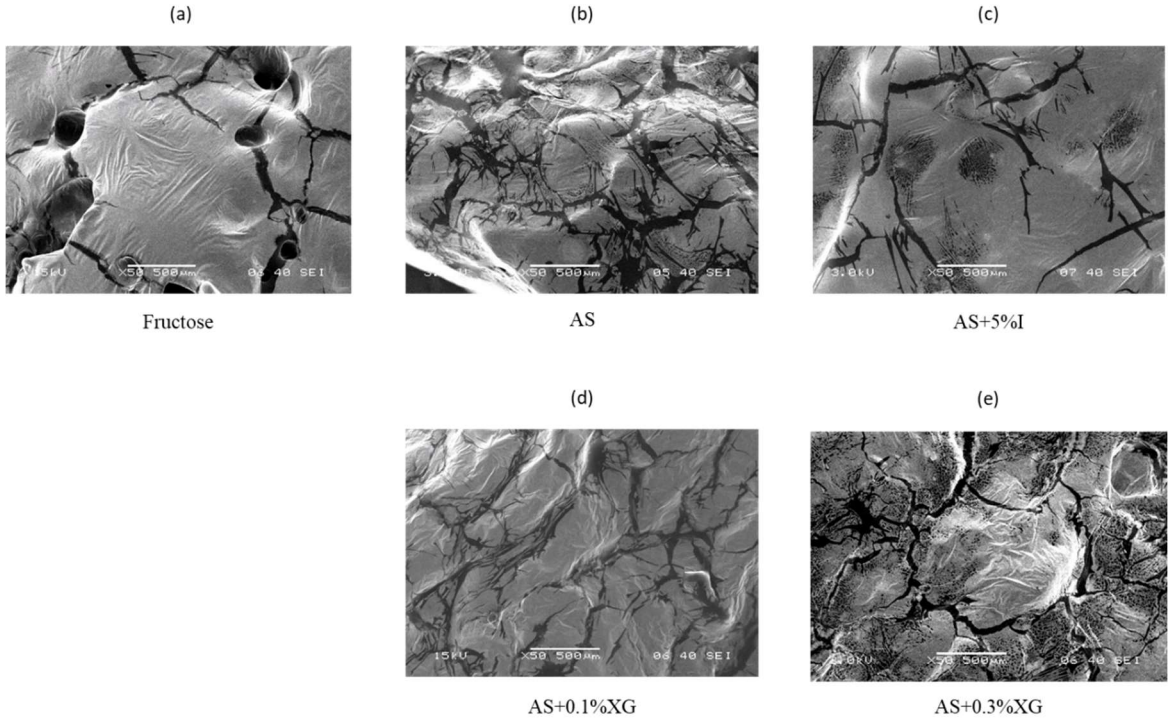

Figure 4.8: Surface microstructure of osmotically dehydrated mango with magnifications of 50 times, in the following osmotic solutions (a) fructose, (b) agave syrup, (c) agave syrup + 5% inulin, and agave syrup + (d) 0.1%XG or (e) 0.3%XG.

4.5.5 Final product composition

Tables 4.4 and 4.5 show the final individual carbohydrate composition of osmotically dehydrated mango slices of 0.4 cm and 1.5 cm thickness, together with water and solids contents at 4 and 8 hours, respectively. Only main components will be first analyzed (marked in bold in Tables 4.4 and 4.5).

4.5.5.1 Thinner samples

For 0.4 cm thickness (Table 4.4), composition of individual sucrose and fructose in dehydrated mangoes in single-solute osmotic solutions of sucrose and fructose, increased approximately 10-fold with respect to the initial composition (Table 4.1), due to impregnation and dehydration. However, when mango slices were immersed in a single glucose solution, the increase of the glucose composition with respect to the initial (Table 4.1) was 25-fold. A plausible explanation for this significant increase for glucose concentration could be related to the marked lower solubility of this particular sugar with respect to fructose and sucrose (Hanover & White, 1993). For instance, a previous study on the adsorption of acetylated model mannans on cellulose surfaces, (Berglund et al., 2020) showed that higher adsorption of the glucomannan on the surface was due to its overall poorer solubility.

Table 4.4: Final carbohydrate composition (g sugar or inulin/100 g final product) in 0.4 cm thickness mango slices after osmotic dehydration in different osmotic solutions

Osmotic solutions	Sucrose	Fructose	Glucose	Inulin	Water content (%)	Total solids content (%)	Solids content recalculated at 17.3% water
Sucrose	38.7±5.6^{a}	2.5 ±0.2^{c}	5.0 ±0.9^{b}	0	34.7±2.6^{c}	61.0±2.4^{a}	77.2±2.1^{a}
Fructose	1.7 ±0.1^{b}	31.6 ±7.1^{a}	1.4 ±3.1^{c}	0	30.7±0.4^{c}	60.3±0.9^{a}	72.0±1.6^{a}
Glucose	1.0 ±0.4^{b}	1.0 ±0.3^{c}	36.4 ±6.4^{a}	0	36.7±1.2^{bc}	60.0±2.1^{ab}	78.3±1.3^{a}
AS	2.5 ±0.4^{b}	36.9 ±0.2^{a}	11.1 ±0.3^{b}	0	36.0±2.1^{c}	60.3±1.2^{a}	78.0±1.0^{a}
AS+5%I	3.9 ±0.9^{b}	24.8 ±3.2^{b}	7.6 ±0.9^{b}	1.4 ±0.2	34.7±3.3^{c}	59.0±2.9^{ab}	74.8±3.5^{a}
AS+0.1%XG	5.8 ±0.6^{b}	26.1 ±0.7^{b}	9.3 ±0.6 ^{b}	0	42.7±1.2^{b}	54.0±0.8^{b}	77.9±1.7^{a}
AS+0.3%XG	6.1 ±0.2^{b}	26.5 ±0.6^{b}	8.6 ±0.8^{b}	0	50.0±0.8^{a}	47.3±0.4^{c}	78.3±1.5^{a}

Note: ^{a,b,c} Means in a column with different superscripts are significantly different ($p < 0.05$)

For multi-solute solutions, fructose content in dehydrated mango after immersion in agave syrup increased 12-fold compared to initial values (Table 4.1), somehow higher (though not significant) than when it is treated with pure fructose solutions, which was surprising

considering that agave syrup has lower percentage of fructose (79%). This could be also due to the presence of glucose (at 20%) in agave syrup. It is important to mention that, at smaller thicknesses such as 0.4 cm, mass transfer is prone to be controlled by external (solution) thermodynamic or mass transfer parameters, such as sugar solubility or solution viscosity.

Adding 5% inulin to agave syrup caused a reduction in fructose content, even though inulin does not have the ability to increase the viscosity of the solution (Zongo et al., 2021). The observed fructose reduction could be caused by the possibility that inulin, being a large molecule, would form a barrier at the mango surface blocking pores and limiting mass transfer, as discussed previously in the SEM image analysis (Figure 4.6c, Figure 4.8c). As well, the final product is enriched with 1.4 g inulin/100 g product.

Impact of osmotic dehydration on minor components (i.e., those absent or present in minor proportion in the osmotic solution) shows that sucrose content is noticeably reduced in osmotic dehydrated mango in fructose osmotic solution (56% less than initial content) and glucose osmotic solution (74%) (Table 4.4). This could be of interest for people requiring low sucrose diet due to intolerance (Benton, 2008; May, 1965).

4.5.5.2 Thicker samples

Table 4.5 shows as expected from previous results of carbohydrate gain/loss and equilibrium values (Figures 4.2-4.4 and Tables 4.2-4.3), that an increase in thickness lowers the overall contents of carbohydrates. However, the decrease for major carbohydrate content in mango slices dehydrated in single-solute osmotic solutions is not proportional to the findings for small thickness (Table 4.4). When immersed in sucrose solution, sucrose content in 1.5cm mango slices increased just 3.5-fold compared to initial values (Table 4.1) due to impregnation and dehydration, instead of 10-fold when slices were 0.4cm-thickness (Table 4.4). For fructose, a 10-fold increase from initial values was kept as found for smaller thickness. This differential behavior in sucrose content could be related to the formation of sucrose layer for thicker samples subjected to high sucrose concentration osmotic solution (as shown in Figure 4.2d, and in SEM analysis of Figure 4.6b). For glucose content of 1.5 cm slices dehydrated in glucose osmotic solutions, however, the increase from initial values

was 16-fold instead of 25-fold for smaller thicknesses. A possible explanation for this difference is that for thicker mango samples, osmotic dehydration takes place most probably under diffusion controlling mechanism, where solubility of glucose in the solution is a minor parameter while internal microstructure and temperature play key roles. For agave syrup osmotic solution, fructose concentration in mango slices is lower than for pure fructose osmotic solutions, as expected in internal diffusive control for mass transfer under a lower external driving force (agave syrup contains 79% fructose). Adding 5% inulin to agave syrup did not have an impact in lowering fructose content in mango slices (23.4 g sugar/100 g product), with respect to agave syrup osmotic solutions (22.3 g sugar/100 g product) but adding xanthan gum has an impact depending on the amount of ingredient added (18.5 and 17.7 g sugar/100 g product for 0.1%XG or 0.3%XG levels, respectively). The reductions with respect to values for agave syrup solutions, in the order of 18 to 20% respectively, are somehow lower for 1.5cm than for 0.4 cm (about 30%) probably due to the more pronounced effect of viscosity in mass transfer for external or mixed control for mass transfer at lower thicknesses.

Table 4.5: Final carbohydrate composition (g sugar or inulin/100 g final product) in 1.5 cm thickness mango slices after osmotic dehydration in different osmotic solutions

Osmotic solutions	Sucrose	Fructose	Glucose	Inulin	Water content (%)	Total solids content (%)	Solids content recalculated at 17.3% water
Sucrose	14.3±1.0^{a}	2.0±0.9^{d}	1.6±0.1^{c}	0	53.3±2.49^{c}	31.5±2.1^{a}	58.0±4.0^{a}
Fructose	4.8±1.5^{c}	29.6±4.0^{a}	1.1±0.4^{c}	0	51.0±2.16^{c}	37.7±0.94^{a}	63.7±2.2^{a}
Glucose	4.4±0.4^{c}	2.0±0.3^{d}	24.3±0.4^{a}	0	50.0±3.27^{bc}	39.3±3.40^{a}	65.0±1.8^{a}
AS	5.3±1.8^{bc}	22.7±2.7^{b}	5.8±1.7^{b}	0	51.3±1.70^{c}	36.0±6.16^{a}	61.0±8.7^{a}
AS+5%I	6.3±0.5^{bc}	23.4±0.3^{b}	7.2±0.4^{b}	1.1±0.1	53.1±0.50^{c}	36.7±6.5^{a}	64.7±0.7^{a}
AS+0.1%XG	10.0±2.7^{b}	18.5±0.6^{c}	6.0±1.2^{b}	0	53.3±0.94^{b}	32.7±2.05^{a}	57.9±3.7^{a}
AS+0.3%XG	6.6±0.6^{bc}	17.7±0.8^{c}	5.6±0.2^{b}	0	60.7±3.68^{a}	27.0±2.94^{a}	56.7±2.1^{a}

Note: ^{a,b,c} Means in a column with different superscripts are significantly different ($p < 0.05$)

Nutritional data from USDA for sweetened dehydrated mango indicates that water and sugar contents are 17.3% and 66%, respectively (USDA, 2021). In Tables 4.4 and 4.5, total solids content at the actual water content of OD mangoes was presented together with estimated sugar content for the recommended USDA water content values (17.3%), these estimations were used for comparison purposes. From Table 4.5 (1.5 cm slices), a total of 27.0±2.94 g sugar/100 g of product and 60.7±3.68 % of water content can be obtained in dehydrated mangoes with 0.3%XG added to agave syrup at 60 °Brix, which represents an estimated 14% decrease in sugar content with respect to recommended values given by USDA. Adding inulin instead of xanthan gum, cause a slight 2.3% decrease in total sugar content with respect to USDA values for dried sweetened mangoes (USDA, 2021), but on the other hand, it is possible to enrich the samples with 1.1 g inulin/100 g product. Finally, when mango slices (1.5 cm) are immersed in sucrose osmotic solution, a total of 31.5 g sugar/100 g of product was attained, which represents a decrease of 12% with respect to USDA values for dried sweetened mangoes (USDA, 2021) pointing out the interesting effect of forming a sucrose layer on the surface of thick slices of mango to block the solids intake. In all cases, osmotically dehydrated mangoes should be further dried by other methods in order to reduce water content to stable values reported by USDA.

