MAGNESIUM ION IMPREGNATION IN POTATO TISSUE TO ENHANCE STRUCTURE INTEGRITY DURING FRYING AND REDUCE OIL ABSORPTION IN POTATO CHIPS

A Thesis

by

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ABSTRACT

Obesity is one of the most concerned health problems, especially in the United States, causing by changing of lifestyle with high consumption of convenient foods (high oil content). Thus, food companies put tremendous efforts on investigating new techniques to produce healthy convenient foods, without compromising on the quality.

In this study, pretreatments including sonication-assisted vacuum impregnation (SVI), non-sonication-assisted vacuum impregnation (NSVI), and sonicated without vacuum impregnation (S) were evaluated, to deliver divalent ions (Mg^{2+}) into raw potato slices, and thus enhancing cell structure integrity to reduce fat uptake of chips during frying. The effect of pretreatments was evaluated based on the following product quality attributes: oil content, moisture content, magnesium content, texture, color, shrinkage, densities, and porosity. The effect of using combined ions of Mg^{2+} and Ca^{2+} was also analyzed. Sensory analysis was carried to determine consumer preferences between SVI and NSVI samples. Scanning electron microscopy (SEM) was used to analyze the microstructural changes of potato samples during the processes (sonication and frying).

The combination of sonication and vacuum impregnation (SVI) showed a synergistic effect on reducing oil uptake, with the highest oil reduction values of 20.1% at 50 min treatment time and 16.6% at 30 min sonication time, in comparison to samples pretreated with NSVI and S, respectively. For potato chips treated under SVI, the lowest oil content was obtained at the highest $MgCl_2$ concentration (20000 ppm) and longest sonication time (50 min), with a value of 0.17 ± 0.01 g/g DM, compared to the control

sample of 0.39 ±0.00 g/g DM. The SVI pretreatment significantly ($p < 0.05$) affected product texture, color, shrinkage, and porosity.

There was no significant difference ($p > 0.05$) between potato chips treated with only $MgCl_2$ and with a combination of $MgCl_2$ and $CaCl_2$ on oil content, color parameter *a** (greenness-redness), shrinkage, bulk density, solid density, and porosity. They were significantly different ($p < 0.05$) in term of texture (hardness) as the addition of Ca^{2+} improved the sample's crispness. The concentration of $MgCl_2$ affected significantly ($p < 0.05$) the mass change, water loss, and magnesium uptake. The changes in magnesium content (ΔMg) was higher for the higer concentration of $MgCl_2$ for both treatments. SVI treated samples absorbed 29% more magnesium than the NSVI samples for the 15000 ppm solution concentration. For the 50000 ppm solution, the SVI samples absorbed 15% more than the NSVI ones.

Sensory evaluation results indicated that pretreated potato chips (SVI – with 15000 ppm $MgCl_2$) are acceptable to consumers. Significant difference ($p < 0.05$) was only observed in the sensory attribute "texture". Potato chips treated with combined $MgCl_2$ and $CaCl_2$ received a significant ($p<0.05$) higher score than the other two treatments. Microscopic analysis of SEM images showed a well-intact cellular structure and thicker middle lamella after SVI treatment compared to the control samples.

The application of sonication-assisted vacuum impregnation is effective for reducing oil uptake and improving overall quality of deep-fat fried potato chips, by means of delivering divalent ions into raw potato tissues, stabilizing the cellular structure.

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CHAPTER I

INTRODUCTION

Potato is one of the most common and popular food sources in everyday life all over the world because of its high productivity (average of 2 kg per single plant) and nutritional value (good source of vitamins and minerals). Ever since potato was found, people added their creativities in cooking potatoes using different methods, such as boiling, steaming, baking, roasting, and frying, etc. (Smith, 2012). Potato chips, one of the giant processed products made from raw potatoes, account for 21% of total potato utilization for processing in 2018 (NASS-USDA, 2019). There were about 281.46 million people who consumed potato chips in the U.S. in 2019, and it's believed the number will still be increased in the future (STATISTA, 2019b). The crispy texture and unique flavor of potato chips given by the frying process make them appealing snacks to consumers. The increasing demand for convenience food is another reason that the savory snacks become so popular. However, it is well known that oil consumption especially saturated fat is related to health problems, such as obesity, coronary heart disease (CHD), cancer and hypertension (Saguy & Dana, 2003). Due to changing lifestyles, snack food industries are now not only driven by convenience, but also healthiness. Consumers are craving for potato chips with lower fat content, but better nutritional value, and also with preserved texture and flavor.

Over the last decades, a large number of scientific works have been stimulated by consumer's expectations and tremendous efforts have been put in investigating the

complex mechanisms of oil absorption during deep fat frying to achieve desired low-fat products (Bouchon, Aguilera, & Pyle, 2003; Dana & Saguy, 2006; Garayo & Moreira, 2002; Moreira & Barrufet, 1998). Deep fat frying is the oldest and most popular frying process used not only in food industries, but also in restaurants because of its simplicity, versatility, and resulting in very desirable sensory characteristics (Arslan et al., 2018). During the frying, some critical phenomenon, such as dehydration, heat transfer, starch genitalization, protein denaturation, and crust formation, take place and affect final oil uptake and texture of the chips (Moreira, 2014; Zhang, Zhang, Fan, Li, & Fan, 2018). Oil uptake is highly affected by the frying time, the structure and initial moisture of the raw material, while independent of frying temperature (Gamble, Rice, & Selman, 1987; Kim & Moreira, 2013; McDonough, Gomez, Lee, Waniska, & Rooney, 1993; Moreira, Palau, Sweat, & Sun, 1995).

Several technologies were developed for oil reduction after having a better understanding of oil uptake mechanism, not only through controlling factors affecting oil absorption mentioned above, but also through using upgraded or novel frying methods. Attempts were first made through physical methods, such as de-oiling steps. The use of centrifuge system in frying was proven reducing oil content significantly and effectively (Kim & Moreira, 2013; Moreira, Da Silva, & Gomes, 2009; Pandey & Moreira, 2012). Higher centrifuge speed results in lower oil content of final crisps. With the highest tested speed, Pandey and Moreira found that the de-oiling step was able to remove 81% of the chip's surface oil (2012).

Another effective way of oil reduction is using vacuum frying, which is a trendy alternative to the conventional deep-fat frying. Vacuum frying is usually carried out under the pressure below 50 Torr (6.65 kPa), at lower frying temperature compared to atmospheric frying (Moreira, 2014). According to Garayo and Moreira (2002), vacuum frying shows a reduction of oil content up to 29% (d.b.) and gives lighter and softer potato chips than chips fried under atmospheric conditions. It has also been proved that vacuum frying can reduce acrylamide formation by 94% of potato chips (Granda, Moreira, & Tichy, 2004) and help the retention of nutritional quality of fried snacks due to lower processing temperature (Da Silva & Moreira, 2008; Perez-Tinoco, Perez, Salgado-Cervantes, Reynes, & Vaillant, 2008; Shyu & Hwang, 2001).

Pre-treatments of raw materials before frying also play an important role in oil reduction and texture improvement. Among them, the most used are blanching, pre-drying, freezing, osmotic dehydration, and coating, which are mainly used to preserve color, retain nourishments, and reduce oil content of final product (Pedreschi, Moyano, Santis, & Pedreschi, 2007).

In the past few years, one of the most popular pre-treatments that researchers interested in is the impregnation of metal ions, such as sodium, calcium, magnesium, and other solutes in raw plant tissue to modify the raw structure of the food material prior to frying, thus resulting in quality final product. The use of metal ions is believed to be relating to oil reduction of fried products, due to the maintained cell integrity (Da Silva, 2018). Bunger, Moyano, and Rioseco (2003) found that by soaking potato strips in 3% w/w NaCl solution for 50 minutes, the oil absorption was able to be decreased by 22%

compared to the control. It has been demonstrated that the use of $CaCl_2$ and $MgCl_2$ could not only cause the firming effect in plant tissue (Kasai, Okamoto, Hatae, & Shimada, 1997; Mierczyńska, Cybulska, Sołowiej, & Zdunek, 2015; Moustacas, Nari, Borel, Noat, & Ricard, 1991; Poovaiah, 1986), which stabilized the structure and resulted in reduced oil absorption, but also significantly reduced acrylamide formation in tortilla chips (Arámbula-Villa, Flores-Casamayor, Velés-Medina, & Salazar, 2018).

Although the use of metal ions, especially calcium ions, in improving the texture of food materials has been studied extensively, limited studies have been performed to provide experimental evidence relating the structure modification to oil reduction in deep-fat frying. In addition, most studies focused on calcium ion (Ca^{2+}) which can complex with cell wall and middle lamella pectin (Morris, 1980). The forming of Ca^{2+}-pectin bridges was proven to improve the cell wall stability profoundly (Demarty, Morvan, & Thellier, 1984; Koch, Naumann, & Pawelzik, 2019). However, the difficulties of finding balance between oil reduction and texture improvement limited the employment of calcium ion in fried product. The concentration of impregnated Ca^{2+} with the lowest oil uptake also results in potato chips with higher hardness (Da Silva, 2018), which may not be accepted by consumers. In addition, it may also bring some bitterness and other off-flavors to the product. Thus, this study uses magnesium ion (Mg^{2+}) which is a good alternative to Ca^{2+} because of the similar divalent behavior in stabilizing cell structure, but with the lower undesired hardness of the final crisps according to Arámbula-Villa (2018). Moreover, increasing magnesium intake is frequently recommended especially for those susceptible to high blood pressure, kidney stones and

bladder stones (Bajaj, 1987b). The effect of using a combination of Mg^{2+} and Ca^{2+} was also evaluated in the study.

To enhance the delivery of metal ions into the potato tissue, the combined technology of ultrasound and vacuum impregnation was introduced in this study. Sonication is considered as a promising technology and used extensively in food industry. Ultrasound technology is widely used in food productions to accelerate mass transfer process (Gamboa-Santos, Montilla, Soria, & Villamiel, 2012; Lagnika, 2018; Rodr ́guez et al., 2018). The most accepted mechanism of how the acoustic waves affect mass transfer is the introducing of cavitation and micro-channels (Fernandes & Rodrigues, 2007; Kek, Chin, & Yusof, 2013). The adequate use of ultrasound is a good assist for product modification or process improvement without causing any degradation (Knorr, Zenker, Heinz, & Lee, 2004).

In addition to ultrasound, vacuum impregnation has also been claimed as a useful technique of introducing desired solution into porous food materials, in a controlled way (Chiralt et al., 1999; Fito, Andr ́és, Chiralt, & Pardo, 1996). By using vacuum impregnation and vacuum frying technology, Moreira and Almohaimeed (2018) produced potato chips with 44% more phenol content than non-impregnated samples.

Because of the limitation and low efficiency of using a single method, a hybrid method of using both sonication and vacuum impregnation to efficiently deliver the divalent ions into potato tissue is introduced in this study. This method should be able to effectively control oil uptake of the potato chips without causing quality degradation.

CHAPTER II

HYPOTHESIS & OBJECTIVES

2.1 Hypotheis

Mg^{2+} should have a similar divalent behavior to Ca^{2+}, which stabilizes the cellular matrix of potato tissues, enhances cell integrity, and consequently affecting the oil uptake during deep-fat frying. The "sponge effect" brought by sonication is expected to enhance the delivery of metal ions into potato tissues during later vacuum impregnation process. The structural alteration of the potato tuber should play a role in oil uptake mechanisms.

2.2 Objectives

The main objective of this study is to gain an in-depth understanding of the effect of impregnated metal ions, using the combined technologies of sonication and vacuum impregnation, on oil uptake during frying and also overall quality of potato chips. The ultimate goal is to develop a process to produce potato chips with lower fat content while keeping their desirable quality attributes. To achieve the goal of this study, the following specific objectives are designed:

1. To evaluate the effect of vacuum impregnation and sonication on oil absorption.
2. To measure the changes of mechanical strength (firmness or hardness) after the pre-treatment on both raw and fried samples.

3. To evaluate the effect of using a combination of $MgCl_2$ and $CaCl_2$ as soaking solutions on oil uptake and other quality attributes.
4. To quantify mass transfer and material balance during the pre-treatment.
5. To perform kinetic studies (moisture loss and oil absorption) of pre-treated potato chips.
6. To study microstructural changes of both potato slices and potato chips induced by pre-treatment using the scanning electron microscope (SEM).
7. To characterize the product quality attributes (PQA) such as moisture content, oil content, color, texture, bulk density, solid density, porosity, and shrinkage using objective methods.
8. To assess the general acceptance of pre-treated potato chips by conducting sensory evaluation.

CHAPTER III

LITERATURE REVIEW

3.1 Potato

The potato (*Solanum tuberosum*) is one of the most important non-cereal world crops (Bajaj, 1987). It is the fourth most important food crop over the world, just after maize, wheat and rice (FAOSTAT, 2008). Potato tubers are characterized by their prolificacy, in which a single plant can produce about 2kg of potatoes (Smith, 2012). As an everyday food all over the world, potato is primarily consumed as a source of carbohydrates, substituting for rice or wheat (Bajaj, 1987).

3.1.1 Structure and Chemical Composition

Figure 1 distinguishes the principle tissue types of a mature potato tuber. The outer skin consists of a layer of cork cells with an averaged depth of 10-11 cells, usually called periderm (Fedec, Ooraikul, & Hadziyev, 1977). Underlying the periderm is the cortex, a thin layer of parenchyma tissue, containing the largest cells in the tuber (Andersson et al., 1994). Vascular storage parenchyma contains cells high in starch, lying adjacent to vascular ring (Reeve, Hautala, & Weaver, 1969). The cells in pith, or water core appeared smaller and had lower starch content (Fedec et al., 1977).

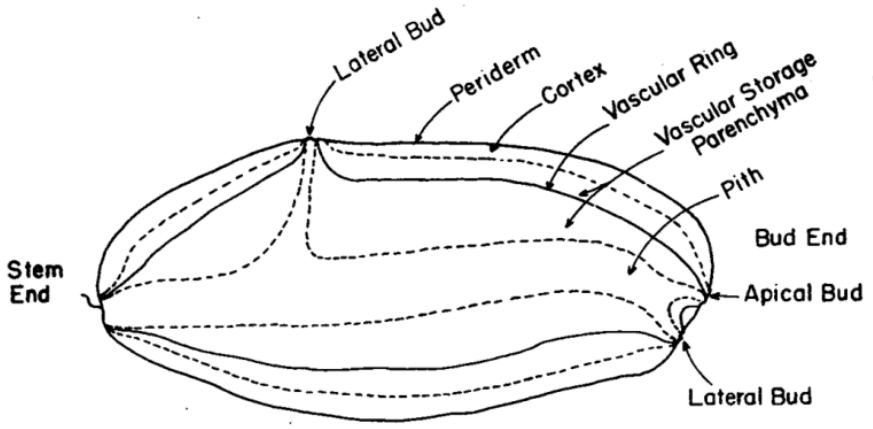

Figure 1 Longitudinal section of a Russet Burbank potato showing principal structure features - adapted (Talburt & Smith, 1967).

Apart from the structure of potato tissues, the chemical composition of potato tubers also plays an important role in their functions and utilizations. It varies with potato variety, maturity, growth condition, storage time, and so on. A medium-sized raw potato consists of about 70-80% moisture and 20-30% of total solids. Dry matter in potato tuber is mainly presented as easily digestible carbohydrates (starch) and a small amount of non-starch polysaccharides as cell wall components (Bajaj, 1987b; Talburt & Smith, 1967). The high starch content makes potato a staple starchy food contributing to energy (carbohydrates) intake to the diet (Kolasa, 1993). It also contains about 2-3% of protein, 1% of ash, and negligible amount of fat. In terms of other nutritional values, potato has also been long valued as a good source of vitamin C (44% RDA), B6 (29%), B1 (16%), folate (16%), and it also provides some minerals such as potassium, iron, and magnesium for diet (Storey, 2007). The raw potato usually has a calcium content of 5-18

mg/100 g fresh potato and magnesium content of 14-18 mg/100 g fresh potato (Bradshaw & Ramsay, 2009).

3.1.2 Cell Wall and the Firming Effect

The mechanical behavior of potato tissue is a critical factor affecting the texture of cooked product and, consequently, contributing to consumer's acceptability and preference. Previous studies have demonstrated that the mechanical properties of plant mainly depend on its structural component (cell wall infrastructure) and turgor pressure (hydrostatic influence) within the cells (Niklas, 1989; Scanlon, Pang, & Biliaderis, 1996). It was evidenced that the increase of cell turgor pressure can increase the firmness of potato tissue (Nilsson, Hertz, & Falk, 1958). The main factors affecting cell turgor pressure are moisture loss and membrane integrity (Scanlon et al., 1996). From macrostructural aspect, the mechanical strength of potato tuber was found correlating to the dry matter (DM) and starch content (Koch, Naumann, & Pawelzik, 2019).

Figure 2 shows the schematic representation of common plant cell wall architecture. The overall cell wall can be viewed as a polymeric network of polysaccharides. These polysaccharides are linked together through covalent, ionic, and hydrogen bonding to together influence the mechanical properties of the cell wall (Jarvis, 2011). The propositions of different polysaccharides in cell wall have been found as: 47-66% of pectic polysaccharides (pectins), 15-28% of cellulose, and 6-10% of hemicellulose (Lisinska & Leszczynski, 1989). Although the proportion of cell wall and the middle lamella in potato tissue is quiet small, their impact on the texture of raw as well as cooked potato tissues is profound (Hoff & Castro, 1969). The cell wall structure

is fairly organized, in which cellulose microfibrils build a skeleton, cemented by a cross-linked phenolic polymer – lignin and also some pectic substances (Chang, Tsai, & Chang, 1993; Kita, 2002). The formed skeleton is embedded in a matrix of hemicellulose, pectic substances and glycoproteins (Andersson et al., 1994). The middle lamella is the outer layer of the cell wall, formed by both adjacent cells, cementing cells together (Demarty et al., 1984). The adhering function of the middle lamella attributes to its major composition: pectic polysaccharides, which act as sticky fluid and hold cells tightly together. Thus, these pectic substances play a key role in preventing cell separation and maintaining the integrity and mechanical strength of the tissue (Marle, 1997).

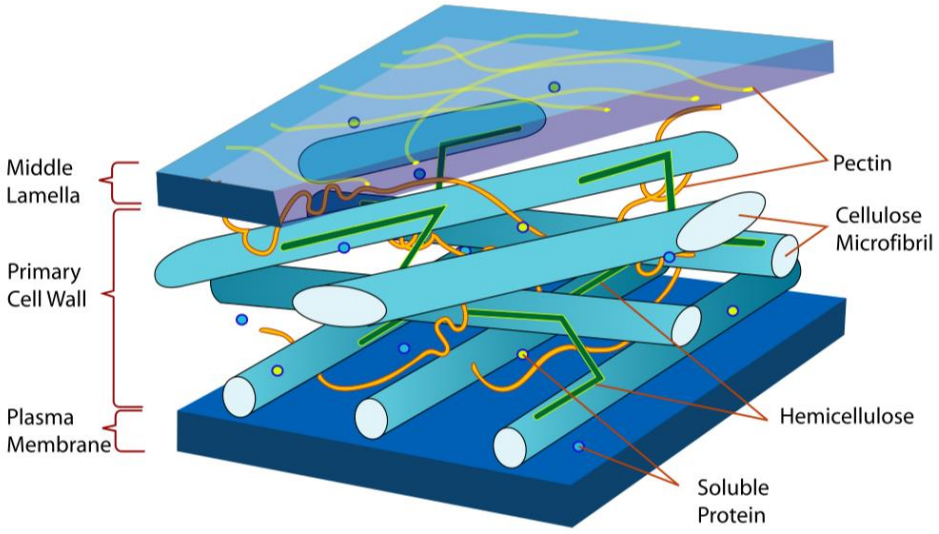

Figure 2 Schematic representation of plant cell wall - adapted (LadyofHats, 2007).

Pectins are heteropolysaccharides, consisting of galacturonic acid backbones, with α (1→4) linkage (Brejnholt, 2009). It is held that when initially synthesized, most carboxyl groups are methyl esterified, and then partially de-esterified once transferred to cell wall by a ubiquitous enzyme: pectin methyl esterase (PME). Those non-esterified and non-branched galacturonic acid residues can then be cross-linked by divalent ions to form junction zones (Jarvis, 1984). The junction zone was described by Grant et al. (1973) using an "egg-box" model (Figure 3). The divalent ion plays a vital role in gelling properties of pectins by reducing charge repulsion and providing cross-bridges (Demarty et al., 1984). Hence, the cell wall integrity or strength can be maintained by controlling the level of divalent ions in the structure system. The formation of such cross-bridges in the potato tuber is a determinant factor on processing properties of potato and the quality of its products such as potato chips and French fries (Murayama et al., 2017).

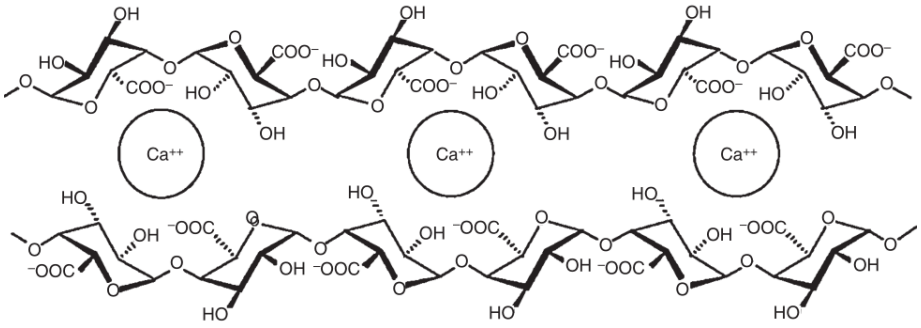

Figure 3 The "egg-box" model illustration the cross-bridges provided by Ca^{2+} - adapted (Brejnholt, 2009).

Numerous studies have been carried out to unravel the mechanisms of the "firming effect" brought by divalent ions, in most cases, calcium and magnesium (Demarty et al., 1984; Koch et al., 2019; Moustacas, Nari, Borel, Noat, & Ricard, 1991; Scanlon et al., 1996), and how this relates to the cooking process (Andersson et al., 1994; Bartolome & Hoff , 1972; Harris, 2009; Marle, 1997). The role of divalent ions in the firming effect can be explained by the cross-linking with pectic substances in cell wall and middle lamella (Grant et al., 1973; Van Buren, 1979), as well as causing changes of turgor pressure within the cell (Falk, Hertz, & Virgin, 1958; Pitt, 1982). This was then supported experimentally by Scanlon (1996) who reported that the firmness of the cell increases as turgor pressure increases. However, the availability of divalent ions in potato tubers was suggested to be a limiting factor. In addition, it was reported that 20% of calcium present in raw potato tissue was lost into the cooking medium during cooking (Van Marle, Van Dijk, Voragen, & Biekman, 1994). Thus, impregnation of these cations into potato tissues using different methods became trendy regarding to structure modification technologies.

The complexity of the mechanism rises when incorporating the cooking procedure, where starch genitalization, protein denaturation, heat and mass transfer all take place which, in turn, affecting the final product texture. For many years, it has been disrupted that which mechanism is the major contributor to the process of firming. However, it was generally accepted that the firming of cooked food products attributes to: the retrogradation of starch, stabilization of the cell wall and middle lamella by activating pectin methyl esterase (PME), leaching of amylose, and the formation of salt

bridges between pectin molecules (Andersson et al, 1994). However, while studying such firming effect, the cooking methods that most scientists used were boiling or steaming, instead of frying, in which different combinations of mechanisms may take place due to the lower moisture system, and the incorporation of oil. Limited studies relating such firming effect to oil absorption in frying process. Tajner-Czopek (2003) found that blanching potato strips into 0.4% $CaCl_2$ or 0.4% $MgCl_2$ prior to frying prevented further loss of the pectic substances and resulted in French fries with markedly improved texture. The impregnation of Ca^{2+} and Mg^{2+} preserved the pectic substances by cross bonding, and thus resulting in firmer French fries. These results are in accordance with those found by Khalil (1999) who reported that soaking potato cylinders in 0.5% $CaCl_2$ and 5% pectin reduced the oil content by 40% and also gave a firmer product with the highest sensory scores. However, the mechanism of how impregnated divalent ions affecting oil uptake is still not fully understood.

3.2 Snack Food

3.2.1 Snack Food Market

Due to the fast-paced lifestyle in modern society, demand for convenience food is expanding world widely. Among all convenience foods, salty snacks segment is one of the most important contributors to the market, especially in the U.S. Salty snack becomes a big part of consumer's routine, with sales of $18 billion in 2018 in the U.S. domestic market (Salty Snacks - US, 2019). According to the Global Industry Guide for Savory Snacks (2018), the total revenue of global salty snacks market was $104.7 billion in 2017.

Salty or savory snacks usually refer to popcorn, pretzels, potato chips, nuts, crackers, meat snacks, tortilla chips and other salty snacks. Within snacks market, salty snacks contribute more than one-fifth of snack sales in the U.S. (Nielsen, 2017). Although the sale is already huge, the snack food market is still expected to grow annually by 2.4% from 2020-2023 (STATISTA, 2019a).

While craving for satisfaction is a key component of the driving force in the development of snacks industry, consumers now are more interested in healthier snack products (Mariscal & Bouchon, 2008). The health trend of demanding for low-fat products is a big challenge for snack industry to improve existing or develop new alternative technologies (Moreira, Castell-Perez, & Barrufet, 1999).

3.2.2 Potato Chips

Potato chips were defined as fried thinly sliced (1.27-1.78mm thick) potatoes, with final fat content of 33-38% and moisture content of 1.3-1.5% to ensure their crispness and stability (Kirkman, 2007; Moreira et al., 1999). As the leading revenue for salty snacks, potato chips have average monthly sales of $1,733 in U.S. per one convenience store in 2018, followed by tortilla chips, nuts, and pretzels (STATISTA, 2019c). The world market for potato chips has a value of $14.3 billion in 2002 (Datamonitor, 2004).

3.2.3 Healthy Snacks

According to Waxman (2004), the high consumption of saturated fat foods is a primary factor for the increased obesity. It is recommended to limit energy intake from saturated fat and undertake a low-fat diet. Thus, several approaches have been

undertaken to make fried snacks more acceptable especially to those health-conscious consumers. In addition to fat, Shukla (1994) also addressed the potential for snack foods as a carrier of nutrients, such as minerals, phytochemicals and vitamins through pre-treatments.

However, techniques resulting in low-fat food products still have not been used widely in food industry because of the difficulties to preserve desired quality attributes such as texture, color, and flavor, with reduced oil uptake (Bouchon & Pyle, 2004).

3.3 Deep-fat Frying

Deep-fat frying or immersion frying can be defined as a cooking process by immersing food materials in an eatable oil or fat, typically at a temperature of 150-200 ℃ (Farkas, 1995; Yamsaengsung & Moreira, 2002). The palatability of deep-fat fried food is related to unique organoleptic and sensory characteristic, including flavor, texture and appearance (Saguy & Dana, 2003). It is one of most widely used unit operations in the food industry because of its operational simplicity, high speed processing, and resulting in palatable products.

Deep-fat frying is a rather complex process in which heat and mass transfer take place simultaneously, resulting in counterflow of water vapor (bubbles) and oil at the surface of the fried food material (Bouchon, 2011). The schematic diagram of heat and mass transfer process is shown in Figure 4. The heat is transferred from the hot oil to the surface of the fried food piece by convection, and then to the core by conduction. Surface water reaches the boiling point first, resulting in rapid dehydration on the surface. Subsequently, the increased pressure caused by the boiling of interior moisture

pushes moisture out to the surface, and finally, to the oil system. Oil infiltration also takes place during the frying process (Alvis et al., 2009; Gertz , 2014; Vitrac, Trystram, & Raoult-Wack, 2000).

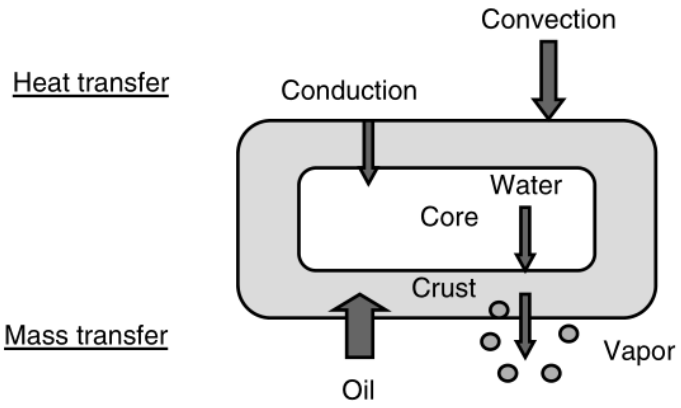

Figure 4 The schematic diagram of simultaneous heat and mass transfer during frying.

The deep-fat fried product is characterized with a dry, porous, crispy crust, and a moist cooked core. During frying, several physicochemical alterations and microstructural changes are involved, making the process difficult to be understood. The major coupled physical and chemical phenomena occurring during frying include starch genitalization, protein denaturation, degradation of the cell wall and middle lamella, water evaporation and consequent dehydration, as well as crust formation (Bouchon & Aguilera, 2001).

3.3.1 Oil Absorption Mechanisms

As mentioned previously, consumers' preferences are moving towards healthier foods low in fat content due to their increased health concerns. Thus, a good understanding of the mechanisms involved in oil absorption is paramount in developing or optimizing manufacturing technologies to meet consumer's demand for low-fat products. Despite extensive scientific works have been done over the past few decades, the mechanisms have not yet been clearly described and debates still exist.