4.6 Conclusion

This study shows how sugar profiles could be modulated in mango (*cv*. Tommy Atkins) after osmotic treatment with different agave syrup formulations, and as well it was possible to elucidate the most important parameters to lower sugar content in dehydrated mangoes. Thickness played an important role in changing the control for mass transfer pointing out the key variables for sugar uptake reduction. For thinner samples, external mass transfer control prevailed and solution properties such as viscosity for multi-solute solutions and sugar solubility for single-solute solutions, controlled sugar uptake. Increasing thickness lowered overall sugar gain and reduced individual sugar compositions since predominant diffusion slow dehydration and solids uptake rates. In particular, for the sucrose osmotic solution, a layer deposited on the surface of the mango created an additional barrier for mass transfer

being effective in lowering sugar uptake. Inulin addition to agave syrup solution decreased sugar uptake due to its ability to form a layer on the mango surface. Xanthan gum thickening capacity showed a positive effect in lowering sugar uptake. To end, it was possible to enrich mango samples with inulin by adding inulin to the osmotic solution.

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Transition vers l'article 3

Le chapitre 4 a consisté en une analyse chromatographique du profil en sucres individuels de la mangue dans des solutions osmotiques de mono et multi-solutés. Les résultats ont montré que la mangue s'est enrichie du sucre prédominant dans la solution osmotique et s'est appauvri en sucres absents dans le cas des solutions à un seul soluté (sucrose, fructose, glucose). Dans le cas des solutions de multi-solutés, l'ajout de la gomme xanthane et d'inuline et une plus grande épaisseur de l'échantillon ont permis de réduire le gain en sucres individuels. Les possibles mécanismes pour lesquels le gain en sucre pourrait être réduit dans certaines conditions ont été visualisés par microscopie différentielle à balayage.

Les chapitres antérieurs ont permis d'observer comment des variables **externes ou d'opération** peuvent agir sur le transfert de matière durant la déshydratation osmotique. Le prochain chapitre (5) permettra d'analyser l'effet de prétraitements de congélation/décongélation et de champ électrique pulsé sur des propriétés **internes** de la mangue (microstructure) pour ainsi moduler le gain en sucres, la perte en eau et le profil de sucres durant la déshydratation osmotique de la mangue dans des solutions de sirop d'agave.

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Zongo, A. P., Khalloufi, S., Mikhaylin, S., & Ratti, C. (2022). *Pulsed electric field and freeze-thawing pretreatments for solids uptake modulation during osmotic dehydration of mango*. *Foods*.

Une partie des résultats obtenus de ce chapitre a été présentée sous forme de communication orale au « *22nd International drying symposium* » du 26 au 29 Juin 2022. Titre: *Sugar uptake during osmotic dehydration of fresh and frozen-thawed mango slices in agave syrup solutions.*

Chapter 5: Pulsed electric field and freeze-thawing pretreatments for sugar uptake modulation during osmotic dehydration of mango

5.1 Résumé

La cinétique de déshydratation osmotique dépend de la microstructure des tissus alimentaires. La modulation de la porosité de la mangue pourrait aider à améliorer de façon sélective l'élimination de l'eau au détriment du gain de sucre. Dans cette étude, des prétraitements de congélation-décongélation (congélation à -36°C pendant 2 semaines et décongélation à 4°C pendant 24 heures) et champ électrique pulsé (1 kV/cm, 10 et 30 nombres d'impulsions), ont été appliqués sur des tranches de mangue de 1 cm d'épaisseur avant la déshydratation osmotique conduite à 40°C pendant 4 heures. Trois solutions différentes de sirop d'agave à 60° Brix avec ou sans polysaccharides ajoutés (inuline ou gomme xanthane) ont été utilisées pendant la déshydratation osmotique. La perte d'eau (*WL*), le gain de sucre (*SG*) et les images de la structure cellulaire de la mangue ont été utilisés pour comparer les effets des prétraitements sur la performance de la déshydratation osmotique de la mangue. Les résultats ont indiqué que le prétraitement par champ électrique pulsé augmentait légèrement la perte d'eau lors de la déshydratation osmotique, contrairement à la congélation-décongélation, qui dans la plupart des cas entraînait une diminution. En ce qui concerne le gain en solides, en raison des dommages plus importants induits par la congélation-décongélation sur les tissus de la mangue, le gain en solides était plus élevé que pour les mangues fraîches et prétraitées par champ électrique pulsé. L'utilisation de la gomme xanthane comme additif à la solution de sirop d'agave a contribué à réduire l'absorption de sucre dans la mangue après la congélation-décongélation en raison d'une augmentation de la viscosité de la solution. Un rapport *WL/SG* similaire a été obtenu avec de la mangue congelée-décongelée en solution avec de la gomme xanthane. Par conséquent, dans le cas de la mangue ayant subi la congélation-décongélation, il est recommandé d'utiliser une solution osmotique à viscosité élevée pour obtenir une faible absorption de sucre dans le produit final.

5.2 Abstract

Osmotic dehydration kinetics depends on food tissue microstructure; thus, modulation of mango porosity could help selectively enhance water removal over sugar gain. In this present study, pretreatments of freeze-thawing (freezing at −36 °C for 2 weeks and thawing at 4 °C for 24 h) and pulsed electric field (1 kV/cm, 10 and 30 pulse numbers), were applied to mango 1 cm-thickness slices prior to osmotic dehydration conducted at 40 °C for 4 h. Three different 60 °Brix agave syrup solutions with or without added polysaccharides (inulin or xanthan gum) were used in the osmotic dehydration operation. Water loss (*WL*), sugar gain (*SG*) and microstructure images were used to compare the effects of pretreatments on mango osmotic dehydration efficiency. Results indicated that pulsed electric field (PEF) pretreatment increased slightly *WL* during osmotic dehydration, contrary to freeze-thawing (F-T), which for most cases led to a decrease. As for solids uptake, due to higher damage induced by F-T to mango tissue, *SG* was higher than for fresh and PEF pretreated mangoes. Using xanthan gum as additive to agave syrup solution, helped to decrease sugar uptake in frozen-thawed mango due to an increase in solution viscosity. A similar *WL/SG* ratio was obtained with frozen-thawed mango in solution with xanthan gum. Therefore, in the case of frozen-thawed mango, it is recommended to use an osmotic solution with high viscosity to obtain low sugar uptake in the final product.

5.3 Introduction

Consumers are encouraged to include more fruits and vegetables in their diet (Sadler et al., 2019). Processed fruits (semi-dried, dried, juice, purees) include a wide range and are available throughout the year, unlike fresh fruits which are seasonal, such as in the case of mango. Classified in the tropical fruit category, mango is available for a short period of time. Its taste, flavor and nutrients (vitamins B1, B2, C, A, antioxidant beta-carotene) contribute to its success with consumers worldwide (Izli et al., 2017). Drying is the most common method used to prolong mango shelf life and additionally contributes to the economy in tropical countries where mango grows most commonly. Methods of mango drying consist of conventional drying (air drying) or non-conventional drying such as osmotic dehydration.

Due to the high temperature used in air-drying technology (often above 70 °C), oxidation and Maillard reactions may occur leading to degradation of beneficial components such as polyphenols, pigments, vitamins, etc. (Drouzas et al., 1999; Sehrawat et al., 2018). Osmotic dehydration, which consists of the partial removal of water through the immersion of a cellular food in a hypertonic solution (sugars are the most used osmotic solute), is recognized as a minimal processing technology due to the medium or low temperature used (Torreggiani, 1993). Osmotic dehydration is a simultaneous countercurrent mass transfer process, where water is lost, and sugar is gained.

Osmotic dehydration's main purpose is to decrease water activity to preserve fruits or vegetables for longer periods. In addition, it is a low energy process technology which produces high quality products due to low temperatures and absence of oxygen which restrains enzymes responsible for browning during processing (Ahmed et al., 2016). Osmotically dehydrated products are reported to have better color, flavor, taste, texture and nutrients retention, close to those of the fresh product (Ahmed et al., 2016). It is also possible to modulate food chemical composition by incorporating high quality nutrients into the final product (Sravani & Saxena, 2021). However, major drawbacks of osmotic dehydration are the uptake of sugar and slowness of water loss. Nowadays, consumers tend to reduce sugar intake in their diet, so increased sugar content in dehydrated mangoes may negatively impact their commercial attribute and limit consumption. Many factors influence osmotic dehydration kinetics and can be optimized for sugar uptake reduction. Microstructure of the

tissue shows variation in pore distribution and interconnectivity within the fruit matrix which affects the pathways for mass transfer in osmotic dehydration (Ahmed et al., 2016). Water loss and solute uptake increase as time, temperature, or solution concentration increase, while an increase in solute molecular weight decreased solute uptake. High temperature favors mass transfer but may lead to undesirable changes to the plant material at temperatures above 50 °C in terms of color, flavor, aroma and nutrients degradation, in addition to enhancing sugar gain which nowadays is unfavored by consumers (Shi & Xue, 2008). The treatment time can be reduced depending on conditions such as concentration, temperature of the osmotic solution, and pre-treatments (pulsed electric field, ultrasound, high hydrostatic pressure, etc.). The rate of osmotic dehydration increases with the solution concentration because osmotic pressure and chemical potential are proportional to concentration (Phisut, 2012).

Several methods have been proposed to address sugar uptake issue in osmotic dehydration, from using centrifugal force (Azuara et al., 1996; Barman & Badwaik, 2017) or high molecular solute such as corn syrup solids (Lazarides et al., 1995) to coating (Matuska et al., 2006) and high viscosity solutions (Zongo et al., 2021). More recently, pretreatments that modify cell structure distribution of the material to be dehydrated were explored to modulate osmotic dehydration kinetics in terms of favoring water loss, with minimal additive uptake. Modification of cell microstructure leads to removal of barriers in mass transfer, such as cell wall integrity and entrapped gas in the pores, and additionally increases porosity (Liu et al., 2019; Phisut, 2012).