The mechanism was first described by Gamble et al., (1987) who stated that most of the oil enters the product after being removed from the fryer due to the condensation of steam, creating a vacuum. The adhering surface oil is sucked into the product because of the pressure gradient through the moisture loss sites. The results indicated that the oil uptake is directly proportional to the amount of moisture lost.

However, in another study, Ufheil and Escher (1996) suggested that oil uptake is a surface phenomenon involving equilibrium between adhesion and drainage of oil upon retrieval of the slice from the oil. By mixing the oil with a fat-soluble and heat stable dye, they successfully located the oil in the product and found that there's no oil penetrate the potato slices during frying. Oil was absorbed only after potato slices have been removed from the frying pan due to adhesion of oil to the slice surface. This was in agreement with the results of Moreira et al (1997), who found that the largest amount of the oil (64%) was taken up by tortilla chips during the cooling period, followed by 20% during frying and 36% left at chip's surface. It was also showed that oil content of the tortilla chips was significantly affected by initial moisture content and particle size distribution,

while independent the oil quality or temperature. Higher initial moisture within the sample and smaller particle size resulted in higher oil uptake. However, the result was found to be contrary according to Dudley (1993), who demonstrated that as initial moisture decreases, the final oil content of tortilla chips increases. Another explanation of the oil uptake mechanisms given by Moreira and Barrufet (1998) used capillary pressure theory.

McDonough et al., (1993) described the mechanisms as a continuous process as part of a replacement between evaporated moisture and oil. As it reached the boiling point, moisture turned to steam and escaped from the chip to the oil, leaving behind a sponge-like network of tunnels which was then filled by absorbed oil.

3.4 Pre-treatments Used in Fried Foods

No matter which mechanism is used to explain the oil uptake process, it is generally agreed that the initial product characteristics, especially structure and moisture or solid content are key factors to oil absorption. Extensive pre-treatments have been carried out prior to frying to obtain reduced-fat products. Among them, most commonly employed are blanching, pre-drying, osmotic dehydration (OD), soaking, and coating.

3.4.1 Blanching

Aguilar, Anzaldúa-Morales, Talamás, and Gastelum (1997) reported that a low-temperature (55-70 ℃) blanching activates the pectin esterase enzyme (PME), thereby resulting reactions decrease porosity and hence reduce oil uptake. Potatoes blanched at 65 ℃ for 30 min were found to produce the lowest oil content French fries. This result is in agreement with the work of Kim and Moreira (2013), who found that blanching

improved oil uptake significantly. The oil reduction was also attributed to surface starch gelatinization (Califano & Calvelo, 1998). However, some researchers found blanching pre-treatment at higher temperature for short time resulted in higher oil content products (Álvarez, Morillo, & Canet, 2000; Pedreschi, Moyano, Kaack, & Granby, 2005). Some authors found no significant difference between blanched and unblanched samples (Al-Khusaibi & Niranjan, 2012). As can be seen, results may be contradictory, depending on specific methods used or treatment conditions sample subjected to. Other advantages of blanching pretreatment including: texture and color improvement, as well as decreasing acrylamide content of the final product. Blanching was found to improve color of the fried product, by means of leaching out reducing sugar on sample surface (Pedreschi et al., 2005). The texture was also improved by low-temperature blanching, due to the formation of stronger intercellular bonds in response to PME activities (Álvarez et al., 2000). A dramatic reduction (~64%) on acrylamide formation was detected using blanching pre-treatment (Mestdagh et al., 2008; Pedreschi, Kaack, & Granby, 2004; Pederschi et al., 2005).

3.4.2 Pre-drying

Pre-drying is also a popular pre-treatment method prior to frying, resulting in potato samples with maintained initial solids content. Most commonly used dehydration techniques in pre-treatments of fried potato products include hot-air drying, microwave drying, freeze drying, and osmotic dehydration (Gamble & Rice, 1987; Lamberg, Hallstroem, & Olsson, 1990; Moreno & Bouchon, 2008; Pedreschi & Moyano, 2005). A significant reduction in oil content of deep-fat fried potato products was reported using

conventional hot-air, microwave, and osmotic dehydration techniques, whereas freeze drying was found to adversely affecting the oil reduction. The opposite result of freeze drying pre-treatment was attributed to the formation of a fragile external porous region which imposes low resistance and leaves oil with enough space to enter the potato tissue. Although the aim of these pre-treatments was to achieve potatoes lower in moisture content, the oil reduction was generally believed not due to the moisture reduction on its own, but due to the structural changes occurring at the surface of potato slices, which reduced surface permeability (Moreno & Bouchon, 2008).

3.4.3 Osmotic Dehydration and Soaking

Osmotic dehydration (OD) is another useful technique providing intermediate moisture food for further preservations such as freezing and drying (Azuara, Cortés, Garcia, & Beristain, 1992). The treatment is carried out by immersing food materials in hypertonic solutions, most commonly sugar (sucrose) and salt (sodium chloride) solutions, leading to moisture loss of the cellular materials (Sereno, Moreira, & Martinez, 2001). The diffusion of water from the interior cellular structure into the solution is driven by osmotic pressure differences, resulting in loss of turgor pressure. During osmotic dehydration treatment, two major simultaneous counter-current flows are noted: moisture flows from food to osmotic solution, causing dehydration, and solutes flow from solution into the food structure (Zhao & Xie, 2004). Accordingly, the employed solution in osmotic treatment could not only affect efficiency of the diffusion, but also adding values or alter structure of the product. Mauro et al., (2016) demonstrated that the addition of calcium lactate to sucrose osmotic solutions enhanced the OD efficiency. The

formation of calcium pectate complex also contributes to cell wall pore reduction, and consequently affecting the oil absorption.

Soaking is another word usually used when correlating to pre-treatment prior to frying process. It is applied with the goal of modifying the composition or structure of food material through partial water removal and impregnation of solutes, thereby reducing oil uptake and improving texture of the fried product. It carries out a short osmotic dehydration (OD), while solute impregnation onto the plant surface is believed as a more important factor rather than the water loss (Moyano, Ríoseco, & González, 2002). Several solutions have been used in the soaking pre-treatment, including NaCl solutions (Bunger, 2003; Kim & Moreira, 2013; Moyano et al., 2002), $CaCl_2$ solutions, and $MgCl_2$ solutions (Arámbula-Villa et al., 2017), as well as some combinations (Moyano & Berna, 2002; Tajner-Czopek, 2003). Similar results were obtained with reduced oil content and improved texture, color and flavor of the final products. In addition to aforementioned explanations of fat reduction, Rubnov and Saguy (1997) studied the surface characteristics of restructured potato products, and proposed that the oil reduction was due to the enhanced crust formation when solutes are concentrated on the surface, acting as a barrier to oil uptake. Based on previous literature, $MgCl_2$ was selected as soaking solution in this study. The effect of using a combination of $MgCl_2$ and $CaCl_2$ was also evaluated through oil uptake and several quality attributes to see if they can provide a synergistic effect.

3.4.4 Coating

The reduction of surface permeability can also be achieved through edible coating. Several hydrocolloids were employed by virtue of their film-forming capability. Some of them can also reduce the outer surface porosity (Bouchon, 2011). Most commercial biopolymer coatings used to inhibit fat uptake are polysaccharide coatings, such as alginates, methylcellulose (MC), hydroxypropyl methylcellulose (HPMC), long fiber cellulose, guar gum, and pectin (Khalil, 1999; Mellema, 2003). It was reported that the oil content of potato chips coated with guar gum (1%) and glycerol (8%) can be reduced up to 34.8% (Yu, Li, Ding, Hang, & Fan, 2016). The coating of 0.5% sorbitol and 1% methylcellulose reduced the oil content of French fries by 40.6% (Garcıa, Ferrero, Bertola, Martino, & Zaritzky, 2002). The mechanism of how coating helps reducing oil uptake was not clearly clarified, but possible contributors could be its water binding or gel forming capability, increased water retention, and decreased fat diffusivity (Garcıa et al., 2002; Khalil, 1999; Mellema, 2003). However, coating was reported bringing some off-flavors and reducing the sensory acceptability of fried potato products (Khalil, 1999).

3.4.5 Ultrasound

Regarding low efficiency of a single pretreatment mentioned above, ultrasound is a promising and novel technology usually used in the process to accelerate heat and mass transfer (Chemat et al, 2017; Rodr ́guez et al., 2018). The application of power ultrasound (frequency of 20-800 kHz) has been widely reported in the pretreatment of fruits and vegetables before drying, while the employment of ultrasound technology

prior to frying could be rarely found in the literature (Antunes-Rohling et al, 2018; Dehghannya, Naghavi, & Ghanbarzadeh, 2016; Fernandes & Rodrigues, 2007; Rodríguez et al., 2018). The sound waves agitate particles in the food material, generating a rapid series of alternative contractions and expansions, which can be viewed as squeezing and releasing a sponge constantly (sponge effect) (De la Fuente-Blanco, De Sarabia, Acosta-Aparicio, Blanco-Blanco, & Gallego-Juárez, 2006). In addition, the production of cavitation and formation of microscopic channels are also main effects associated with acoustic waves, which facilitate removing strongly bounded water in solid matrix and diminishing diffusion boundary layer and thereby enhancing convective mass transfer (Cárcel, Garcia-Perez, Riera, & Mulet, 2011; De la Fuente-Blanco et al, 2006).

Numerous studies have proposed the ultra-sonication as an innovative emerging technology to reduce oil uptake in frying process. For instance, Karizaki and others (2013) investigated the effect of ultrasound-assisted osmotic dehydration on oil absorption and the quality of fried potatoes. Results demonstrated that the oil content of pretreated potato slabs was lowered by 12.5% (d.b.) as compared to control samples. The use of ultrasound was also found to shortening the treatment time by 67% and improving the color of produced French fries. The result was then confirmed by Dehghannya, Naghavi, and Ghanbarzadeh (2016), who reported that potato strips pretreated with both ultrasound and dehydration decreased the oil update significantly. Ultrasonic waves were confirmed showing a synergistic effect on reducing oil absorption when used in combination with osmotic dehydration as pretreatment prior to frying (Dehghannya &

Abedpour, 2018). Potato strips pretreated with ultrasound and osmotic solutions together showed significant less oil content compared to those pretreated with either ultrasound or osmotic dehydration separately. Mohammadalinejhad and Dehghannya (2018) also investigated the feasibility of ultrasound application to produce low-fat French fries through deep-fat frying. By evaluating the influence of various ultrasound frequency-time combinations, they found the lowest oil uptake was obtained by subjecting potato strips to ultrasound at the frequency of 40 kHz for 30 min. Such ultrasound treatment showed the highest oil reduction of 23.18% among all samples and also showed the highest moisture diffusivity. Accordingly, it was generally believed that ultrasound is a good pre-treatment before frying which yields promising results in reducing both moisture content and fat uptake (Oladejo et al., 2017).

The microstructure changes induced by ultrasound were also investigated by many researchers using different microscopy methods (mainly SEM-scanning electron microscopy). It was observed that potato samples treated with ultrasound showed a more ruptured structure, with collapsed, damaged cells and more pores (Lagnika et al., 2018; Miano, Rojas, & Augusto, 2019; Su, Zhang, Adhikari, Mujumdar, & Zhang, 2018). The SEM images also proved the formation of microchannels in potato tissues. The cell disruption was mainly owing to the osmotic solution because longer time of osmotic dehydration without ultrasound also caused similar breakdown of cell structure, thus the concentration of osmotic solution had a larger impact on potato microstruture (Karizaki et al., 2013). This finding was in accordance with observations by Oladejo, Ma, Qu, Zhou, and Wu (2017) that sweet potato samples treated with ultrasound in distilled water

showed less cell rupture in comparison with those treated in osmotic dehydration, with and without ultrasound. However, Lagnika (2018) observed that the structure of osmotic treated sweet potatoes showed less disruption than ultrasound treated samples. Theoretically, the formation of microscopic channels in potato matrix could enhance mass transfer of both moisture and oil during frying. The lower oil uptake was indicated to be a result of solutes uptake the surface of tissue, and consequently obstructing oil suction into the structure (Oladejo et al., 2017). In summary, the utilization of sonication in this study was expected to (1) enhance the delivery of treated divalent ions (Mg^{2+} and/or Ca^{2+}) into potato tissue, thereby modifying the structure with maintained cell integrity; and (2) create cavitation and micro-channels, thus leaving more spaces and pathways for further vacuum impregnation.

3.4.6 Vacuum Impregnation

Vacuum impregnation is another valuable technique, usually used to introduce desired ingredients into a porous structure of foods. It is highly affected by structure of food tissues (pores), and process conditions (vacuum pressure; vacuum time; restoration time; temperature) (Zhao & Xie, 2004). It has been reported that the vacuum treatment leads to a significant increase in water loss (WL) and solid gain (SG), enhancing mass transfer process between treated foodstuffs and impregnating solution (Martínez-Monzó, Martínez-Navarrete, Chiralt, & Fito ,1998; Fito, 1994; Fito, Andrés, Chiralt, & Pardo, 1996). The mass transfer during VI was described by Fito and Pastor (1994) on the basis of the Hydrodynamic Mechanism (HDM). The exchanging between gases occluded in the intercellular spaces and external impregnating liquid was driven by the combined

effect of pressure gradients and capillary actions. The existence of the wide intercellular spaces which are responsible for the porosity was affirmed by Cryo-SEM observations of Fito and others (2001). The effectiveness of VI was confirmed by Martínez-Monzó et al. (1998), who successfully observed the impregnated solutes in the apple pores.

Several researches have been carried out using VI technique as a pretreatment prior to frying, either to modify the structure or to fortify the formula. Moreira and Almohaimeed (2018) used vacuum impregnation of red rootbeet, together with vacuum frying technology in producing healthy potato chips. With optimal vacuum impregnation and frying conditions, they produced potato chips with 32% more total phenolic compounds, which is an important health promoting compound contains a high level of antioxidant capacity. Using the similar methods, Lopez and Moreira (2019) also increased phenolic compounds significantly in potato chips vacuum impregnated with green tea. To produce calcium fortified potato chips, Tiwari, Joshi, Varghese, & Thakur (2018) investigated the effect of several concentrations of calcium chloride and different VI conditions on calcium levels in final product. The results showed that the calcium concentration in potato chips was increased from 154.65 mg (control) to 700 mg/100g under best treatment conditions. Thus, in this study, VI could be a potential way to further enhance the delivery of metal ions into the potato structure, thereby increasing cell integrity against oil absorption during deep-fat frying.