Pretreatments such as freeze-thawing (F-T) and pulsed electric field (PEF) are well known for their direct effect on material microstructure and have been used to enhance drying kinetics (Taiwo et al., 2001; Toepfl & Knorr, 2006). In many countries, freezing is necessary to keep mangoes for longer periods due to short production seasons throughout the year, and thus mangoes are often frozen-thawed prior to osmotic dehydration. The first step in F-T process is freezing, during which ice formation modifies the tissue structure by depolymerization of cell walls, cell membrane breakage and osmotic pressure alteration (Li et al., 2018), in addition to degassing (Phisut, 2012). The second step is thawing, which leads to softening of the tissue through ice melting and drip loss (Li et al., 2018). Due to these

physicochemical modifications induced to the tissue, F-T has been successfully used to enhance air drying of apple, eggplant and beetroot (Vallespir et al., 2018), blueberries (Zielinska et al., 2015), okra (Xin et al., 2021) and garlic (Feng et al., 2020). In the case of osmotic dehydration, rarer use of F-T as a pretreatment has been reported, but it has been used in the case of apples (Lazarides & Mavroudis, 1995)**.** This study showed an increasing effect of F-T pretreatment on sugar gain. PEF is the application of short repeated high voltage pulses to a biological tissue (Gürsul et al., 2016). During PEF application, when the electrical potential difference of the cell's membrane, also known as transmembrane potential, reaches a threshold value of 0.2–1.5V (Vorobiev & Lebovka, 2009; Weaver & Chizmadzhev, 1996), it can induce a temporarily loss of membrane semi-permeability called electro-permeabilization (Vorobiev & Lebovka, 2009) and the openings in cell membranes through pore formation or expanding of initial pore size, known as electroporation (Asavasanti et al., 2011; Chauhan et al., 2019). The PEF treatment outcome is related to field strength, pulse number, energy, frequency and total treatment time (Angersbach et al., 2000; Vorobiev & Lebovka, 2008). An estimation of cell permeabilization induced by PEF can be made through the disintegration index, Zp. Tedjo et al. (2002) reported that an increase in Zp in mango tissue (*cv.* Kent) after PEF treatment is proportional to the increase of field strength and pulse numbers. Increases in water loss and solids uptake after osmotic dehydration of PEF treated mango and apples were found in osmotic dehydration studies from Amami et al. (2006) and Tedjo et al. (2002) respectively. Some authors, though, have observed lower sugar uptake after PEF pretreatment, such as the work on osmotic dehydration of kiwifruit in a 61.5 °Brix sucrose solution (Traffano-Schiffo et al., 2017). Therefore, it could be possible to optimize the conditions of the PEF pretreatment to modulate tissue microstructure in order to favor water loss over sugar uptake.

Thus, the present study explores the impact of pretreatments such as F-T and PEF on mango slices so as to modify the fruit tissue microstructure before osmotic dehydration with the aim of obtaining higher water loss with minimal sugar uptake.

5.4 Materials and Methods

5.4.1 Mango samples preparation for experiments

Fresh Tommy Atkins mangoes were purchased in a local supermarket and kept at ambient temperature (20 °C) for 4–5 days before further processing. Firmness was measured with a texturometer EZ-test (Shimadzu, Kyoto, Japan), as explained in Section 5.4.1.1, to select mangoes for the experiment. Then, mangoes were washed, rinsed, peeled, and cut manually into cuboid slices of 2.5 cm width, 5 cm length and 1 cm thickness. The samples weighed approximatively 12 g ± 2. Random samples were then selected for further physicochemical analysis.

5.4.1.1 Firmness analysis

Firmness of fresh samples (5 replicates) were measured with an EZ-test texturometer (Shimadzu, Kyoto, Japan) following a slightly modified protocol from Tedjo et al. (2002). A cuboid shaped mango sample was placed between a flat probe and a flat platform, both separated by a 10 mm distance. Firmness was recorded as the maximum force in Newtons (N) required to compress the sample to a depth of 5 mm on the platform with a speed of 1 mm/s.

5.4.1.2 Titratable acidity, pH and soluble solids

Mango flesh was homogenized in a blender (Hamilton Beach, Markham, ON, Canada) to obtain a puree. Fifty (50) mL of puree was used to measure the pH with an automatic titrator Orion T910 (Thermofischer scientific, Ottawa, ON, Canada). For titratable acidity measurements, 40 mL of water was added to 10 mL of puree into a beaker and homogenized. Then, titration was done in an automatic titrator Orion T910 with 0.1 N NaOH solution until point of neutrality (Jayasena & Cameron, 2008). Triplicates were made for each analysis. Results were reported as percentage of citric acid:

$$\text{Percentage citric acid} = \frac{\text{Titer} \times 0.0064 \times 100}{10\ (\text{ml juice})} \tag{5.1}$$

where factor for citric acid was 0.0064 (AOAC, 1990).

Total soluble solids (°Brix) was measured in mango puree with a refractometer Atago (PAL-1, Tokyo, Japan).

5.4.2 Pre-treatments

5.4.2.1 Freeze-thawing

Mango cuboid samples were frozen at −36 °C in a Sanyo medical freezer (MDF 235, Gunma, Japan) for 2 weeks, then thawed at 4 °C in a refrigerator for 24 h before osmotic dehydration. F-T was used as a control for total tissue destruction for comparison purposes with PEF pre-treatment. Please note that fresh mangoes were cut in the same cuboid shape on the day of the osmotic dehydration experiment to be used as control for F-T and PEF.

5.4.2.2 Pulsed Electric Field

PEF treatment was carried out in a PEF-Cellcrack III batch system (DIL, Quakenbrück, Germany) with output voltage up to 30 kV, and frequency of 2 Hz. The treatment chamber consisted of two parallel stainless electrodes separated by 300 mm distance. Two liters of tap water (~0.2 mS/cm) was added to the treatment chamber and served as a conductor between the electrodes.

Cuboid mango slices (2.5 cm × 5 cm, 1 cm thick) were first weighed and then about 420 g were added to the water in the treatment chamber. Pre-treatment was made at 1 kV/cm field strength, and number of pulses applied were 10 and 30. After each treatment, mango slices were gently blotted onto a paper to remove superficial water.

Prior to osmotic dehydration, pretreated samples were tested to estimate the tissue damage provoked by PEF treatments through disintegration indexes based on firmness or electrical conductivity (please refer to details in the following sections). Control was prepared according to (Grimi et al., 2010; Wiktor et al., 2016) by freezing samples at −36 °C for 24 h

followed by thawing at 21 °C for 24 h. Samples were then kept at ambient temperature before firmness and electrical conductivity measurements.

Membrane permeabilization is induced during PEF through formation of pores. To measure the extent of permeabilization, a disintegration index Z is the most common indicator since its value increases with the pretreatment intensification (PEF strength, pulses number, pulse width and duration). The disintegration index Z ranges from 0 (intact membrane) to 1 (totally disintegrated membrane) (Lebovka et al., 2002). Two methods were used for the disintegration index determination. The firmness method and the electrical conductivity method were used to assess the change in mango tissue conductivity due to PEF treatment.

5.4.2.3 Tissue disintegration evaluation

5.4.2.3.1 Firmness method

Firmness (F) of fresh, frozen-thawed (used as control as described in the previous paragraph) and PEF treated samples was measured with an EZ-texturometer as described in (Section 5.4.1.1). At least, 5 samples were measured for each treatment at ambient temperature. Disintegration index of the tissue based on firmness (Z_F) was then estimated with the following equation (Olivera et al., 2013):

$$Z_F = \frac{F_i - F_t}{F_i - F_d} \qquad (5.2)$$

where F_i, F_d and F_t are the firmness (N) of fresh, F-T (totally destroyed tissue) and PEF treated mango samples, respectively.

5.4.2.3.2 Electrical conductivity method

Electrical conductivity (σ) was obtained indirectly through electrical resistance measurements by using a multimeter (Mastercraft model 052-0052-2, Toronto, ON, Canada) connected to two lab-made plate copper electrodes (2.5 cm × 5 cm, 1 mm thick) between

which a cuboid shaped mango sample (fresh, F-T or PEF treated) was placed. At least 5 samples were tested for each type. The equation below was used to calculate the electrical conductivity according to Zareifard et al. (2003):

$$\sigma = \frac{E}{R \times S} \tag{5.3}$$

where σ is electrical conductivity (S/m), E is sample thickness (m); R is the electrical resistance (Ω) and S represents the surface of the electrode (m^2). Then, the disintegration index of the tissue ($Z\sigma$) based on electrical conductivity was estimated as in Lebovka et al. (2002):

$$Z_\sigma = \frac{\sigma_i - \sigma_t}{\sigma_i - \sigma_d} \tag{5.4}$$

where σi, σt and σd are the values of electrical conductivity (S/m) for initial, for PEF and F-T treated mango, respectively.

5.4.3 Osmotic solutions

Osmotic solutions used for this study were based on a 'lab-made' model of agave syrup (AS) to which inulin or inulin + xanthan gum was added. Agave syrup (AS) consisted of a mix of simple sugars to mimic proportions in dry weight in real agave syrup (79% fructose, 20% glucose, and 1% sucrose) in distilled water at 60 °Brix. Then, 5% inulin or (0.3% xanthan gum + 5% inulin) were added to obtain two other osmotic solutions with presence of long-chain polysaccharides (these solutions were labelled as AS+5%I and AS+5%I+0.3%XG, respectively). The concentrations of inulin and xanthan gum were chosen for effective sugar reduction based on previous published results (Zongo et al., 2021). An Atago Pocket refractometer PAL-2 (Tokyo, Japan) was used to measure soluble solids content (°Brix) of the solutions.

5.4.4 Osmotic dehydration

Osmotic solution was heated up to 40 °C in a water bath (Fischer scientific, model Isotemp 1016 S, Pittsburgh, PA, USA), before immersing mango samples, which were identified in individual cages. The solution to sample ratio was 1:100 (*w:w*) to avoid dilution. Osmotic dehydration was carried out for up to 4 h. F-T samples and PEF-treated samples were osmotically dehydrated in two separate experiments and each experiment had its own control (non-treated samples). A sample was taken out of the solution every 1 h, rinsed quickly and blotted with paper, then weighed.

Afterwards, osmotic dehydrated samples were lyophilized at 20 °C, 30 millitorr vacuum for 72 h. Freeze-dried samples were then weighed (Mettler Toledo AB104-S, Greifensee, Switzerland) to obtain their dry mass (M_d). Water Loss (*WL*) and Solids Gain (*SG*) represent, respectively, the water removed and the solids uptake from the mango samples after osmotic dehydration, based on initial mass of mango samples:

$$WL\ (\%) = 100 * \frac{(P_O - M_{dO}) - (P - M_d)}{P_O} \quad (5.5)$$

$$SG\ (\%) = \ 100 * \frac{M_d - M_{dO}}{P_O} \quad (5.6)$$

where M_{d0} is the initial dry matter (g); M_d, final dry matter (g); P_0, initial sample mass (g), and P, the final sample mass (g). For water loss unit was g of water/100 g fresh mango and for sugar gain it was g of sugar/100 g of fresh mango. Additionally, osmotic dehydration efficiency (*ODE*) was estimated as the ratio of water loss to sugar gain at equilibrium:

$$ODE = \frac{WLeq}{SGeq} \quad (5.7)$$

ODE was expressed in g water lost/g solids gained and *WLeq* (g of water/100 g fresh mango) and *SGeq* (g of sugar/100 g of fresh mango) represent, respectively, water loss and sugar at equilibrium.

5.4.5 HPLC analysis for sugar profiles

Fresh, frozen-thawed and PEF-treated, mango samples before and after osmotic dehydration, were used for the HPLC analysis. The same method as in chapter 4, section 4.4.5 was followed for sample and standard preparation and injection.

5.4.6 Microscopic analysis

Fresh, frozen-thawed and PEF-treated mango samples before and after osmotic dehydration were individually immersed in classic plant fixator (FAA) (Kim, 2019), a mixture of 10 mL of formaldehyde 37%, 35 mL distilled water, 5 mL of glacial acetic acid and 50 mL of alcohol 99%. Before microscopic observation, the samples were taken out of the FAA and cut with a slicer into 1-cm cubes. The microscopic images were taken at the mango slice surface by confocal method (Loginova et al., 2011) with a Leica SP8 microscope (Ontario, ON, Canada). Image J software program (version 2.1.0/1.53c, Java 1.8.0, National Institutes of Health, Bethesda, MD, USA) was used for images extracting and scaling at 100 μm (Koch et al., 2022).

5.4.7 Statistics analysis

Experiments were made in triplicate, except for firmness (5 replications). The statistics analysis was carried on with Rstudio software (RStudio-1.2.5033, Integrated Development for R. RStudio, Inc., Boston, MA, USA). Data were subjected to ANOVA (analysis of variance) and means were compared with Tukey test. The confidence level used was 95%.

5.5 Results

5.5.1 Mango physico-chemical characteristics

Table 5.1 shows the physico-chemical characteristics of mangoes used for osmotic dehydration experiments before F-T and PEF pretreatments. Average values of pH, titratable acidity, soluble solids, moisture content, and firmness are reported. The average pH value of fresh mango was 3.86 ± 0.37. Mango is considered an acidic fruit due to its content in citric and malic acids, with a pH generally lower than 6. Values of pH in literature for ripened Tommy Atkins mango ranged from 3.2 to 4.5 (Dutra et al., 2005; Lucena et al., 2000; Santos et al., 2008). The values reported in this study for pH are therefore in accordance with literature. Furthermore, titratable acidity which is correlated to pH, was 0.57 ± 0.07 g citric acid/100 g fresh mango which is close to value reported by Sulistyawati et al. (2018) for *cv.* Kent (0.58 ± 0.02). Soluble solids (12.16 ± 2.56) and moisture content (85 g water/100 g fresh mango ± 0.03) agreed with the literature. For instance, results in Maldonado-Celis et al. (2019) and Sulistyawati et al. (2018) presented soluble solids of 15.7 ± 0.3 and 14.98, respectively, while moisture content was stated as 84.87 ± 1.76 (Sulistyawati et al., 2018) and 87.24 ± 1.14 (Tedjo et al., 2002).

Table 5.1: Characteristics of mango for experimentation

Parameters	Values
Total soluble solids (°Brix)	12.16 ±2.56
Titratable acidity (g citric acid/100 g fresh mango)	0.57 ±0.07
pH	3.86 ±0.37
Moisture (g water/100 g fresh mango)	85 ±0.03
Firmness (N)	49.21 ±11.36

Average firmness value was 49.21 N ± 11.36. Compared to the literature, mango firmness used in this study was two times the value of 25.49 N reported by Sulistyawati et al. (2018) for ripe Kent mango, while it was close to the 52.31 N firmness value reported by Santos et al. (2008) for Tommy Atkins. Many factors influence mango ripeness: number of days since full flowering (Yahia, 1998), geographical region, microclimatic conditions of the mango trees, long distance during transport (i.e., from Brazil to Quebec), storage condition, etc.

(Gianguzzi et al., 2021). These can lead to heterogeneous batches that may have different physico-chemical characteristics (Galán Saúco & Lu, 2018; Gianguzzi et al., 2021; Lalel et al., 2003; Sivakumar et al., 2011) among which firmness is predominantly impacted, consequently leading to difficulty in managing a uniform batch of mangoes. In this study, to select mangoes with uniform ripeness as accurately as possible, the parameters presented in Table 5.1 were all considered.

5.5.2 Effects of F-T and PEF pretreatments on osmotic dehydration kinetics

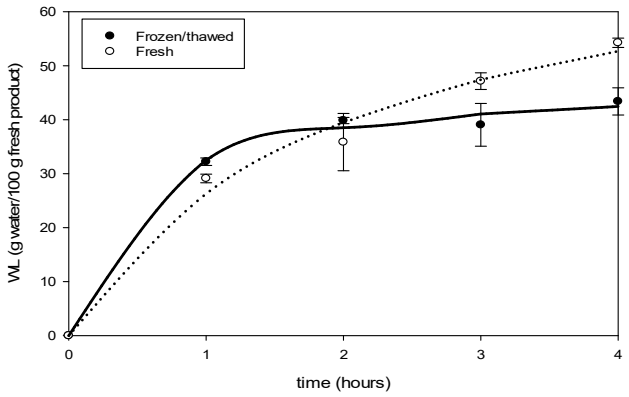
Figure 5.1 illustrates the results for water loss (a, b, c) and solids gain (d, e, f) during osmotic dehydration of fresh and frozen-thawed mangoes in AS, AS+5%I and AS+5%I+0.3%XG osmotic solutions.

5.5.2.1 Water Loss

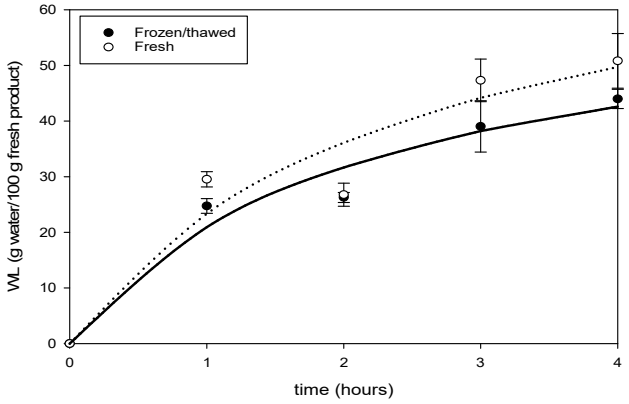
Figures 5.1 (a–c) and 5.2 (a–f) illustrate results for water loss (*WL*) during osmotic dehydration of fresh, F-T and PEF treated mangoes in AS, AS+5%I and AS+5%I+0.3%XG osmotic solutions.

WL increased with time ($p < 0.05$) during osmotic dehydration of mango. It can be observed from Figure 5.1 (a, b) that F-T pretreatment decreased *WL* during osmotic dehydration of mango samples in agave syrup solutions with and without added inulin, while slightly improving it when xanthan gum was added to agave syrup/inulin solution (Figure 5.1c). While for PEF 10 and 30 pulses, *WL* of the samples increased slightly for the three osmotic solutions. Mango osmotically dehydrated with AS+5%I+0.3%XG showed lowest *WL* for fresh, F-T and PEF samples compared to AS and AS+5%I. The different behavior reported in AS+5%I+0.3%XG is related to high viscosity of this solution due to addition of a thickening agent such as xanthan gum (Zongo et al., 2021). Increasing markedly, the solution viscosity may cause a change in the control for mass transfer during osmotic dehydration, from internal (solid matrix) to external (solution). Thus, a pretreatment such as F-T impacting on the cellular matrix may have less effect on *WL*. Similar results of F-T on *WL* were found for apple (Lazarides & Mavroudis, 1995), strawberries (Taiwo et al., 2003), African star apples (Falade & Adelakun, 2007) and pomegranate seeds (Bchir et al., 2012).

AS (a)



AS+5%I (b)



AS+5%I+0.3%XG (c)

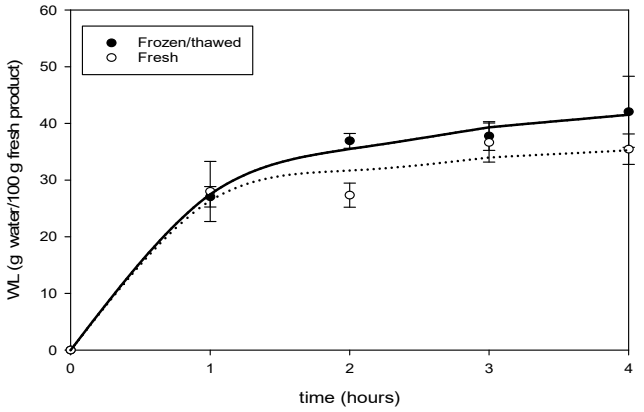


Figure 5.1: Water loss of Fresh and F-T mango during osmotic dehydration in AS (a), AS+5%I (b) and AS+5%I+0.3%XG (c) solution.

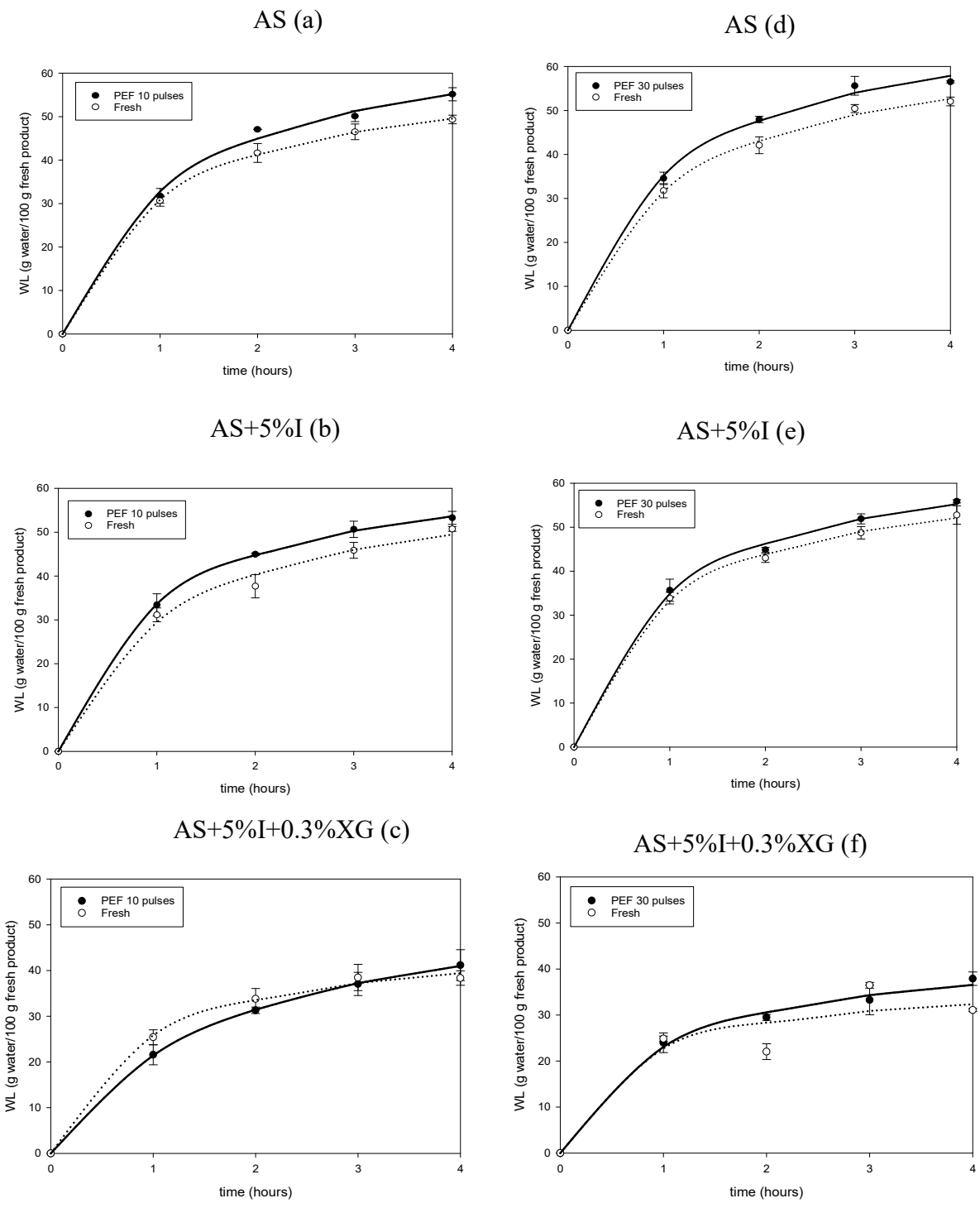


Figure 5.2: Water loss of Fresh and PEF mango during osmotic dehydration in AS (a,d), AS+5%I (b,e) and AS+5%I+0.3%XG (c,f) solutions.