CHAPTER IV

MATERIALS AND METHODS

4.1 Potatoes

Potatoes (*Solanum tuberosum*) of the Snowden variety, used for this experiment, were provided by the CSS Farms, LLC, Dalhart, TX. This cultivar is commonly used in the chips market because of its high specific gravity of about 1.075 and uniform round to slightly flat shape (Mwatuwa & Christensen, 2016; Talburt & Smith, 1967). Potatoes were stored at 7 ℃ and 90% relative humidity. Before frying, the potatoes were reconditioned in a dark place at ambient temperature for 5-10 days to lower the amount of reducing sugars, which may cause undesirable browning during the frying process.

4.2 Sample Preparation

Potatoes were selected according to their specific gravity, ranging from 1.078 to 1.080. The selected potatoes were washed, peeled, and sliced (thickness of 1.60±0.10 mm) using a Mandolin slicer (Matfer model 2000). Transverse slices from the middle portion of the tuber were selected (~4 slices/tuber) and then cored to a round shape through a metal cutter with internal diameter of 50.8mm. The cut slices were rinsed in reverse osmosis (RO) water at room temperature (21.0±1.0 ℃) to remove the surface attached starch and then blotted dry gently with a paper towel.

4.3 Pre-treatments

The pre-treatments used in this study included immersing samples in soaking solutions (RO water, $MgCl_2$, or a combination of $MaCl_2$ and $CaCl_2$) at several concentrations, and simultaneously subjecting the samples to ultrasound followed by

vacuum impregnation with 10000 ppm (1% w/v) $MgCl_2$. Six concentrations of $MgCl_2$ were used as soaking solutions during sonication, including 0 ppm (RO water), 5000 ppm, 10000 ppm, 15000 ppm, 20000 ppm, and 50000 ppm. For each of these concentrations, three sonication times were used: 10, 30, and 50 minutes. A mixture of 15000ppm $MgCl_2$ and 15000ppm $CaCl_2$ each (50% v/v) was also used as soaking solution to compare the effect between only Mg^{2+} and the combination of Mg^{2+} and Ca^{2+} on potato chips' quality. Vacuum impregnation conditions were kept constant (100 ml $MgCl_2$/slice; 600 mm Hg pressure; 10 min vacuum time; 10 min restoration time). Different pretreatment parameters and levels were selected to do specific assessments. The detailed experimental design is described in Section 4.3.4. Once the pretreatment was done, samples were removed from the solution, dried with blotting paper, and then fried. Each test was carried out in triplicate.

4.3.1 Sonication Process

A Bransonic ultrasonic tank (B52 model, Branson Co., Shelton, CT), with capacity of 5 L and power of 240 W, was used in the experiment and filled with ice (made of RO water). Before the experiment, about 1kg of ice was added to 4 L of RO water to obtain a temperature of 4 ℃ in the tank. Another 1kg of ice was added above the sample to maintain the temperature throughout the experiment, thus preventing drastic temperature rise due to sonication process. About 16 potato slices, contained in an air tight plastic bag, filled with soaking solution (100 ml) and sealed with a FoodSaver vacuum sealer, were submerged in the ultrasonic bath (Figure 5). Samples were

submitted to ultrasonic waves with constant frequency (47 kHz) continuously throughout the treatment periods.

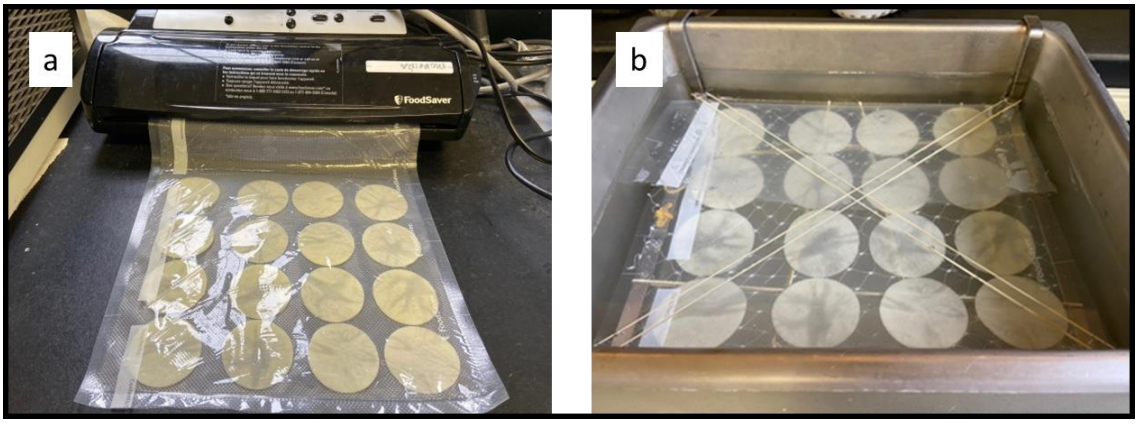

Figure 5 Samples sealed in soaking bags (a) and loaded in ultrasonic tank (b).

4.3.2 Vacuum Impregnation

The vacuum impregnation system is composed of a desiccator, a vacuum pump, a connecting tube, and glass jars to hold potato slices (Figure 6). Each potato slice treated with ultrasound was put in a glass container and then filled with 100 ml of impregnating solution (10000 ppm $MgCl_2$). The concentration of 10000 ppm was found sufficient to carry out vacuum impregnation at the ratio of 100 ml $MgCl_2$/slice. The experiment used predetermined constant vacuum pressure of 600 mm Hg, vacuum time of 10 min and restoration time of 10 min. The vacuum impregnation condition was kept constant in order to eliminate more variables or errors in the experiment, thus the comparison makes more sense. The constant vacuum impregnation step was added to the study after a complete sonication process to evaluate if adding such step will bring

synergistic effect on quality characteristics of the final product. The VI treatment was carried out at ambient temperature (21.0 ±1.0 ℃) by applying vacuum for 10 min, and afterwards restoring to atmospheric pressure in 10 min, while keeping potato slices remained immersed.

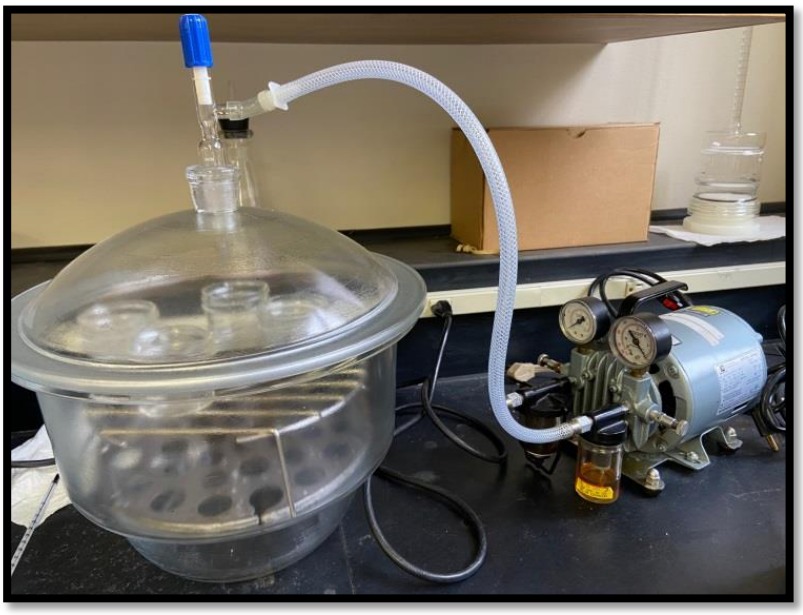

Figure 6 The vacuum impregnation system used in this study.

4.3.3 Frying Process

A deep-fat fryer (George Foreman Spin Frying Machine Model GSF026B) of capacity 2.6 L was used in the study for frying of pretreated and untreated (control) samples. The fryer consists of a centrifuge system with the rotating speed of 457 ±1 rpm. Four potato slices (~12 g) were loaded into the fryer basket, placed with a round aluminum mesh screen above them to make sure all samples were kept submerged in the

oil throughout the frying period (Figure 7). Once the temperature of the frying oil (canola oil – brought from local HEB market) reached 165 ℃, the basket was lowered and potato slices were fried for 4 minutes to get fully cooked potato chips, with the moisture content below 2% (w.b.). After frying, the basket with samples were lifted up from the hot oil and then centrifuged for 40 sec. The chips were removed from the basket after the de-oiling step. They were allowed to cool down for 2 min at room temperature (21.0 ±1.0 ℃) and the oil on chip's surface was removed by gently blotting with paper towel. The samples were stored in mason jars, labelled properly, and then placed in a desiccator for further analysis.

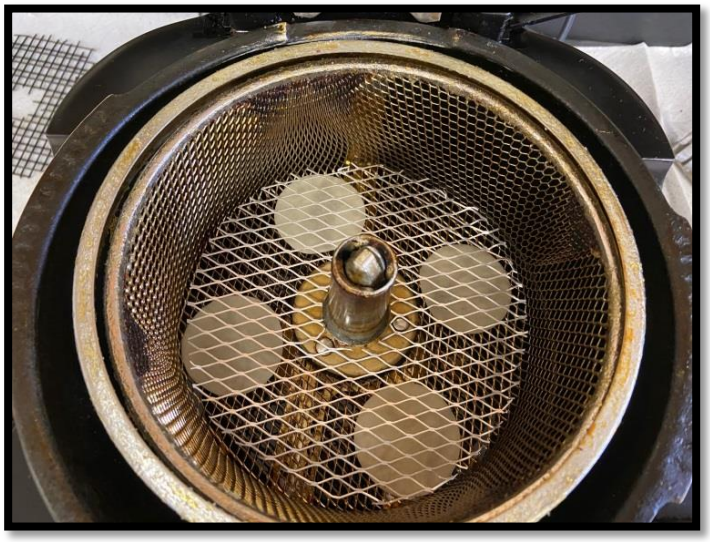

Figure 7 Samples loaded in the deep-fat fryer basket.

4.3.4 Experimental Design

The experimental design consisting four different pretreatment types is presented in Table 1. To evaluate whether the combination of sonication and vacuum impregnation (VI) has a synergistic effect on reducing oil uptake, three different categories of pretreatment, including the sonication-assisted vacuum impregnation (SVI), non-sonication-assisted vacuum impregnation (NSVI), sonicated without vacuum impregnation (S) were carried out and compared on oil uptake. For NSVI treatment, potato slices were immersed in soaking bags (without applying ultrasound) filled with 15000 ppm $MgCl_2$ solution for 10, 30, and 50 min at a ratio of 16 potato slices/100 ml $MgCl_2$, followed by vacuum impregnation procedures. The ratio (16 potato slices/100 ml) was predetermined to assure an adequate amount of fluid medium for 16 slices. The soaking temperature was controlled at about 4℃ using RO ice in the ultrasonic bath to eliminate errors brought by temperature differences in the result. During the S treatment, potato slices were soaked in 15000 ppm $MgCl_2$ at a ratio of 16 potato slices/100 ml $MgCl_2$ and simultaneously subjecting to ultrasound. After the completion of sonication, samples were removed from the soaking bag, and then soaked in glass containers at a ratio of 1 slice/100 ml $MgCl_2$ (10000 ppm) for 20 min, which is the duration of the VI process. The SVI was carried out by combining sonication and vacuum impregnation (VI) steps. Samples were sonicated in soaking solutions (RO water, only $MgCl_2$, or a combination of $CaCl_2$ and $MgCl_2$) at different concentrations for different treatment times, followed by constant VI. After the pretreatment, potato slices were removed from

solutions and blotted dried gently with paper towel. They were then fried for 4 min and centrifuged for 40 s.

Table 1 General experimental design.

Treatment	Soaking solution	Treatment time [min]	Concentration [10^3 ppm]	VI
Control	---	---	---	No
SVI	$MgCl_2$, $MgCl_2 + CaCl_2$	10, 30, 50	0, 5, 10, 15, 20, 50	Yes
NSVI	$MgCl_2$	10, 30, 50	15	Yes
S	$MgCl_2$	10, 30, 50	15	No

Control: no treatment; SVI: sonicated and vacuum impregnation; NSVI: no sonicated, only vacuum impregnation; S: sonicated only, no vacuum impregnation

4.4 Analytical Methods

4.4.1 Moisture Content

Fried chips were grounded manually. About 5 g samples were measured and put into a forced air oven at 105°C for 24 h (AACC, 1986). The moisture content of potato slices before and after the pre-treatment was also determined by using conventional oven, at 105 °C for 72 h (AACC, 1986). The test was performed in triplicate.

4.4.2 Oil Content

Total oil content of potato chips was determined by Soxtec System HT (Pertorp, Inc., Silver Spring, MD) extraction unit (Figure 8) with petroleum ether for 3 h (AACC, 1986). The oil content (d.b.) was calculated using the following equation. The analysis was carried out in triplicate.

$$OC(d.b.) = \frac{w_2 - w_1}{m_s} \quad (1)$$

where w_2 and w_1 are measured weights (g) of the holding cups after and before the extraction, respectively, and m_s (g) is the weight of the dried and ground potato chips in the thimble.

Figure 8 Soxtec System HT extraction unit used in the study.

4.4.3 Magnesium (Mg^{2+}) Content of Potatoes

About 0.5 g of ground dry samples was wet-digested through MARS 6 microwave digestion system (CEM Corporation) with 10ml nitric acid. After the completion of digestion, the resultant solution was diluted to 50 ml with deionized (DI) water, transferred to centrifuge tubes and stored in the refrigerator. The magnesium concentration was then quantified using the inductively coupled plasma mass spectrometer (iCAP 7000 Plus Series ICP-OES from Thermoscientific). Additionally, the teledyn Cetac Technologies ASX-560 Autosampler was used to eliminate sample

contamination and human error when sampling for the instrument. Measurements were taken in triplicate.

4.4.4 Specific gravity of Potatoes

The specific gravity of the potatoes was measured using the method described by Da Silva (2018). Each potato was weighed individually in a custom-made system that consisted of a thin wire basket placed inside a beaker with pure water on top of an analytical scale (0.01 g resolution, Sartorius, Wood Dale, IL). The weight in air (w_a) was obtained by placing the potato directly over the scale. The weight in water (w_w) was determined by submerging the potato in the pre-tared basket under water.

4.5 Product Quality Attributes

4.5.1 Texture

A puncture test was performed on both raw (pre-treated and untreated) and the fried samples using a Brookfield Texture Analyzer (TA-CT3 Texture Technologies Corporation, Scardale, NY), shown in Figure 9.

4.5.1.1. Raw potato firmness

The texture of raw potato slices was described in terms of their firmness. The firmness of raw potato slices was defined as the maximum force of penetration (Steffe, 1996). Since the texture of potato slices was softer and less fragile than chips, the cone-shape probe was selected to ensure the penetration into the raw potato structure. The sample was placed on the middle of a hollow fixture plate (2-point support - 0.018 m hollow TA-DEC Pot).

The probe used for the raw potato slices was a cone with the angel of 30 º(TA-17). The probe was lowered into the sample at 0.1 mm/s and a target distance of 3 mm was set to ensure the rupture of the slices.