Considering the effect of PEF pulses number on mango *WL,* increasing the number of pulses from 10 to 30 pulses had a slight positive effect only for AS+5%I ($p > 0.05$). PEF could improve cell permeabilization by creating pores on the tissue surface through electro plasmolysis (Donsı` et al., 2010). These new pores could be used as supplementary pathways for water transport out of the tissue during dehydration, particularly in osmotic dehydration. Prior studies have already reported similar results on *WL* increment after PEF treatment of mango and other fruits. A previous study on osmotic dehydration of mango in a sucrose 50 °Brix solution after PEF treatment was conducted (Tedjo et al., 2002), where they reported a slight increase in *WL* after PEF pretreatment compared to untreated mango. Similar *WL* increase was reported by Dermesonlouoglou et al. (2016) for kiwifruit, Rastogi et al. (1999) for carrots, and Amami et al. (2005) for apples. Contrary to expectations, no significant difference was found between 10 and 30 pulses treatment on mango *WL* with AS and AS+5%I+0.3%XG osmotic solutions, although the energy applied to mango samples increased with number of pulses (4 and 13.5 kJ/kg for 10 and 30 pulses, respectively). In the literature, there are no consistent reports on the correlation between PEF pulse number and *WL* increase during osmotic dehydration. For instance, authors in Wiktor et al. (2014) found that *WL* of osmotically dehydrated apples in sucrose 60 °Brix solution increased as PEF pulses increased from 10 to 50 at 5 and 10 kV/cm field strength. Similarly, Ade-Omowaye et al. (2002) succeeded in improving *WL* of PEF treated bell peppers. On the other hand, pulse number increment presented no correlation in improving *WL* in apples (Taiwo et al., 2003), indicating that PEF effect may depend on the fruit type. Due to their different impact on the tissue, PEF treatment showed positive impact on *WL,* unlike F-T which led to a decrease.

5.5.2.2 Solids Gain

Figures 5.3 (a–c) and 5.4 (a–f) illustrate solids gain evolution in F-T and PEF mango and their respective controls (fresh).

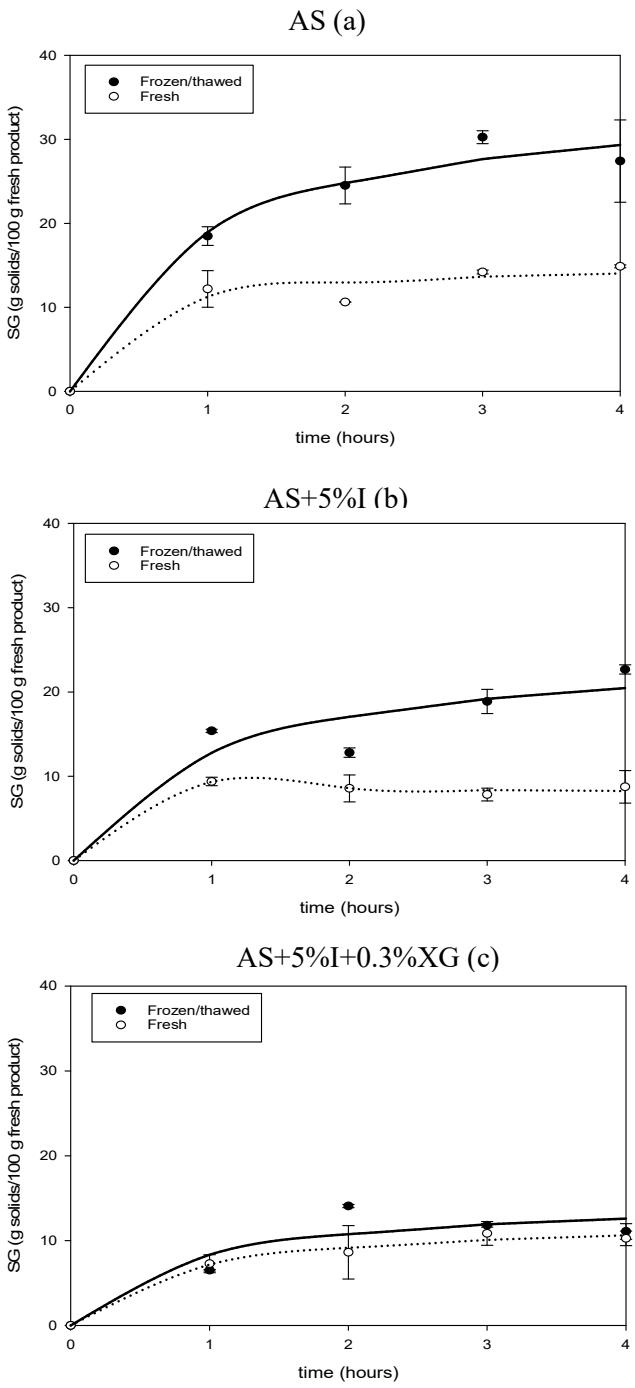


Figure 5.3: Solids gain of Fresh and F-T mango during osmotic dehydration in AS (a), AS+5%I (b) and AS+5%I+0.3%XG (c) solutions.

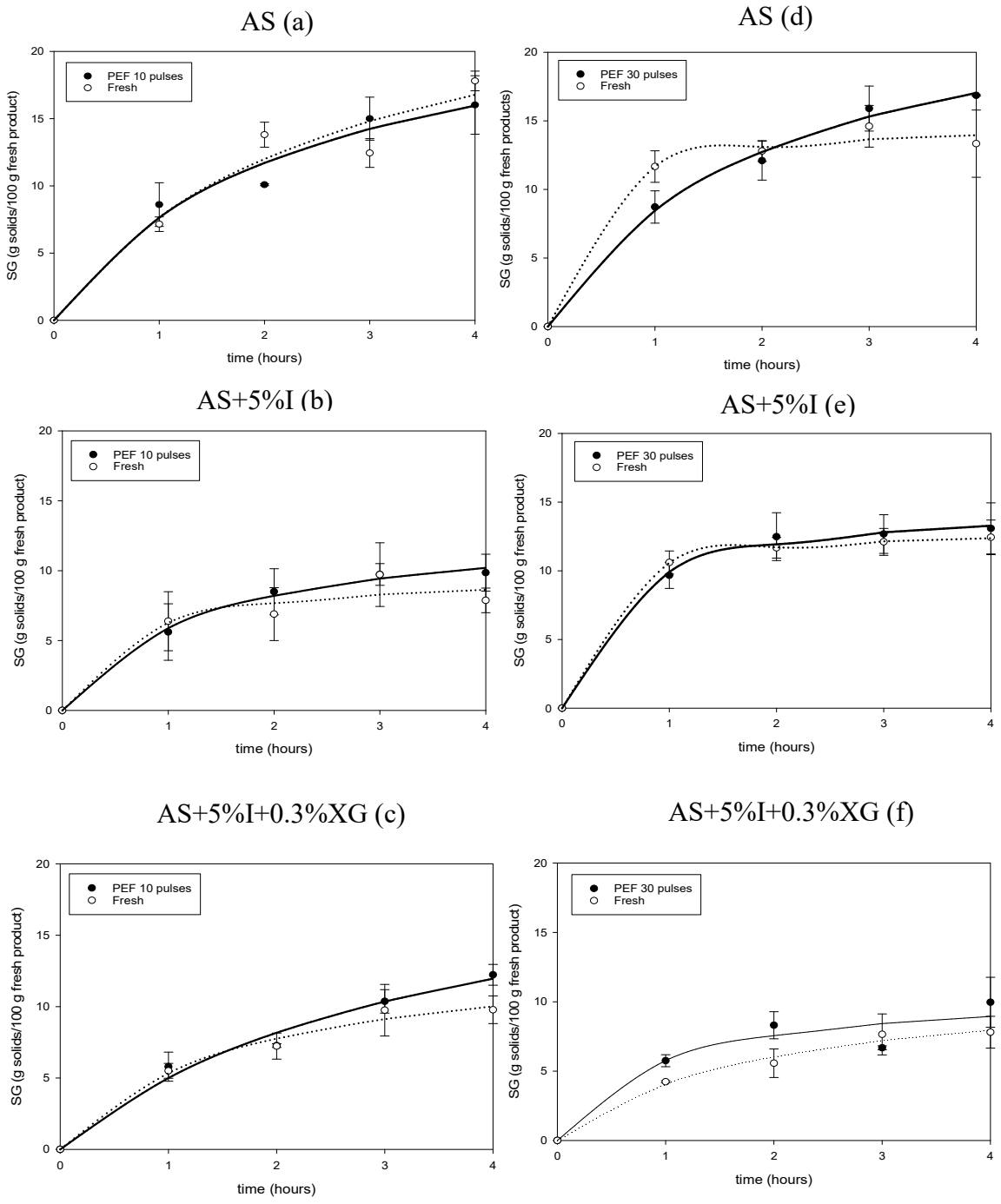


Figure 5.4: Solids gain of Fresh and PEF mango during osmotic dehydration in AS (a,d), AS+5%I (b,e) and AS +5%I+0.3%XG (c,f) solutions.

Solids gain (*SG*) increased with time and reached the highest values at 4 h. For F-T mango, a two-fold increase in solids was observed compared to fresh in AS and AS+5%I solutions, except for AS+5%I+0.3%XG solution, for which the increase was just 1.09%. Final values of solids were 14.88%, 8.74%, 10.84% for fresh mango and 27.42%, 22.68%, 11.08% for F-T ones in AS, AS+ 5%I and AS+5%I+ 0.3%XG osmotic solutions, respectively (Figure 3a–c). As can be seen, solids gain remains similar for both fresh and F-T mango in AS+5%I+0.3%XG. Regarding PEF pretreatment (Figure 5.4 a–f), compared to fresh mango, PEF slightly increased *SG* at 10 pulses for AS+5%I and AS+5%I+0.3%XG, while it increased *SG* for all three osmotic solutions after PEF 30 pulses pretreatment. However, increasing pulses number from 10 to 30 did not show a significant effect on *SG* improvement. In addition, there was a variable effect of pulse numbers according to the osmotic solution.