4.5.1.*2*. Potato chips hardness

The property of hardness was used to describe the mechanical strength of fried potato chips, which was identified as the peak force recorded to break the fragile chips (Steffe, 1996).

The probe used for the chips was a spherical ball with the diameter of 12.7 mm (TA-18). The speed of the probe was 0.1 mm/s and the target distance were 4 mm to ensure the rupture of chips.

About 16 samples were tested for each treatment.

Figure 9 Brookfield Texture Analyzer used in this study.

4.5.2 Color

The color of potato chips was measured with a Hunter Lab Colorimeter LabScan XE (Hunter Associates Laboratory, Reston, VA). Six samples were randomly selected and measured each time. *L** (lightness-darkness), *a** (redness-greenness), and *b** (yellowness-blueness) values were recorded and used to evaluate the color of the chips. All readings were made at room temperature.

4.5.3 Degree of Shrinkage

Degree of diameter shrinkage of the samples was measured using an electronic digital caliper (MG Tool Co., NY), shown in Figure 10.

The degree of shrinkage (S) was calculated by:

$$S = \frac{D_o - D_t}{D_o} \times 100 \quad (2)$$

where D_o is the initial diameter [mm] of the raw sample and D_t is the diameter [mm] of the sample after frying at 165 ℃ for 4 min.

Figure 10 The digital steel caliper used in the study.

4.5.4 Density

The solid density was measured by placing manually grounded (using a mortar) pre-weighed and de-fatted samples in a compressed helium gas multi-pycnometer (Quantachrome & Trade, NY, USA). Solid density, ρ_s (kg/m^3) was determined by dividing the weight of the sample (Kg) by its solid volume (m^3). The solid volume (V_s) was calculated by the following equation. The test was conducted in triplicate.

$$V_s = V_c - V_r \cdot \left(\frac{P_1}{P_2} - 1\right) \quad (3)$$

where V_c is the volume of sample cell [cm^3]; V_r is the reference volume [cm^3]; P_1 is the pressure reading after pressurizing the reference volume, and P_2 is the pressure reading after including V_c. P_1 and P_2 values are given by the pycnometer. Micro cell was used in this study, giving the values of V_c and V_r 13.045 cm^3 and 7.379 cm^3, respectively.

The bulk volume was measured using the liquid displacement technique with 10% alcohol solution on a custom-built apparatus shown in Figure 11 (Da Silva & Moreira, 2008). Volumes before and after adding the samples were recorded. The bulk volume was determined by the difference between these two recorded volumes. Bulk density, ρ_b (kg/m^3) was determined by dividing the weight of the chips by its bulk volume.

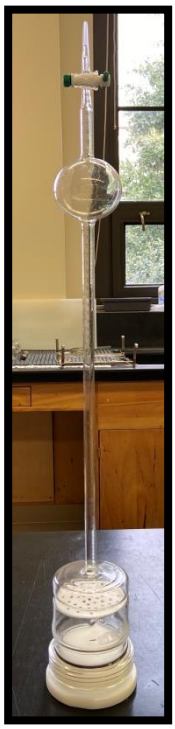

Figure 11 The custom-built apparatus for bulk density used in the study.

4.5.5 Porosity

The porosity ($\emptyset$) of potato chips was calculated as:

$$\emptyset = 1 - \frac{\rho_b}{\rho_s} \quad (4)$$

4.5.6 Sensory Evaluation

The sensory evaluation of produced potato chips was carried out by randomly selecting 50 panelists from Texas A&M University (faculty, students, and staff). The rate of each sample was based on its appearance, color, odor, texture, flavor, and overall quality of the chips. A nine-hedonic scale (Da Silva & Rosana G. Moreira, 2008) was used to score the samples, in which "1" represented the lowest quality and "9" represented the highest quality. Scores higher or equal to "5" were considered acceptable.

Three different treatments were evaluated: (1) Control (no treatment); (2) Chips sonicated (30 min) in 15000 ppm $MgCl_2$ (food grade), followed by vacuum impregnation; (3) Chips sonicated (30 min) in a combination of $MgCl_2$ and $CaCl_2$ (food grade; 750 mg/50 ml of each), followed by vacuum impregnation. The samples were coded with 3 randomly selected digital numbers, without any explanations about the treatment given to each coded sample. The purpose of this "blind" test was to avoid bias in the results. Samples were also sprinkled with some sea salt (about 10 mg/chip) before serving to the panelists. Potato chips were distributed to panelists with a cup of water to rinse their mouth and clean their taste buds in between samples. Panelists were also told to read the instructions carefully before starting the evaluation. The sensory evaluation sheet used in the study is shown in Figure 12.

Sensory Evaluation of Potato Chips

Instructions: Please evaluate each sample for each quality parameter and use the number scale below to mark the box which you feel best describes how you like/dislike the sample. Please take a sip of water in between to clean your palate. **Please Do Not compare the samples, evaluate them individually.** If you have any additional comments, please write them below each table.
Thank you for your patience and honest evaluation!

Dislike Extremely	Dislike very much	Dislike moderately	Dislike slightly	Neither like nor dislike	Like slightly	Like moderately	Like very much	Like Extremely
1	2	3	4	5	6	7	8	9

Sample #538	Appearance	Color	Odor	Texture	Flavor	Overall Quality

Comments:

Sample #352	Appearance	Color	Odor	Texture	Flavor	Overall Quality

Comments:

Sample #464	Appearance	Color	Odor	Texture	Flavor	Overall Quality

Comments:

Figure 12 The sensory evaluation sheet used in the study.

4.6 Mass Transfer during Pre-treatment

Four potato slices were numbered with a marker and their weights were measured using an analytical balance (Sartorius, 0.0001 g resolution, Wooed Dale, IL) before and after the pretreatment (SVI or NSVI). Seven concentrations (0, 1000, 5000, 10000, 15000, 20000, 50000 ppm) of soaking solutions ($MgCl_2$) were selected to analyze the mass transfer process. Five treatments were selected to do specific material

balance, including control (no treatment), sonicated in 15000 ppm $MgCl_2$ for 30 min, followed by VI (SVI15), non-sonicated (soaking) in 15000 ppm $MgCl_2$ for 30 min, followed by VI (NSVI15), sonicated in 50000 ppm $MgCl_2$ for 30 min, followed by VI (SVI50), and non-sonicated in 50000 ppm $MgCl_2$ for 30 min, followed by VI (NSVI50). The detailed experimental design is shown in Table 2.

Material balances assume that mass changes are mainly due to the exchange of moisture and Mg^{2+}, and based on that, other changes such as amylose leakage or loss of pectic substances were neglected.

a) Mass change (ΔM), [%]

$$\Delta M = \frac{m_t - m_0}{m_0} \times 100 \quad (5)$$

where m_0 and m_t are the initial and final mass of potato slices in wet basis [g], respectively.

b) Water loss (ΔWL), [%]

$$\Delta WL = \frac{w_{wo} \cdot m_o - w_{wt} \cdot m_t}{w_{wo} \cdot m_o} \times 100 \quad (6)$$

where m = mass in wet basis [g]; w_w = mass fraction of water (w/w); the subscripts *o* and *t* represent before and after the pretreatment, respectively.

c) Magnesium (Mg^{2+}) change (ΔMg), [%]

$$\Delta Mg = \frac{w_{mg_t} \cdot m_t - w_{mg_0} \cdot m_0}{w_{mgo} \cdot m_0} \times 100 \quad (7)$$

where m = mass in dry basis [g]; w_{mg} = mass fraction of Mg^{2+} (w/w).

Table 2 Experimental design for mass transfer during the pretreatment.

Treatment	Treatment time	$MgCl_2$ Concentration [10^3 ppm]
SVI	30	0
		1
		5
		10
		15
		20
		50
NSVI	30	0
		1
		5
		10
		15
		20
		50
Control	---	---

4.7 Kinetics of Moisture Loss and Oil Absorption

The kinetics of moisture loss and oil absorption was determined by varying the frying time from 20 sec to 720 sec. Potato slices were soaked in 15000 ppm $MgCl_2$ and treated with ultrasound simultaneously for 30 min, followed by VI (600 mm Hg pressure, vacuum time 10 min, restoration time 10 min). The pretreatment parameters (concentration, sonication time) were determined and selected mainly based on oil reduction, color, and texture of the crisps. After completion of the pretreatment, potato slices were deep-fat fried for 20 s, and then centrifuged for 40 s. Subsequently, oil content and moisture content of the chips were determined. Steps were repeated for the frying times of 40, 60, 80, 100, 120, 160, 180, 240, 300, 360, 480, 600, and 720 sec. Each assay was performed in triplicate.

4.8 Microstructural Changes

Scanning electron microscopy (SEM) was used to analyze the microstructural changes of potato samples. Potato slices without treatment (control) and treated with 15000 ppm $MgCl_2$ for 30 min sonication, followed by VI were both freeze-dried for 24 h. Potato chips produced from the above two treatments were also dried and de-fatted. Samples were mounted on aluminum stubs with a two-sided adhesive tape and sputter coated with gold using a sputter coater (TedPella Cressington 108). The microstructural changes were then observed using a scanning electron microscope (JEOL JCM-5000 Neoscope) at an accelerating voltage of 15 kV.

4.9 Statistical Analysis

JMP® Pro v. 13.1.0 statistic software was used to run statistical analysis in this study. One-way analysis of variance (ANOVA) was used to determine the overall significant difference. Tukey-Kramer's HSD test ($\alpha = 0.05$) was used to compare all pairs of means and Dunnett's test was used for comparing all other means with a control group's means. Figures were plotted using the 2010 MS Excel® software, as well as JMP® Pro v. 13.1.0 software.

CHAPTER V

RESULTS AND DISCUSSION

5.1 Effect of Pre-treatment on Oil Uptake

5.1.1 Effect of Sonication on Oil Content

Oil content of potato chips is an important indicator of their palatability and acceptability to consumers. Due to the growing tendency for healthy and low-fat foods, the oil content also becomes quality and safety factors affecting consumers' choice. Thus, it's crucial to investigate how applied techniques influencing oil absorption of potato chips. The effect of sonication on potato chips' oil uptake was evaluated by comparing sonicated samples (SVI) with non-sonicated samples (NSVI) treated with 15000 ppm $MgCl_2$ solution at 10, 30, and 50 min. The control samples showed an oil content of 0.3932 ± 0.0034 (g oil/g solid).

Figure 13 shows the oil content (d.b.) of potato chips treated with or without sonication process. The highest value of oil content was obtained under NSVI treatment at 10 min (0.3443 ± 0.0150 g oil/g solid), while the lowest oil uptake was obtained in sonicated samples at the treatment time of 50 min (0.2315 ± 0.0111 g oil/g solid). The statistical results in Table 3 show that the sonication treatment significantly reduced oil uptake at all times ($p < 0.05$). The highest oil reduction achieved by sonication was 20.06% at 50 min treatment time, compared with non-sonicated samples. In both SVI and NSVI treatments, statistical difference only exists between treatment time of 10/30 min, and 10/50 min. There was no evidence showing significant difference between 30 min and 50 min. One possible explanation for this result could be the concentration of

soaking solution used (15000 ppm) was not high enough to cause further structural changes in the potato tissue, which, in turn, impacting oil absorption of potato chips. This was confirmed later (see Section 5.1.3) where significant differences exist between 30 and 50 min of sonication times at the concentration of 20000 ppm. It was reported by previous researchers that the retention of metal ions by plant cell wall was dependent not only on cell wall structure, but also on starch content, as well as type and concentration of treated metal ions, thus limitation exists in the absorption of metal ions (Beveridge & Murray, 1976; Fortuna et al., 2013; Lester & Grusak, 1999; Muschitz, Riou, Mollet, Gloaguen, & Faugeron, 2015).

The significant oil reduction by the application of ultrasound was in accordance with those obtained by Karizaki et al. (2013), Dehghannya et al. (2016), Oladejo et al. (2017), Da Silva (2018), and Dehghannya and Abedpour (2018). The effectiveness of ultrasound treatment on oil reduction was reported mainly depending on different osmotic solutions used in the study and their concentrations, as well as the ultrasound conditions applied to the samples. The effect of sonication can be attributed to the cavitation or microchannels formed during the treatment, which enhances the delivery of magnesium ions into potato structure, thereby maintaining cell integrity, impairing oil absorption during deep-fat frying.

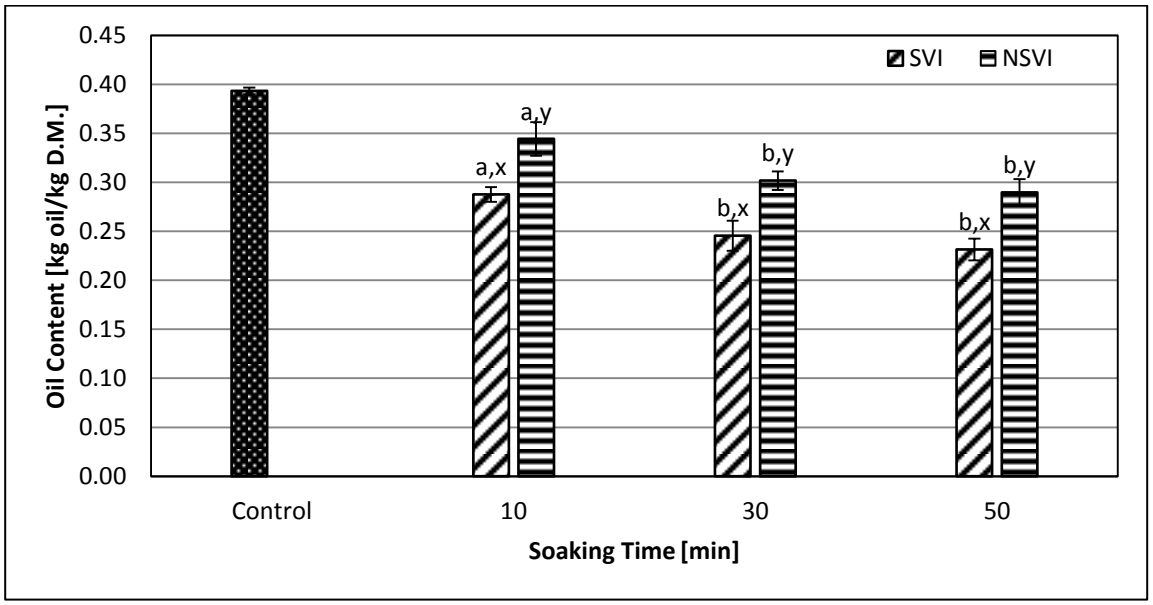

Figure 13 Oil content of potato chips treated with (SVI) and without sonication (NSVI) in 15000 ppm $MgCl_2$ for 10, 30 and 50 min, followed by VI (10000 ppm).

Table 3 Oil content of potato chips (fried for 4 min at 165 ℃) pre-treated with and without sonication in 15000 ppm $MgCl_2$ for 10, 30 and 50 min, followed by VI.

Soaking Time [min]	**Oil Content [g oil/g solid]**	
	SVI	**NSVI**
Control	0.3932 ± 0.0034	
10	${}_{x}0.2876 \pm 0.0074^{a}$	${}_{y}0.3443 \pm 0.0150^{a}$
30	${}_{x}0.2455 \pm 0.0153^{b}$	${}_{y}0.3017 \pm 0.0094^{b}$
50	${}_{x}0.2315 \pm 0.0111^{b}$	${}_{y}0.2896 \pm 0.0136^{b}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y} and column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$). SVI = sonication-assisted vacuum impregnation; NSVI = soaking without ultrasound treatment, followed by vacuum impregnation.

5.1.2 Effect of Vacuum Impregnation on Oil Content

The effect of vacuum impregnation on oil uptake was also evaluated by comparing samples pretreated with sonication-assisted vacuum impregnation (SVI) with those that did not undergo the vacuum impregnation steps (S). Significant difference ($p < 0.05$) was observed at the treatment time of 30 min (Table 4). However, there was no significant difference between SVI and S samples at 10 min and 50 min, indicating vacuum impregnation did not affect oil uptake significantly. The reason for this might be that samples sonicated for a short time did not create enough cavitation or microchannels in the structure, thus leaving less space for further ion impregnation. On the other hand, subjecting samples to ultrasound for too long might destroy the structure, thereby less effective on controlling oil absorption.

Figure 14 shows the comparison between three pretreatments: SVI, NSVI, and S. Different letters shown above each bar indicate that significant difference exists, in which a and b represent differences between treatment times within each pretreatment, whereas *x* and *y* represent differences between pretreatments within each treatment time. The experimental results affirm that the combination of sonication and vacuum impregnation (VI) has a synergistic effect on reducing oil uptake.

Table 4 Oil content of potato chips (fried for 4 min at 165 ℃) sonicated in 15000 ppm $MgCl_2$ for 10, 30 and 50 min, followed with or without vacuum impregnation (VI).

Soaking Time [min]	Oil Content [g oil/g solid]	
	SVI	S
Control	0.3932 ± 0.0034	
10	${}_{x}0.2876 \pm 0.0074^{a}$	${}_{x}0.3029 \pm 0.0171^{a}$
30	${}_{x}0.2455 \pm 0.0153^{b}$	${}_{y}0.2943 \pm 0.006^{a}$
50	${}_{x}0.2315 \pm 0.0111^{b}$	${}_{x}0.2467 \pm 0.0113^{b}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y} and column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$). SVI = sonication-assisted vacuum impregnation; S = sonication, followed soaking for 20 min.

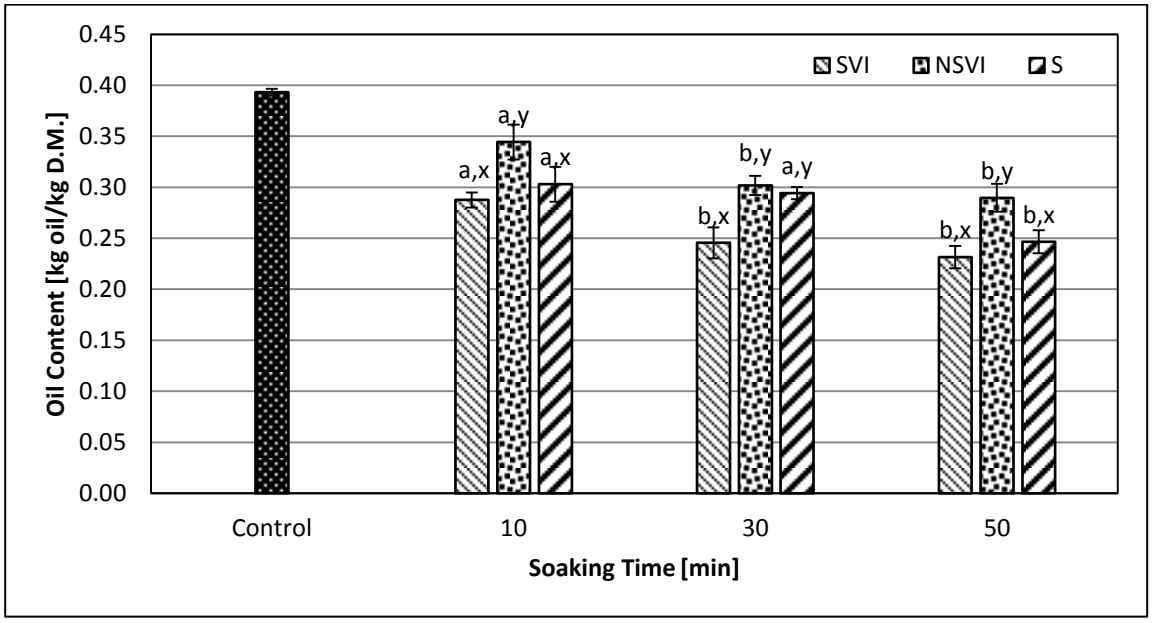

Figure 14 Comparisons of effects between SVI, NSVI, and S pretreatments on potato chips' oil content.

5.1.3 Effect of Sonication-assisted Vacuum Impregnation (SVI) on Oil Content

The oil content (g oil/g solid) of potato chips pretreated with SVI under three different sonication times (10, 30, 50 min) and four different $MgCl_2$ concentrations (5, 10, 15, 20 $\times 10^3$ ppm) is shown in Figure 15. The control samples represent potato chips not undergone any pretreatment, showing an oil content of 0.3932 ± 0.0034 (g oil/g solid).

Different letters shown above each bar indicate significant difference exists, in which *a-d* represent differences between concentrations within each sonication time, whereas *x-z* represent differences between sonication times within each concentration. It appears that low concentration of $MgCl_2$ (5000 ppm) cannot affect oil uptake significantly ($p > 0.05$) at all sonication times, while higher concentrations (10, 15, 20 $\times 10^3$ ppm) of $MgCl_2$ impeded oil uptake significantly ($p < 0.05$). The greatest oil reductions were observed from 10000 ppm to 15000 ppm, with the reduction of 22.56%, 24.72%, and 28.37% at the sonication time of 10, 30, and 50 min, respectively. At 15000 ppm $MgCl_2$ concentration, significant changes on oil uptake were only noticed from 10 to 30 min, but not 30 to 50 min, probably due to the rupture of potato cell structure. Same phenomenon was also observed at 10000 ppm of $MgCl_2$. This finding agrees with Karizaki's (2013) findings that the longer time of osmotic treatment and ultrasound-assisted osmotic treatment both caused damages to the potato cell structure.

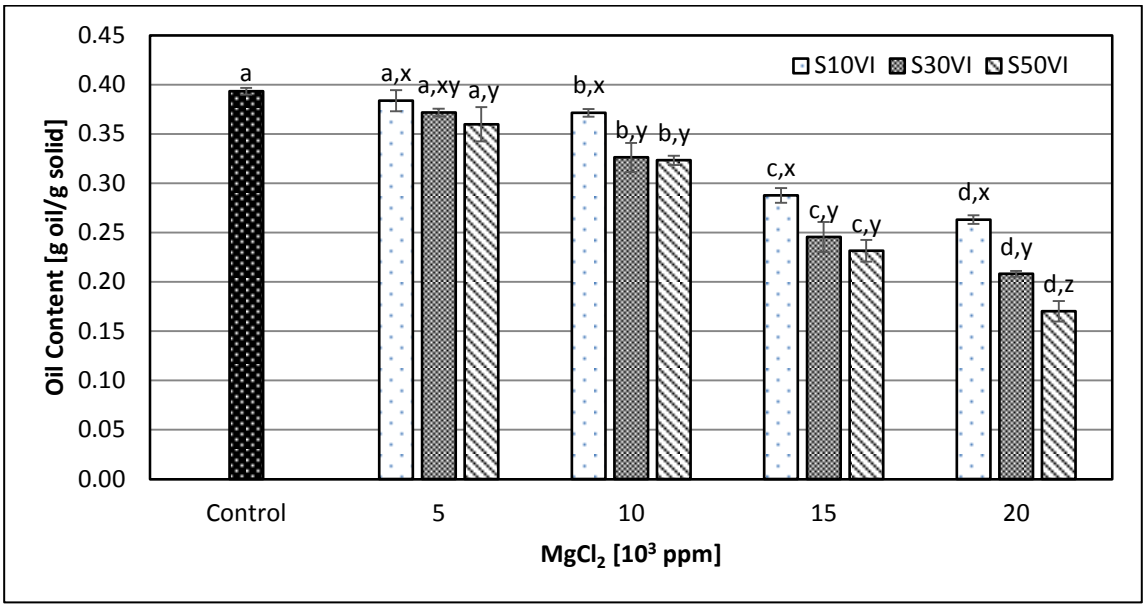

Figure 15 Oil content of potato chips pre-treated with sonication and vacuum impregnation under different sonication conditions (sonication time and concentration).

5.1.4 Effect of Combined Metal Ions (Mg^{2+}+Ca^{2+}) on Oil Content

Table 5 shows the comparison between using only $MgCl_2$ and using a combination of $MgCl_2$ and $CaCl_2$ as soaking solution on oil uptake of potato chips. The result showed no evidence that significant difference exists ($p > 0.05$) on oil content between two treatments.

Table 5 Oil content of potato chips (fried for 4 min at 165 ℃) sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI).

Soaking solution	**Oil Content [g oil/g solid]**
$MgCl_2$	0.2455 ± 0.0153^{a}
$MgCl_2$ + $CaCl_2$	0.2601 ± 0.0010^{a}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column[a,b] indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.2 Texture

5.2.1 Effect of Sonication-assisted Vacuum Impregnation on Potato Slices Firmness

The effect of different sonication times and $MgCl_2$ concentrations on the raw potato slice textures is presented in Figure 16. Potato slices without any treatment (control) showed a firmness of 0.96 ± 0.02 N. As concentration goes up, the firmness of pretreated potato slices firstly increased, due to the formation of Mg^{2+}-pectate bridges, resulting in the "firming effect", as well as the increase of cell turgor pressure (Haydar et al., 1980; Tajner-Czopek, 2003). However, after reaching the highest firmness value at 10000 ppm $MgCl_2$ concentration, an adverse effect was observed as the texture of raw potato slices became very leathery. The loss of rigidity was caused by osmotic gradient between cell turgor and outside solutions (Mauro et al., 2016). Thus, the pressure difference acts as a driving force, pushing intra and inter-cellular moisture migrating out of the slices to the surrounding solution. This behavior was also observed by Da Silva (2018) when using calcium chloride ($CaCl_2$) as the soaking solution. Turgor pressure is defined as the hydrostatic force inside the plant cell, which pushes the plasma membrane against the cell wall (Falk, Hertz, & Virgin, 1958). Changes of turgor pressure within the cell are affected by water loss and membrane integrity (Scanlon et al., 1996). The firmness of potato tissue was suggested exhibiting a positive correlation with cell turgor pressure (Nilsson et al, 1958). As evidenced by Scanlon and others (1996), both longitudinal and shear firmness was significantly higher under hypotonic condition than hypertonic condition, with 3% and 7% (w/v) mannitol solutions, respectively.

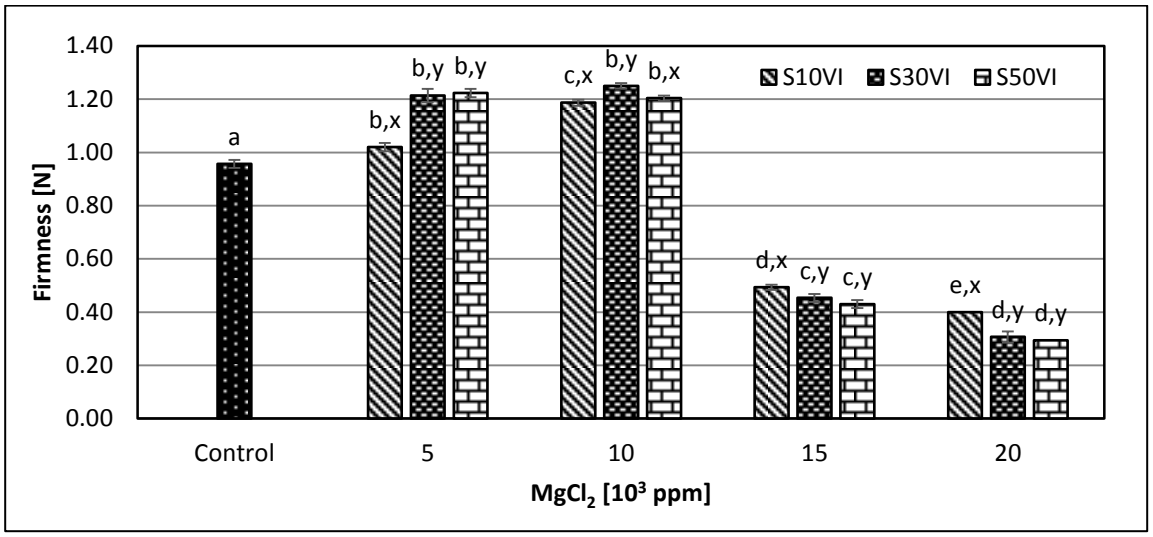

Figure 16 Firmness of potato slices pretreated with sonication-assisted vacuum impregnation for each sonication time and $MgCl_2$ concentration.

Table 6 shows the comparison of firmness between sonicating potato slices in only $MgCl_2$ solution and in a combination of $MgCl_2$ and $CaCl_2$ solutions (750 mg/50 ml of each). The result suggests that there was a statistical significance ($p < 0.05$) between the two treatments. Samples pretreated with both Mg^{2+} and Ca^{2+} showed a significant higher value on firmness, owing to the better performance of Ca^{2+} on "firming effect".

Table 6 Firmness of potato slices sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI)**.**

Soaking solution	Firmness [N]
$MgCl_2$	0.45 ± 0.02^{a}
$MgCl_2$ + $CaCl_2$	1.16 ± 0.01^{b}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column^{a,b} indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.2.2 Effect of Sonication-assisted Vacuum Impregnation on Potato Chips Hardness

Figure 17 presented the textual effect of the pretreatment on potato chips, indicated by the hardness, which is the peak force recorded to break the sample. The hardness of treated potato chips is a critical factor in its sensorial quality. It's also an indication of the crispness, influenced by various parameters such as sample thickness, frying method, measuring method, and raw material characteristics. The control sample which has not undergone any pretreatment showed a hardness value of 1.75 ±0.14 N. It was noted that the concentration of $MgCl_2$ started to make significant difference ($p < 0.05$) at 10000 ppm, compared to the control. Statistically significant ($p < 0.05$) effect of sonication time was observed at potato chips pretreated with 15000 and 20000 ppm $MgCl_2$. On the basis of the results obtained it is possible to state that the pretreatment led to increased hardness of potato chips and it was significantly affected by the concentration of $MgCl_2$ solution. The presence of Mg^{2+} contributed to the improvement of potato chips' texture. This was in agreement with those found by Tajner-Czopek (2003), who stated that the use of Ca and Mg salts can improve the texture quality of French fries, due to the formed bridges between divalent ions and pectic substances. Consequently, the firming effect was resulted from the decrease of pectin solubility, stabilizing the middle lamella and cell walls.