In comparison to fresh and PEF mango, F-T pretreatment prompted higher solids gain after osmotic dehydration. This could be related to the type of damage induced on the tissue. F-T can result in both physical (cell rupture by ice crystal growth) and chemical damages (biochemical reactions after cell fracture of mango tissue), destroying the cellular compartments through pectin hydrolysis, leading to cell separation and rupture (Delgado & Rubiolo, 2005; Khuwijitjaru et al., 2022; Li et al., 2018; Reeve, 1970). After thawing, melted ice crystals left spaces or voids that can be used by sugar molecules to enter easily into the tissue (Ando et al., 2016), and thus a greater flux of solids enter the matrix, as shown for *SG* in Figure 5.3 (a–c). During osmotic dehydration, *WL* is a simultaneous countercurrent flow to *SG* and has been pointed out to be controlled by diffusion (Lazarides & Mavroudis, 1995). Thus, greater solids flux entering the matrix due to F-T pretreatment may accumulate near the surface and act as a barrier for *WL*. In addition, cell walls collapse, and the deformation of cellular network (shown later through microstructure results) may increase the tortuosity of the tissue which is inversely proportional to water diffusion coefficients. In the case of PEF, electroporation is the phenomena responsible for tissue microstructure modification through increment of porosity (Arevalo et al., 2004), which may explain the observed slight increase in solids uptake. The new pores induced in the mango tissue may have different sizes as to the original ones which could favor selectively water molecules as was observed for *WL* increment in section 5.5.1. Previous studies have shown that, after PEF treatment of apples, pore measurement indicated that PEF generated pores had smaller mean sizes than untreated

ones (Bazhal et al., 2003). In agreement with the present results, other authors also showed a slight increase of *SG* with PEF pretreatment in osmotically dehydrated mango (Tedjo et al., 2002), apples (Amami et al., 2005) and kiwifruit (Dermesonlouoglou et al., 2016). On the other hand, some studies found no change (Wiktor et al., 2014) or lower solids uptake (Traffano-Schiffo et al., 2016) compared to fresh samples. The less destructive effect of PEF on the mango sample could explain the minor increase of *SG* compared to F-T.

As can be seen from Figures 5.3 (b,c) and 5.4 (b,c,e,f) addition of polysaccharide inulin or xanthan gum lowered sugar gain of mango in both F-T and PEF samples compared to pure AS solution. This could be explained, respectively, by the high molecular weight of inulin, and by the increase of solution viscosity for xanthan gum (Zongo et al., 2021). Inulin also reduced solids gain due to formation of a layer on the surface which creates an external resistance to solids uptake. Results on use of natural syrup to reduce sugar uptake are scarce in the literature, as most studies focused on the organoleptic and nutrients properties of the final product as in the case of sugar beet molasses (Filipović et al., 2022), maple syrup (Rupasinghe et al., 2010) or honey (Chauhan et al., 2011). However, some authors have shown similar effect of high viscosity and polysaccharide content with corn syrup solids solution for osmotic dehydration of papaya (El-Aouar et al., 2006) and mango (Zongo et al., 2021).

5.5.3 Effects of F-T and PEF pretreatments on equilibrium values during osmotic dehydration

Tables 5.2 and 5.3 show equilibrium *WL* and *SG* values (*WLeq* and *SGeq*) for F-T and PEF mango with their respective controls (fresh). Additionally, osmotic dehydration efficiency (*ODE)* is presented. Due to difficulty in controlling mango maturity in this chapter, results were compared for F-T and PEF with controls used the same day as the experiment

Table 5.2 : Equilibrium and efficiency ratio of fresh and frozen-thawed mango during osmotic dehydration

Pre-treatment	***WLeq*** **(g Water/100 g Fresh Product)**			***SGeq*** **(g Solids/100 g Fresh Product)**			***ODE*** **(g Water Lost/g Solids Gained)**		
	AS	AS+5%I	AS+5%I +0.3% XG	AS	AS+5%I	AS+5%I +0.3% XG	AS	AS+5%I	AS+5%I +0.3% XG
Fresh	*50.70 ±0.35[a]	49.08 ±3.47[a]	35.67 ±0.37[a]	14.61 ±0.63[b]	9.92 ±2.77[b]	10.55 ±1.13[a]	3.46 ±0.05[a]	4.57 ±0.63[a]	3.42 ±0.33[a]
Frozen-Thawed	41.23 ±3.23[b]	42.30 ±1.00[b]	41.07 ±0.71[a]	29.50 ±3.42[a]	20.63 ±2.40[a]	11.57 ±0.52[a]	1.51 ±0.10[b]	2.13 ±0.31[b]	3.61 ±0.11[a]

* Within the same category of variables (*WLeq*, *SGeq* or *ODE)*, mean values in same column with different letters are significantly different ($p < 0.05$).

In terms of *WL*, Table 5.2 shows that *WLeq* is higher for fresh mango than for F-T mango, except for AS+5%I+0.3%XG solution in agreement with *WL* maximum values shown in Figure 5.1 (a–c). Higher *SGeq* (Table 5.2) in mangoes was obtained after F-T treatment as found previously. The *ODE* values showed that fresh mango had better efficiency than F-T pretreated mangoes and lower values of *SGeq*. In addition, F-T mangoes dehydrated in AS+5%I+0.3%XG solution presented an interesting *ODE* (3.61 ± 0.11) with a reasonable solids' intake (11.57 ± 0.52 g solids/100 g fresh product), similar to fresh mango behavior.

Table 5.3: Equilibrium and efficiency ratio of frsh and PEF treated mango during osmotic dehydration

Pre-treatment	***WLeq*** **(g Water/100 g Fresh Product)**			***SGeq*** **(g Solids/100 g Fresh Product)**			***ODE*** **(g Water Lost/g Solids Gained)**		
	Osmotic Solutions			**Osmotic Solutions**			**Osmotic Solutions**		
	AS	AS+5%I	AS+5%I +0.3% XG	AS	AS+5%I	AS+5%I +0.3% XG	AS	AS+5%I	AS+5%I +0.3% XG
Fresh10	47.93 ± 1.00[a]	48.31 ± 1.16[a]	38.42 ± 0.64[a]	14.04 ± 1.95[a]	8.80 ± 1.56[a]	9.76 ± 0.44[a]	3.49 ± 0.59[a]	5.68 ± 1.05[a]	3.92 ± 0.14[a]
PEF/10 pulses	52.63 ± 1.38[a]	51.97 ± 1.61[a]	42.00 ± 4.82[a]	15.51 ± 1.89[a]	9.79 ± 0.84[a]	11.30 ± 0.72[a]	3.44 ± 0.39[a]	5.33 ± 0.31[a]	3.63 ± 0.26[a]
Fresh30	50.36 ± 1.61[A]	50.76 ± 1.18[A]	32.57 ± 4.21[A]	13.97 ± 1.96[B]	12.27 ± 0.72[A]	8.25 ± 1.11[A]	3.67 ± 0.52[A]	4.15 ± 0.27[A]	3.95 ± 1.05[A]
PEF/30 pulses	55.09 ± 1.12[A]	53.90 ± 0.71[A]	36.07 ± 1.68[A]	17.05 ± 1.23[A]	12.89 ± 1.64[A]	8.61 ± 0.78[A]	3.37 ± 0.30[A]	4.25 ± 0.53[A]	4.15 ± 0.20[A]

Fresh10 and Fresh30 are the controls (untreated mango) for PEF10 and PEF30 respectively.

*Within the same category of variables (*WLeq*, *SGeq* or *ODE)*, mean values in same column with different letters are significantly different ($p < 0.05$). Lower case letters (a, b) compared fresh10 and PEF/10 pulses samples. Uppercase letters (A, B) compared fresh30 and PEF/30 pulses samples.

PEF pretreatment increased slightly *WLeq* and *SGeq* at 10 pulses and 30 pulses compared to fresh mango (Table 5.3). In general, PEF did not show a significant improvement of *ODE* values (about 3 to 5 for fresh and PEF treated samples). Compared to similar *ODE* information presented in Table 5.2 for F-T pretreatment (about 1 to 4), PEF pretreatment presented a higher *ODE* value which remained closer to fresh mango. These results may indicate that it is not necessary to pretreat mango with PEF due to the small impact on mass transfer

5.5.4 Effects of F-T and PEF pretreatments on mango tissue

5.5.4.1 Effect of F-T on mango firmness and electrical conductivity during osmotic dehydration

Table 5.4 shows firmness (N) and electrical conductivity (S/m) of fresh and F-T mango, together with the respective ratio of fresh and F-T values. Values of fresh mango firmness were discussed in 5.5.2.1. As for electrical conductivity, a low value recorded for untreated mango (0.004 S/m ± 0.001) is often reported for food if it is not pretreated in brine solution (Olivera et al., 2013). For instance, for fresh potato cylinder of 30 mm diameter, electrical conductivity was found to be less than 0.1 S/m (Olivera et al., 2013) and a similar range was measured for carrot particles (6 mm–13 mm) (Zareifard et al., 2003). This only increased after pretreatment, demonstrating an effect on the tissue properties.

The firmness ratio of fresh and F-T mango (4.0 ± 2.26) indicated a four-fold decrease of firmness after the F-T treatment. It is well known that firmness of fruit tissue is reduced through F-T due to cell wall breakdown and collapse (Charoenrein & Owcharoen, 2016). As for the electrical conductivity ratio, it shows a value of 0.12 ± 0.02, indicating that F-T samples are about eight times more conductive than fresh mango. Electrical conductivity measures the ability to conduct a current, and in a food matrix it increases with the electrolytes' leakage (ions, soluble solids) through the open pores generated during the thawing process, contributing to higher electrical conductivity values. These results strongly support softening theories for mango tissue because of F-T.

Table 5.4: Firmness and electrical conductivity ratio of fresh and F-T mango

	Firmness	Electrical Conductivity
Fresh	49.21 N ± 11.36	0.004 S/m ± 0.00
F-T	12.40 N ± 5.00	0.14 S/m ± 0.01
Ratio (Fresh/F-T)	4.00 ± 2.26	0.12 ± 0.02

5.5.4.2 Effect of F-T and PEF on mango disintegration index

Figure 5.5 provides results of disintegration index *Z* obtained for mango after PEF treatment of 10 pulses and 30 pulses at 1 kV/cm and 1 kHz. The maximum *Z* value (1) corresponding to F-T samples has been added to Figure 5.5 as a reference. In this Figure, the electrical conductivity *Z* method results are represented in light grey and firmness *Z* results, in dark grey. As can be seen from Figure 5.5, both methods show that disintegration index *Z* increases with PEF pulse number. The minimum *Z* values were 0.16 and 0.36 while the maximum were 0.42 and 0.56, respectively, for 10 and 30 pulses. This agrees with other studies on the effect of number of pulses on tissue disintegration index. For instance, Tedjo et al. (2002) showed an increase of *Z* disintegration values of mango from 0.18 to 0.58 with the increase in number of pulses from 10 to 100. Similarly, Dermesonlouoglou et al. (2018) reported an enhancement of disintegration index *Z* in Goji Berry (up to 0.38) with increasing the number of pulses (750, 1500, 7500) at 2.8 kV/cm. The tendency of *Z* values observed by both electrical conductivity and firmness methods is similar, though the firmness method gave higher values of *Z* than with the electrical conductivity method. As non-destructive methods are becoming a trend in evaluating food material properties in the food industry, the electrical method is promising because it keeps the integrity of the product, does not necessitate high-cost material and is simple to use than the texturometer for firmness evaluation. However, this lab-made tool that has been developed for the mango electrical conductivity analysis in this study is still in the experimental stage and further research is needed to improve the technology for more accuracy.

The modification of the disintegration index observed through the results of the present study indicated that PEF treatment improved permeabilization of mango tissue. This confirms that formation of pores happened after PEF pretreatment facilitating water loss and solids gain (Figures 5.3 and 5.4). The totally destroyed cellular structure caused by F-T (Z = 1),

drastically reduces internal barrier for mass transfer, allowing more solids uptake due to loss in selective permeability. PEF treated sample, on the other hand, induces minor modifications of cellular tissue creating new paths for loss of small molecules of water along with solids uptake.