Table 7 shows the comparison between using only $MgCl_2$ and using a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) as soaking solution on the hardness of potato chips. As expected, the result indicates that the use of both Mg and Ca resulted in significantly harder potato chips. This is because Ca^{2+} was found a more

effective firming agent than other divalent ions (Mauro et al., 2016; Murayama et al., 2017; Tajner-Czopek, 2003).

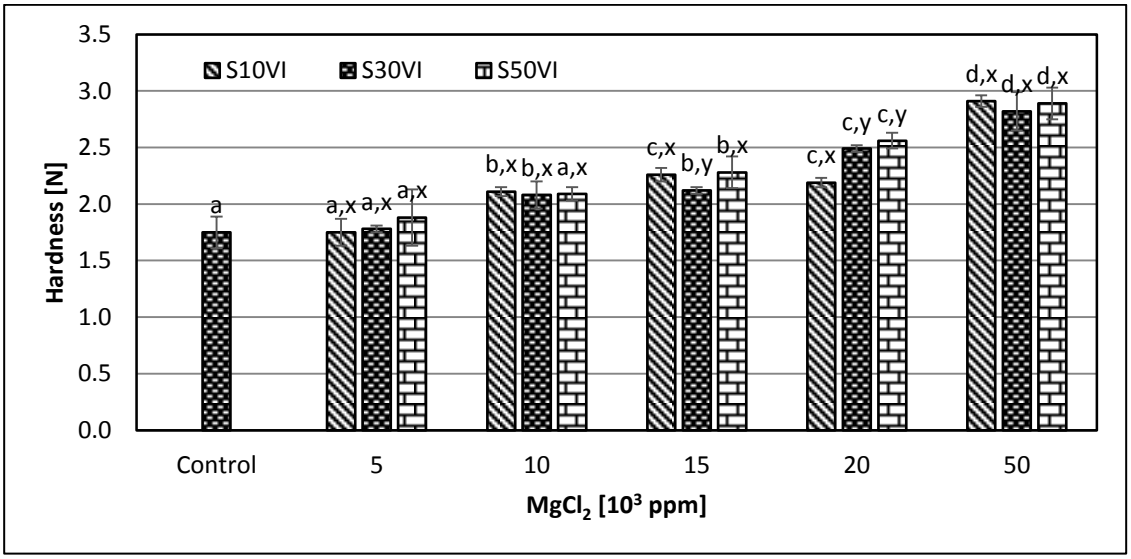

Figure 17 Hardness of potato chips pretreated with sonication-assisted vacuum impregnation for each sonication time and $MgCl_2$ concentration.

Table 7 Hardness of potato chips sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI).

Soaking solution	Hardness [N]
$MgCl_2$	2.12 ± 0.03^{a}
$MgCl_2$ + $CaCl_2$	2.35 ± 0.04^{b}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column^{a,b} indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.3 Color

5.3.1 Effect of Sonication-assisted Vacuum Impregnation on Color

As one of the most important contributors to consumers' preference on the food product, color can affect their decisions to buy the product at their first glances. The color parameter L^* is a measurement of lightness. The range of L^* values is between 0-100, in which higher L^* value indicates lighter potato chips, whereas lowers value indicates darker potato chips. Table 8 shows the effect of $MgCl_2$ concentration and sonication time during pretreatment on potato chips lightness. Pretreated potato chips showed significant difference with control samples, according to Dunnett's test ($p < 0.05$). Both concentration and sonication time were found significantly influencing the color parameter L^*. Samples sonicated for longer time appeared lighter, while the impregnated $MgCl_2$ made the chips darker. However, no statistical significance ($p > 0.05$) was found for the interaction of sonication time and concentration (Table 9). In other words, there was no different effect of sonication time depending on the concentration.

Table 8 Effect of $MgCl_2$ concentration and sonication time during pretreatment on color parameter *L**.

$MgCl_2$ [10^3 ppm]	***L****		
	10 min	**30min**	**50min**
Control	62.21 ±0.22		
5	$_{x}$61.28 ±0.49^{a}	$_{y}$63.94 ±1.12^{a}	$_{y}$64.37 ±1.03^{a}
10	$_{x}$58.73 ±1.42^{b}	$_{xy}$61.21 ±0.93^{a}	$_{y}$62.06 ±1.25^{ab}
15	$_{x}$55.82 ±0.96^{c}	$_{y}$58.08 ±0.34^{b}	$_{z}$60.43 ±1.01^{b}
20	$_{x}$46.27 ±1.89^{d}	$_{y}$50.38 ±1.12^{c}	$_{y}$51.34 ±1.34^{c}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y,z} and column^{a,b,c,d} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

Table 9 Two-way (Factorial) ANOVA results for color parameter *L** at different sonication times and concentrations.

Source	**Nparm**	**DF**	**Sum of Squares**	**F Ratio**	**Prob > F**
t	2	2	103.14354	39.4315	<.0001*
Conc	3	3	982.13221	250.3109	<.0001*
t*Conc	6	6	6.99382	0.8912	0.5168

The color parameter *a** is a measurement of redness to greenness of the sample, in which negative (-) values indicate sample is greener, whereas positive (+) values indicate sample is redder. It was observed that the pretreated potato chips showed significant difference with control samples, except for samples sonicated in 10000 ppm $MgCl_2$ for 10 min, according to Dunnett's test at the 95% level (Table 10). Significant effect ($p < 0.05$) was found for both factors (sonication time and concentration), as well as their interaction (Table 11). Samples sonicated for a longer and with higher $MgCl_2$ concentration appeared redder.

Table 10 Effect of $MgCl_2$ concentration and sonication time during pretreatment on color parameter *a**.

$MgCl_2$ [10^3 ppm]	*a**		
	10 min	**30min**	**50min**
Control	-2.34 ±0.20		
5	$_{x}$-2.04 ±0.08^{a}	$_{y}$-1.83 ±0.05^{a}	$_{z}$-1.35 ±0.08^{a}
10	$_{x}$-2.31 ±0.05^{b}	$_{y}$-1.72 ±0.05^{a}	$_{x}$-2.15 ±0.12^{b}
15	$_{xy}$-1.52 ±0.04^{c}	$_{x}$-1.41 ±0.06^{b}	$_{y}$-1.57 ±0.06^{a}
20	$_{x}$-0.93 ±0.05^{d}	$_{xy}$-0.64 ±0.06^{c}	$_{y}$-0.41 ±0.26^{d}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y,z} and column^{a,b,c,d} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

Table 11 Two-way (Factorial) ANOVA results for color parameter *a** at different sonication times and concentrations.

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
t	2	2	0.7974056	42.4403	<.0001*
Conc	3	3	9.6705000	343.1283	<.0001*
t*Conc	6	6	0.9476167	16.8116	<.0001*

The color parameter *b** is a measurement of blueness to yellowness, in which negative (-) values indicate sample is bluer, whereas positive (+) values indicate sample is yellower. Values of color parameter *b** of potato chips treated under different conditions were shown in Table 12. Significant effect ($p < 0.05$) was found for both factors (sonication time and concentration), as well as their interaction, indicating the value of color parameter *b** changes as a function of time and concentration (Table 13). The visual difference for overall color of potato chips treated with different concentrations was presented in Figure 18. This supported the statistical results that

potato chips treated with 20000 ppm $MgCl_2$ for 30 min appeared significantly ($p < 0.05$) darker, redder, and yellower in comparison with other samples, resulting in an unacceptable color for consumers.

The color development of fried potato chips was a result of Maillard reaction, a form of non-enzymatic browning, which was mainly affected by the content of reducing sugar and amino acids, as well as the frying conditions (Marquez & Anon, 1986). It was reported that blanching pretreatment and soaking in NaCl solution yielded lighter potato chips because of the leaching out of reducing sugars (Pedreschi et al., 2007; Pedreschi, Moyano, Kaack, & Granby, 2005). Therefore, the darker effect of the pretreatment in this study may be explained by the presence of divalent ions stabilized the cellular structure of potato tissues, thus obstructing browning reactants from leaching out to the surroundings (Patton, 1948).

Table 12 Effect of $MgCl_2$ concentration and sonication time during pretreatment on color parameter *b**.

$MgCl_2$ [10^3 ppm]	***b****		
	10 min	**30min**	**50min**
Control	15.48 ± 0.07		
5	$_{x}14.85 \pm 0.27^{a}$	$_{x}15.04 \pm 0.07^{a}$	$_{x}14.58 \pm 0.23^{a}$
10	$_{x}16.09 \pm 0.21^{b}$	$_{y}17.30 \pm 0.04^{b}$	$_{y}17.44 \pm 0.19^{b}$
15	$_{x}14.43 \pm 0.25^{a}$	$_{y}16.59 \pm 0.21^{c}$	$_{z}17.19 \pm 0.18^{b}$
20	$_{x}17.90 \pm 0.62^{c}$	$_{xy}18.33 \pm 0.32^{d}$	$_{y}19.18 \pm 0.19^{c}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y,z} and column^{a,b,c} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

Table 13 Two-way (Factorial) ANOVA results for color parameter *b** at different sonication times and concentrations.

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
t	2	2	10.847217	74.8141	<.0001*
Conc	3	3	63.608289	292.4743	<.0001*
t*Conc	6	6	7.926428	18.2231	<.0001*

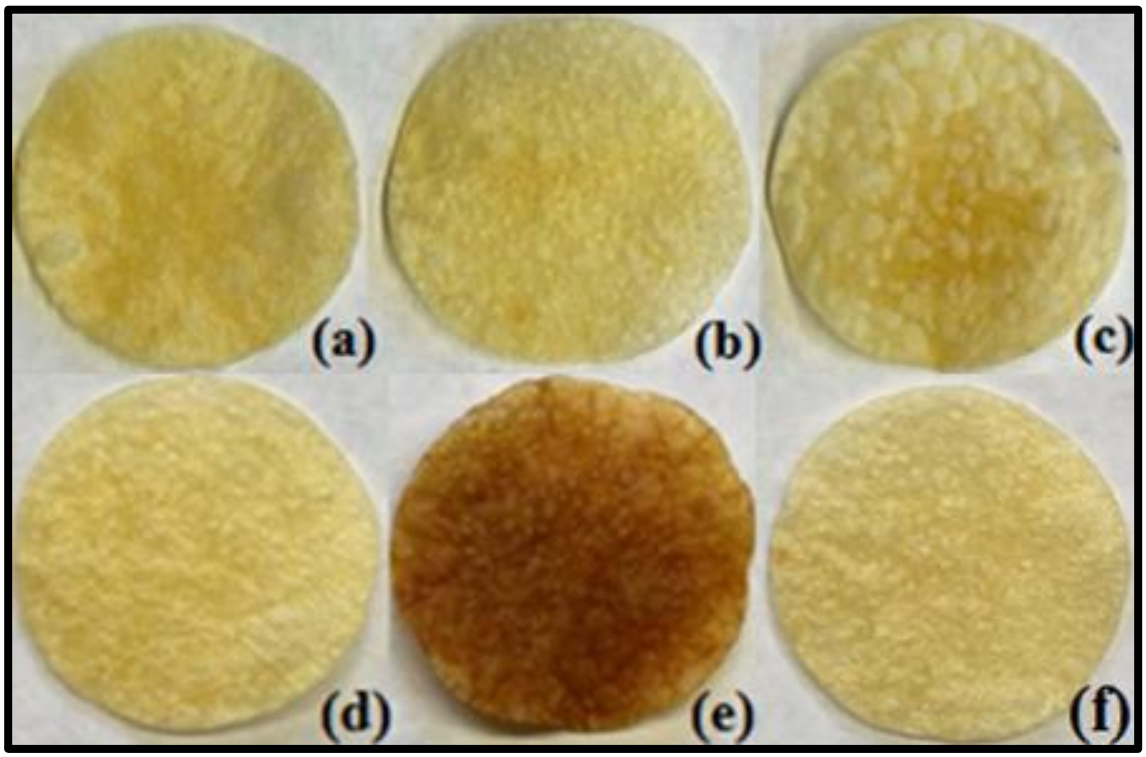

Figure 18 Potato chips sonicated in: (a) control (no treatment); (b) 5000 ppm; (c) 10000 ppm; (d) 15000 ppm; (e) 20000 ppm $MgCl_2$; (f) $MgCl_2$ + $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by VI.

5.3.2 Effect of Combined Metal Ions (Mg^{2+}+Ca^{2+}) on Color

The effect of using only Mg^{2+} and using combined Mg^{2+} and Ca^{2+} on color of potato chips was shown in Table 14. The results suggest that significant differences ($p < 0.05$) exist in *L** and *b** values for the two pretreatments. However, no significant difference ($p > 0.05$) was detected for *a** values between two pretreatments.

Table 14 Color parameters of potato chips sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ (750 mg/50 ml of each) and $CaCl_2$ for 30 min, followed by vacuum impregnation (VI)

Soaking solution	*L**	*a**	*b**
$MgCl_2$	58.08 ± 0.34^{a}	-1.41 ± 0.06^{a}	16.59 ± 0.21^{a}
$MgCl_2$ + $CaCl_2$	55.07 ± 1.11^{b}	-1.30 ± 0.05^{a}	19.03 ± 0.08^{b}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column^{a,b} indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.4 Shrinkage

5.4.1 Effect of Sonication-assisted Vacuum Impregnation on Shrinkage

Table 15 showed the diameter shrinkage (%) of both control and pretreated samples. It was observed that the pretreated potato chips showed significant difference ($p < 0.05$) with control samples on shrinkage, except for samples sonicated in 5000 ppm $MgCl_2$ for 10 min, according to Dunnett's test at the 95% level. Significant effect ($p < 0.05$) was also found for both factors (sonication time and concentration), as well as their interaction, indicating the value of shrinkage changes as a function of time and concentration (Table 16). The results showed that the shrinkage increases with $MgCl_2$ concentration and sonication time.

Table 15 Effect of $MgCl_2$ concentration and sonication time during pretreatment on shrinkage.

$MgCl_2$ [10^3 ppm]	**Shrinkage [%]**		
	10 min	**30min**	**50min**
Control	5.05 ±0.20		
5	$_{x}5.09 \pm 0.13^{a}$	$_{y}5.50 \pm 0.05^{a}$	$_{y}5.49 \pm 0.12^{a}$
10	$_{x}5.32 \pm 0.04^{a}$	$_{y}7.34 \pm 0.24^{b}$	$_{y}7.86 \pm 0.28^{b}$
15	$_{x}10.71 \pm 0.20^{b}$	$_{x}12.03 \pm 0.77^{c}$	$_{y}14.82 \pm 0.57^{c}$
20	$_{x}11.35 \pm 0.14^{c}$	$_{y}13.31 \pm 0.15^{d}$	$_{z}18.93 \pm 0.17^{d}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y,z} and column^{a,b,c,d} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

Table 16 Two-way (Factorial) ANOVA results for diameter shrinkage at different sonication times and concentrations.