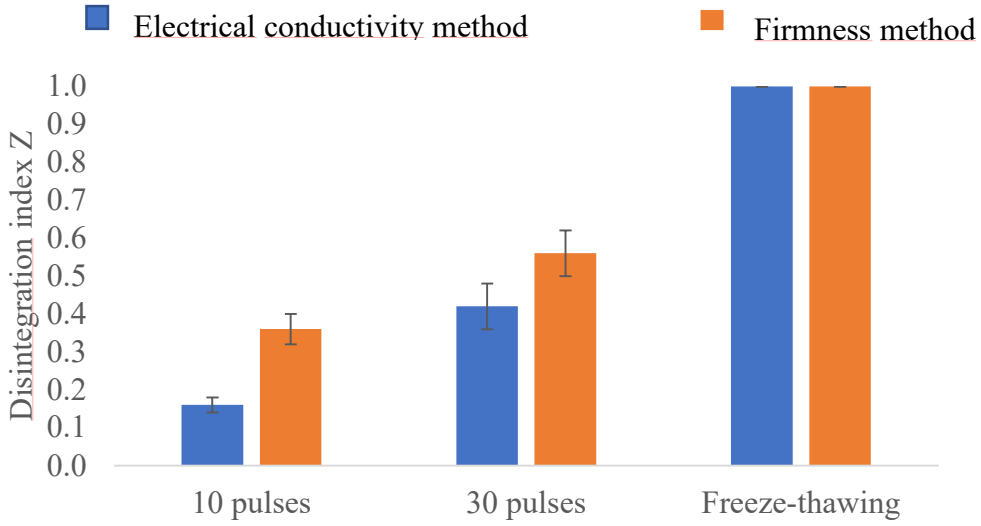


Figure 5.5: Disintegration index Z with electrical conductivity and Firmness methods.

5.5.4.3 Effects of F-T and PEF pretreatments on mango microstructure during osmotic dehydration

Figure 5.6 illustrates surface microstructure of fresh (Figure 5.6a), F-T (Figure 5.6b) and PEF treated mango (Figure 5.6 c,d for PEF10 and PEF30, respectively) before osmotic dehydration.

Figure 5.6a shows in fresh mango a dense distribution of oval cells, each surrounded by a cellular membrane. A thin space marking the separation between different intact cells can also be observed. After F-T pretreatment (Figure 5.6b), larger spaces and structural collapse are perceived throughout. Additionally, multiple puncture holes all over the tissue are noticeable. According to Bomben & King (1982), these holes are attributed to spaces

previously occupied by ice before the thawing step. The observed damage to cell membrane disabled its capacity to act as a semipermeable barrier during movement of solutes (Delgado & Rubiolo, 2005) and, thus, it can explain the increase in sugar uptake of F-T mango compared to fresh (Figure 5.3 a–c) and PEF (Figure 5.4 a–f). The increased tortuosity caused by cell wall collapse may explain the difficulty for water in using the diffusion paths, resulting in lower *WL* (Figure 5.1 a, b). On the contrary, cellular structure of mango treated with 10 and 30 pulses (Figure 5.6 c,d) showed oval and round cells which still had intact cell wall and cell membrane as in the fresh mangoes (Figure 5.6a). These images indicate that electroporation phenomenon preserves cell structure better than F-T treatment (Figure 5.6b). As PEF treatment increased (from 10 pulses to 30 pulses), cells appear to be closer to each other with more porosity. This may be a consequence of water leakage during electro plasmolysis, leading to pores formation which is beneficial for water removal out of the cells, leading to cell shrinkage. Then, cells separate from each other with formation of small voids.

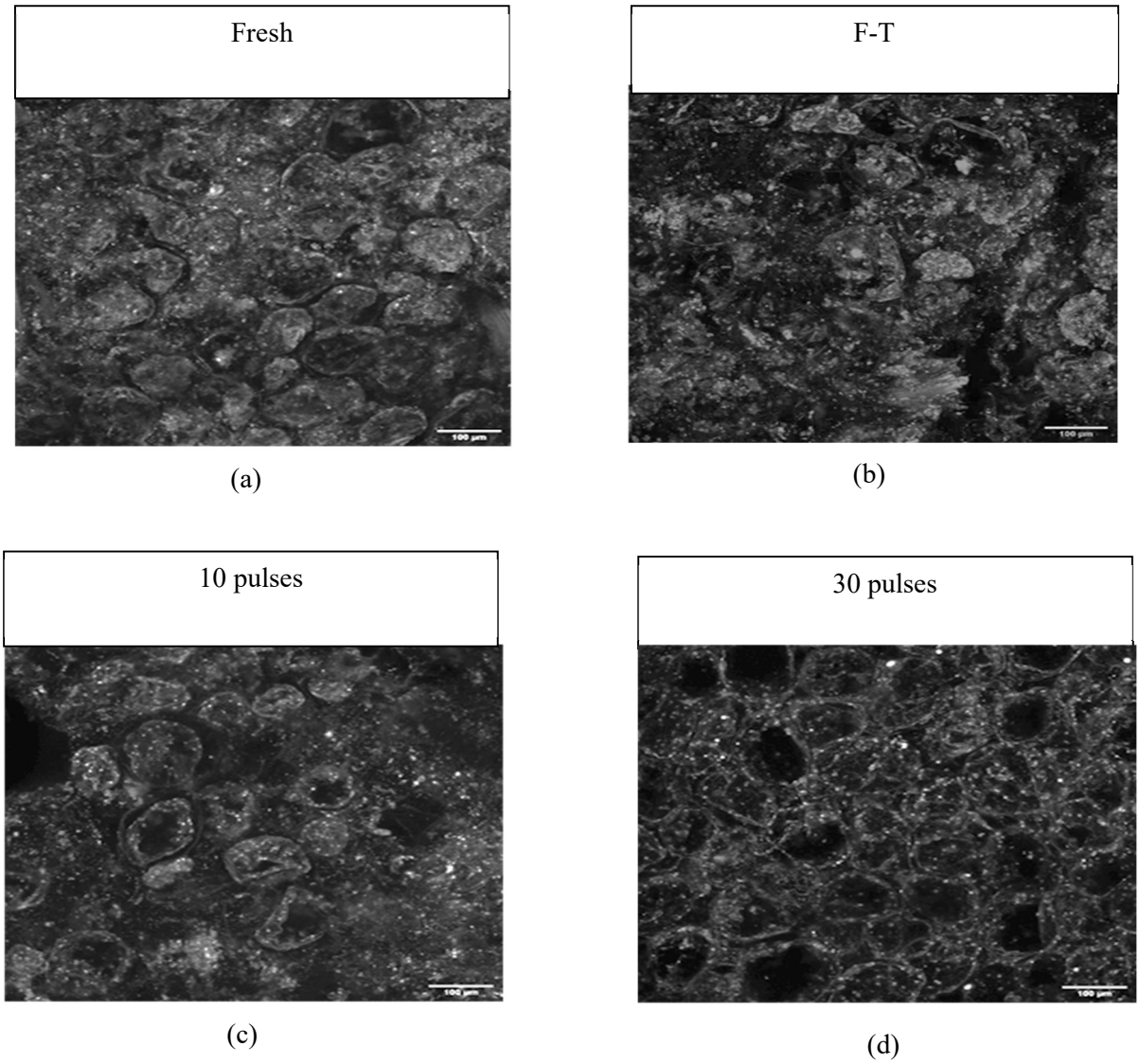


Figure 5.6: Microstructures of non-osmotic treated mango: fresh mango (a); after F-T (b); after 10 pulses (c) after 30 pulses (d). The white scale bar represents 100 μm.

5.5.5. HPLC sugar profiles

5.5.5.1 Individual sugar profiles of fresh, frozen-thawed and PEF treated mango during osmotic dehydration

Tables 5.5 and 5.6 show evolution of sucrose, fructose, glucose, and inulin in the mango during osmotic dehydration in different solutions for frozen-thawed and PEF pretreatment respectively. Firstly, the main observation about sucrose is its negative values in fresh, frozen-thawed and PEF treated samples, indicating a loss sugar during osmotic dehydration. Fructose was the major solute gained, followed to a less extent by glucose. Individual sugar profiles were previously shown in chapter 4 to be dependent on the composition of mango and the osmotic solutions facilitating uptake of solute which is present in the osmotic solution while leading to the leakage of other mango own sugars which are absent or negligeable in the osmotic solution. Such was the case for fructose. However, few values in Table 5.5 (-0.30 for glucose or 1.19 for sucrose) and table 5.6 (-0.91, -0.37, -0.29) were of different tendency as expected and could be related to the sensitivity of the RID detector and possible errors during HPLC analysis and the limited repetitions made, due to cost and time constraints. Difficulty in accurately controlling the mango maturity which directly affects the fruit firmness and ripening state also could be a reason leading to only approximate homogeneity in the fruits batch. In addition, PEF treatment may have not been homogeneous during contact with mango tissue, probably due to the large width of the treatment cell which could reduce its efficiency. These could be possible reasons for the unexpected values. Therefore, only individual sugar evolution is commented in this part and not the total sugar gain. Nevertheless, results indicated the same tendency shown in chapter 4 for individual sugars in mango. Inulin was found in the mango and its content shows little variation from the different osmotic solutions, probably due to its large molecules which restrain its uptake into the mango. Regarding the pretreatment's effects, fructose, glucose and inulin showed increasing tendency for frozen-thawed mango compared to fresh ones. While few changes were observed for PEF treated samples.

Table 5.5: Individual sugar gain or loss (g/100 g of fresh mango) for fresh and frozen-thawed mango during osmotic dehydration

	Osmotic solutions	Sucrose	Fructose	Glucose	Inulin
Fresh	AS	-0.53 ±1.77	6.11 ±4.58	3.83 ±0.48	0
FT	**AS**	**-2.52 ±0.52**	**16.04 ±4.58**	**3.46 ±1.23**	0
Fresh	AS+5%I	-0.53 ±0.90	8.54 ±2.00	1.46 ±0.41	0.16 ±0.00
FT	**AS+5%I**	**-0.93 ±0.20**	**19.37 ±1.96**	**4.54 ±0.36**	0.35 ±0.07
Fresh	AS+5%I+0.3%XG	1.19 ±0.16	9.76 ±0.04	-0.30 ±0.02	0.69 ±0.01
FT	**AS+5%I+0.3%XG**	**-2.05 ±0.33**	**17.72 ±1.83**	**1.92 ±0.52**	1.09 ±0.16

Table 5.6: Individual sugar gain or loss (g/100 g of fresh mango) for fresh and PEF treated mango during osmotic dehydration

	Osmotic solutions	Sucrose	Fructose	Glucose	Inulin
Fresh	**AS**	**-1.99 ±0.79**	**7.10 ±2.02**	**0.77 ±0.80**	**0**
PEF10		-3.82 ±0.22	8.25 ±1.62	-0.91±0.31	0
PEF30		-2.32 ±0.85	14.83±2.24	3.02 ±0.60	0
Fresh	**AS+5%I**	**-2.10 ±0.86**	**5.52 ±0.91**	**1.15 ±0.32**	**0.40 ±0.02**
PEF10		-2.74 ±0.55	5.93 ±0.10	1.25 ±0.04	0.41±0.02
PEF30		-1.76±1.00	8.24 ±3.65	1.81 ±1.04	0.43 ±0.17
Fresh	**AS+5%I+0.3%XG**	**-2.76 ±0.72**	**9.31 ±0.46**	**-0.37 ±0.35**	**0.51 ±0.26**
PEF10		-2.09 ±0.68	9.70±0.54	-0.29 ±0.09	0.42 ±0.02
PEF30		-3.04 ±0.90	9.58 ±0.29	1.05 ±0.42	0.50 ±0.05

5.5.5.2 Final individual sugar content of fresh, frozen-thawed and PEF treated mango during osmotic dehydration

Final sugar content in the osmotic dehydrated mango is reported in Tables 5.7 and 5.8 and expressed as g/100 g of OD mango. In frozen-thawed mango subjected to osmotic dehydration, each sugar increased compared to the fresh mango. But while the increment was minimal for sucrose, glucose and inulin (in solution containing inulin), fructose content was almost 50% higher in frozen-thawed mango than the fresh mango. This considerable change

in fructose amount is due to the fact it is the major sugar of the solutions and would have the highest chemical potential gradient for diffusion towards the mango tissue.

Table 5.7: Final individual sugar content (g/100 g of OD mango) for fresh and frozen-thawed treated mango during osmotic dehydration

	Osmotic solutions	**Sucrose**	**Fructose**	**Glucose**	**Inulin**
Fresh	AS	7.23 ±1.31	15.95 ±1.48	5.03 ±0.80	0
FT	**AS**	**4.52 ±0.10**	**21.09 ±1.92**	**5.37 ±0.56**	**0**
Fresh	AS+5%I	8.53 ±1.73	16.98 ±0.73	4.98 ±0.15	0.76 ±0.11
FT	**AS+5%I**	**7.65 ±0.16**	**30.72 ±3.09**	**8.14 ±0.61**	**1.74 ±0.18**
Fresh	AS+5%I+0.3%XG	5.62 ±1.38	15.08 ±1.39	4.45 ±0.59	0.65 ±0.17
FT	**AS+5%I+0.3%XG**	**6.65 ±0.34**	**27.90 ±2.08**	**7.84 ±0.59**	**1.09 ±0.16**

Table 5.8: Final individual sugar content (g/100 g of OD mango) for fresh and PEF treated mango during osmotic dehydration

	Osmotic solutions	**Sucrose**	**Fructose**	**Glucose**	**Inulin**
Fresh	**AS**	7.23 ±1.31	15.95 ±1.48	5.03 ±0.80	**0**
PEF10		**4.38 ±0.03**	**15.12 ±1.44**	**4.00 ±0.19**	0
PEF30		**8.15 ±1.77**	**34.35 ±3.51**	**1.81 ±1.04**	0
Fresh	**AS+5%I**	8.53 ±1.73	16.98 ±0.73	4.98 ±0.15	0.76 ±0.11
PEF10		**6.98 ±0.83**	**16.93 ±0.58**	**4.99 ±0.19**	**0.74 ±0.02**
PEF30		**7.79 ±0.76**	**18.53 ±3.85**	**5.27 ±1.10**	**0.68 ±0.20**
Fresh	**AS+5%I+0.3%XG**	5.62 ±1.38	15.08 ±1.39	4.45 ±0.59	0.65 ±0.17
PEF10		**6.50 ±0.68**	**16.16 ±0.04**	**4.62 ±0.35**	**0.61 ±0.03**
PEF30		**3.81 ±0.01**	**16.14 ±1.32**	**4.58 ±0.42**	**0.77 ±0.03**

5.6. Conclusions

This present study focused on reducing sugar uptake in mango by applying pretreatments of F-T or PEF before osmotic dehydration. F-T pretreatment was not effective in reducing sugar uptake and did not increase *WL*. However, addition of xanthan gum to agave syrup solution helped to lower sugar uptake of the final product. Therefore, in the case of fresh mango

shortage, industry can use agave syrup solution with xanthan gum to produce low calorie mango. PEF pretreatment, slightly increased *WL,* and *SG*. Microscopic images have shown that F-T creates more damage on the tissue than PEF. The smaller pores induced by PEF pretreatment may be the reason for enhanced water removal compared to F-T. However, due to the short availability of fresh mango, F-T can often be necessary during off-season. In such case, using thickening agents such as xanthan gum to enrich the osmotic solution is a good alternative to lower sugar uptake during osmotic dehydration of mango.

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Conclusion générale

Cette étude avait pour but d'investiguer différentes méthodes pour optimiser le processus de déshydratation osmotique des mangues dans un sirop d'agave afin de réduire le gain en sucres ajoutés et d'améliorer la valeur nutritionnelle de la mangue. Dans la littérature, plusieurs auteurs se sont intéressés à réduire le gain en sucres lors de la déshydratation osmotique ainsi qu'à la recherche de solutions osmotiques alternatives pour remplacer les solutés les plus utilisés tel que le sucrose. Bien qu'il existe des études avec le miel et le sirop d'érable, le sirop d'agave est peu étudié à notre connaissance et il n'a pas été utilisé dans la déshydratation osmotique de la mangue comme il a été le cas dans cette présente thèse. Cette étude avait comme hypothèse que la composition de la solution osmotique ainsi que la combinaison de technologies novatrices pouvaient permettre une déshydratation osmotique sélective de la mangue avec pour but de favoriser la déshydratation (perte en eau) et réduire l'entrée en sucres dans la mangue. L'hypothèse a été confirmée en partie. En effet, la première partie de résultats de la thèse a permis de démontrer qu'en utilisant des polysaccharides tels que la gomme xanthane et l'inuline dans la composition de la solution osmotique, il a été possible de réduire le gain en sucres de la mangue. Cela était dû d'une part à l'effet épaississant de la gomme xanthane qui a augmenté la résistance externe au mouvement des solutés, ce qui a eu pour conséquence de diminuer l'entrée en sucres. L'impact de l'épaisseur de la mangue a été étudié ce qui a permis de montrer que de grandes épaisseurs sont favorables à la réduction du gain en sucres. Aussi l'effet de la viscosité de la solution osmotique est plus important sur les petites épaisseurs que les grandes épaisseurs à cause du changement de contrôle pour le transfert de matière.

La deuxième partie de résultats (Chapitre 4) a fait suite au chapitre précédent en étudiant par analyses chromatographiques la modulation du profil de sucres obtenu après la déshydratation osmotique des mangues dans les différentes solutions osmotiques utilisées. Il a été possible de montrer que selon la composition de la solution osmotique, le profil en sucres de la mangue a été modulé. En utilisant des solutions osmotiques à un seul soluté, la mangue s'est enrichie en ce soluté tout en s'appauvrissant en ces autres solutés natifs. Il était donc possible par exemple de choisir une solution osmotique qui contenait peu ou pas de sucrose par rapport à la mangue fraîche, de façon à réduire la teneur en sucrose de la mangue,

ce qui peut être bénéfique pour les personnes qui ont des restrictions de sucrose dans leur régime alimentaire. C'était le cas avec les solutions à un soluté composé de fructose ou de glucose, et la solution de sirop d'agave dans lesquelles la perte de sucrose de la mangue a été rapportée lors de la déshydratation osmotique. L'étude chromatographique a permis également de mettre en évidence la présence d'inuline dans la mangue en utilisant les solutions osmotiques enrichies en inuline. Ce qui s'avère un résultat important car les propriétés prébiotiques de ce composé pour le microbiote intestinal ont été largement démontrées dans la littérature permettant d'apporter un nouveau produit sur le marché croissant des produits transformés bioactifs. D'autre part, les images de microscopie électronique à balayage ont permis de visualiser le mode de dépôt des carbohydrates et particulièrement pour le sucrose, elle se dépose en formant une couche sur la surface de la mangue. Ce qui a constitué une excellente barrière pour l'entrée en solides dans la mangue. Le glucose a montré des couches rugueuses sur la mangue contrairement à celle du fructose qui était lisse. Ceci a affecté le gain en sucres selon les épaisseurs de la mangue. Pour les petites épaisseurs (0.4cm), où le contrôle de la diffusion était externe, le glucose avait un gain plus élevé dans la mangue comparé au fructose (solutions à un seul soluté). Alors que pour les grandes épaisseurs (1.5 cm), où le contrôle était interne, le gain en glucose était similaire au gain en fructose.

Le dernier chapitre de résultats de la thèse a consisté à l'application des prétraitements de congélation/décongélation et de champ électrique pulsé afin de moduler la structure microscopique du tissu de la mangue pour moduler le transfert de matières en faveur de la perte en eau. Bien que l'indice de désintégration ait montré un impact sévère de la congélation/décongélation sur le tissu, la perte en eau était négativement influencée par ce prétraitement qui a aussi augmenté le gain en sucres. Cependant, la solution osmotique contenant la gomme xanthane a permis d'obtenir un gain en sucres proche de celle de la mangue fraîche confirmant l'impact de la viscosité élevée sur le gain en sucres. L'ajout de la gomme xanthane comme un ingrédient épaississant à la solution osmotique est un résultat qui peut être avantageux pour l'industrie agroalimentaire ou les petites unités de transformation de la mangue. La mangue peut être congelée et utilisée pendant les périodes où elle n'est pas disponible. Et l'utilisation d'ingrédient épaississant pourrait permettre d'obtenir un rendement de perte en eau par rapport au gain en sucres similaire à celui de la

mangue fraîche. Les conditions utilisées pour le prétraitement de champ électrique pulsé dans cette thèse n'ont pas permis de montrer un impact significatif sur l'amélioration de la perte en eau de la mangue et l'effet était négligeable sur le gain en sucres.

Perspectives pour les travaux futurs

La demande en produits transformés à faible teneur en calories et ayant des effets bioactifs est croissante. Cette étude a permis de répondre en partie à cette demande par les résultats de sucres ajoutés réduits obtenus ainsi que l'apport en inuline. Il serait intéressant pour des travaux futurs d'utiliser le vrai sirop d'agave pour la déshydratation osmotique de la mangue, et de conduire une étude nutritionnelle d'une part pour identifier et quantifier les nutriments qui seront imprégnés dans la mangue; d'autre part une étude sensorielle afin de tester l'acceptabilité du produit par les consommateurs. Le sirop d'agave contient un composé antibactérien, le méthylglyoxal, il serait possible d'étudier la durée de préservation de mangues déshydratées dans du sirop d'agave et de suivre la durée de stockage et la qualité microbiologique. Également, une étude d'impact économique pourrait être faite pour la faisabilité de mangues transformées à base de sirop d'agave. Aussi, d'autres paramètres de champ électrique pulsé peuvent être testés par exemple l'intensité de champ électrique pulsé pourrait être augmentée (car nous avons été limités par l'équipement dans le cadre de ce projet) pour avoir un effet plus marqué sur la perte en eau. Il existe plusieurs technologies novatrices d'amélioration du transfert de matières lors de la déshydratation osmotique. D'autres techniques autres que celles utilisées dans cette thèse peuvent être employées, l'ultrason, les hautes pressions hydrostatiques par exemples. De plus, une étude du séchage complémentaire de la mangue déshydratée par osmose pourrait être réalisée afin d'obtenir des mangues qui se conservent plus longtemps. Finalement, une utilisation d'autres ingrédients bénéfiques tels que des fibres peuvent être utilisés afin d'améliorer la qualité nutritionnelle de la mangue et de proposer plusieurs gammes de produits à base de mangue ayant un profil nutritionnel varié.