Source	**Nparm**	**DF**	**Sum of Squares**	**F Ratio**	**Prob > F**
t	2	2	81.43415	433.2628	<.0001*
Conc	3	3	524.47683	1860.287	<.0001*
t*Conc	6	6	48.91623	86.7514	<.0001*

5.4.2 Effect of Combined Metal Ions (Mg^{2+}+Ca^{2+}) on Shrinkage

The effect of using only Mg^{2+} and using combined Mg^{2+} and Ca^{2+} on shrinkage of potato chips was shown in Table 17. The results suggest that no significant differences ($p > 0.05$) exist for the two pretreatments.

Table 17 Diameter Shrinkage (%) of potato chips sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI).

Soaking solution	Shrinkage [%]
$MgCl_2$	12.03 ± 0.77^{a}
$MgCl_2$ + $CaCl_2$	12.35 ± 0.61^{a}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column[a,b] indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.5 Density and Porosity

5.5.1 Effect of Sonication-assisted Vacuum Impregnation on Density and Porosity

Bulk density and solid density were measured to calculate the porosity of potato chips, which has been correlated with the oil uptake (Aguilar, Anzaldúa-Morales, Talamás, & Gastélum; Bouchon et al., 2003; Pinthus, Weinberg, & Saguy, 1995). The results of control sample and samples treated with different concentration of $MgCl_2$ are presented in Table 18. There was statistically significant ($p < 0.05$) effect of concentration on bulk density and solid density. The results also suggest that samples treated with $MgCl_2$ at concentrations above 10000 ppm showed a significant ($p < 0.05$) lower value on porosity, which was consistent with the effect of the concentration on oil reduction presented in Section 5.1.3. The lowest porosity was obtained at 20000 ppm, with a value of 0.66 ± 0.01.

Table 18 Effect of pretreatment on potato chips' bulk density, solid density, and porosity.

$MgCl_2$ [10^3 ppm]	Bulk Density [kg/m^3]	Solid Density [kg/m^3]	Porosity
Control	365.15 ± 7.12^{a}	1419.26 ± 4.13^{a}	0.74 ± 0.01^{a}
5	380.47 ± 4.39^{ab}	1418.81 ± 4.85^{a}	0.73 ± 0.00^{ab}
10	396.06 ± 7.37^{b}	1428.17 ± 11.58^{a}	0.72 ± 0.00^{b}
15	430.07 ± 7.95^{c}	1408.67 ± 8.34^{a}	0.69 ± 0.01^{c}
20	438.95 ± 6.40^{c}	1289.98 ± 4.66^{b}	0.66 ± 0.01^{d}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column^{a,b,c,d} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

5.5.2 Effect of Combined Metal Ions (Mg^{2+}+Ca^{2+}) on Density and Porosity

There was no significant difference ($p > 0.05$) on bulk density, solid density, and porosity between potato chips treated with Mg^{2+} and a combination of Mg^{2+} and Ca^{2+} (Table 19).

Table 19 Densities and porosity of potato chips sonicated in only $MgCl_2$ and in a combination of $MgCl_2$ and $CaCl_2$ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI).

Soaking solution	Bulk Density, ρ_b [kg/m^3]	Solid Density, ρ_s [kg/m^3]	Porosity, $\emptyset$
$MgCl_2$	430.07 ± 7.95^{a}	1408.67 ± 8.34^{a}	0.69 ± 0.01^{a}
$MgCl_2$ + $CaCl_2$	439.38 ± 2.59^{a}	1415.82 ± 5.69^{a}	0.69 ± 0.00^{a}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same column^{a,b} indicate significant difference ($p < 0.05$) according to one-way ANOVA test.

5.6 Sensory Evaluation

The sensory analysis was conducted based on six quality attributes, including appearance, color, odor, texture, flavor, and overall quality. Samples undergone three different treatments (Control, $MgCl_2$, $MgCl_2$ + $CaCl_2$) were served to the panelists (Figure 19). The resulting scores from 50 panelists were shown in Table 20. Significant difference ($p < 0.05$) was only observed in the sensory attribute "texture". Potato chips treated with combined $MgCl_2$ and $CaCl_2$ received a significant higher score than the other two treatments. They were commented as "crisper" by some panelists, which was in agreement with the statistical results regarding to "hardness" presented in Section 5.2.2. The quality attribute "flavor" was the least preferred with the lowest mean scores among all three treatments, in which control samples received the lowest score of 6.25 ± 0.89 and they were commented as "oily and salty". However, all samples showed mean scores above 5, indicating they can be considered acceptable. No bitterness was sensed for pretreated potato chips, which was disagree with those obtained by Yang and Lawless (2005) who found that the use of $CaCl_2$ and $MgCl_2$ brought adverse effect of bitterness and off flavor. In summary, the pretreatments produced potato chips with improved texture and retained sensory quality.

Table 20 Sensory evaluation results for potato chips processed under different pretreatments

Sample	Appearance	Color	Odor	Texture	Flavor	Overall
Control	7.11 ± 0.78^{a}	6.91 ± 0.88^{a}	7.69 ± 1.16^{a}	6.92 ± 1.31^{a}	6.25 ± 0.89^{a}	7.14 ± 0.86^{a}
$MgCl_2$	7.89 ± 1.03^{a}	7.09 ± 0.78^{a}	7.62 ± 1.24^{a}	7.77 ± 1.22^{a}	6.88 ± 1.64^{a}	7.71 ± 0.99^{a}
$MgCl_2 + CaCl_2$	7.78 ± 0.83^{a}	7.73 ± 1.04^{a}	7.54 ± 1.07^{a}	8.08 ± 0.95^{b}	7.13 ± 1.25^{a}	7.86 ± 0.76^{a}

Values are presented as means of replicates, followed by standard deviations. Different letters within the same column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).

Figure 19 Potato chips (fried for 4 min at 165 ℃) served in sensory evaluation.

5.7 Mass Transfer during Pre-treatment

Figure 20 shows the overall mass change (%) of potato slices under SVI (sonication-assisted vacuum impregnation) and NSVI (non-sonication-assisted vacuum impregnation) pretreatments in various concentrations of $MgCl_2$ with the treatment time of 30 min. The positive mass change values indicate potato slices gained mass during the pretreatment, while the negative values indicate mass loss. As observed from the graph, the mass of potato slices increased in both treatments at lower concentrations, and then

started to decrease from a certain point. The mass gain at lower concentration (≤10000 ppm) of $MgCl_2$ and the mass loss at higher concentrations were reflected statistically in Section 5.2.1 as the firmness of raw potato texture firstly increased to a maximum value around 10000 ppm and then decreed to a lower value. The changes in mass of pretreated potato slices were due to the differences in osmotic pressure within the cell (cell turgor) and outside solutions ($MgCl_2$ concentrations), in which lower concentrations created hypotonic conditions, and higher concentration resulted in hypertonic conditions (Lin & Pitt, 1986).

The experimental data were fitted to the 4-parameter logistic model, demonstrated as solid (SVI) and dash (NSVI) curves in Figure 20. It was shown that potato slices treated with and without sonication responded differently in mass change with increasing $MgCl_2$ concentration. The mass change value of samples without ultrasound treatment decreases continuously with the $MgCl_2$ concentration until reaching a plateau at around 45000 ppm. However, for potato slices treated with ultrasound, the mass change value was almost constant at concentrations below 10000 ppm while decreasing sharply from 10000 ppm to 20000 ppm. The final equilibrium of mass change for both treatments can be attributed to the deposit of the metal ions in the microchannel (SVI) or sample surface (NSVI) which impede further diffusion of water and solutes (Oladejo et al., 2017).

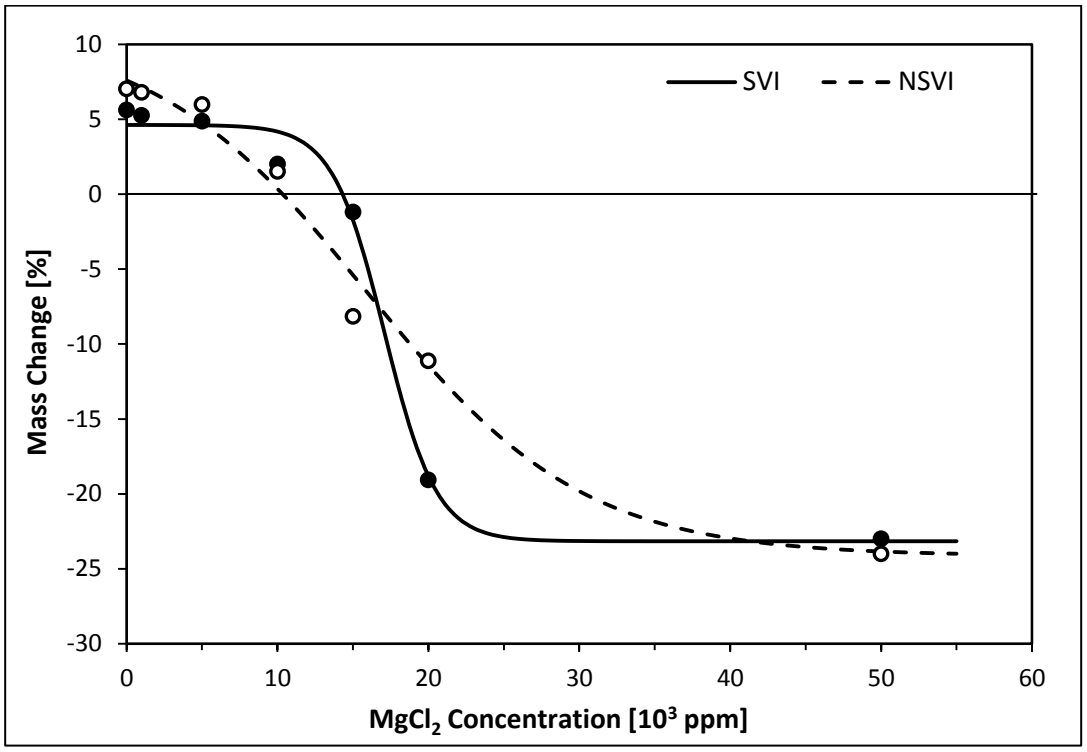

Figure 20 Mass change (%) of potato slices pretreated (SVI and NSVI) in different concentrations of $MgCl_2$ with the treatment time of 30 min.

Equation (8) presents the logistic model used in this study, where ΔM represents the % of mass gain (positive values) or loss (negative values). Its parameters are shown on Table 21:

$$\Delta M(\%) = d + \frac{a-d}{1+e^{b(C-f)}} \quad (8)$$

where: b is curve slope, f is the inflection point, a is the lower asymptote, d is the upper asymptote; and C is the concentration [10^3 ppm].

Table 21 Parameter estimates for the logistic model.

Parameter	Estimate	Std Error	Lower 95%	Upper 95%	R^2
Non-Sonicated-Assisted Vacuum Impregnation					**0.99**
Curve Slope (*b*)	-0.141	0.057	-0.253	-0.029	
Inflection Point (*f*)	15.969	2.407	11.251	20.687	
Lower Asymptote (*a*)	-24.143	2.234	-28.507	-19.784	
Upper Asymptote (*d*)	10.976	5.008	1.158	20.793	
Sonicated-Assisted Vacuum Impregnation					**0.99**
Curve Slope (*b*)	-0.577	0.114	-0.800	-0.355	
Inflection Point (*f*)	17.098	0.537	16.044	18.151	
Lower Asymptote (*a*)	-23.164	1.458	-26.021	-20.304	
Upper Asymptote (*d*)	4.617	0.763	3.122	6.112	

The point of inflection can be used as a predictor for the $MgCl_2$ solution concentration where there should not be gain or loss of mass (sample and solution has same osmotic pressure) during that specific processing time (Da Silva, 2018). Table 21 shows that the point of inflection for the SVI samples happened at a higher concentration of $MgCl_2$ on the sonication process (f =17 x 10^3 ppm) than in the NSVI process (f = 16 x 10^3 ppm). Both estimated values were closer to the experimental concentration of 13 x 10^3 ppm of $MgCl_2$ where the texture (Figure 16) of the samples after treatment was similar to the samples that did not undergo the $MgCl_2$ treatment. According to two-way ANOVA results presented in Table 22, the effects of both factors concentration and pretreatment type (SVI or NSVI) on mass transfer were significant ($p < 0.05$). The interaction of both effects was also found significant ($p < 0.05$), indicating that the effect of sonication differs for different concentrations. In other words, there was different effect of vacuum impregnation with or without the assistance of sonication depending on the concentration.

The result suggests that NSVI samples reached the osmotic-pressure equilibrium sooner than SVI samples, which is contrary to those reported by Da Silva (2018). This is probably due to the incorporation of the vacuum impregnation process after sonication, which may have affected the mass transfer process.

Table 22 Two-way (Factorial) ANOVA results for the effects of $MgCl_2$ concentration and pretreatments on mass transfer.

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Conc	6	6	5239.9147	15889.76	<.0001*
SVI/NSVI	1	1	1.5604	28.3904	<.0001*
Conc*SVI/NSVI	6	6	132.4011	401.4992	<.0001*

Table 23 and 24 present experimental data concerning magnesium content of untreated (control) and pretreated (SVI and NSVI) potato slices and fried potato chips, respectively, in 15000 and 50000 ppm $MgCl_2$ for 30 min treatment time. Pretreated samples showed a significant ($p < 0.05$) higher amount of magenesium content than control samples according to Dunnett's test. This affirms the effiency of the pretreatment on magnesium (Mg^{2+}) impregnation into potato tissues. This is in agreement with a previous study conducted by Tajner-Czopek (2003) who also found that the presence of Ca^{2+} or Mg^{2+} in blanching solutions significantly helped the retention of pectic substances in French fries by binding with them in the cell walls and the middle lamella. The statistical results also show that magnesium content was significantly ($p < 0.05$) higher when treated with higher concentration of $MgCl_2$. As expected, the sonicated

potato slices and chips showed higher magnesium contents than non-sonicated samples, indicating the ultrasonic waves facilitated the delivery of Mg^{2+}.

Note that there was a small percentage loss of magnesium content after frying (Tables 23 and 24). For the SVI process, potato slices impregnated with 15000 ppm $MgCl_2$ solution lost around 7% of magnesium during frying and those treated with the 50000 ppm $MgCl_2$ concentration lost about 8%. However, for the slices that were not sonicated (NSVI), there were about 7% and 17% loss of magnesium for the samples impregnated with a concentration of 15000 and 50000 ppm, respectively. It seems that without sonication, the VI samples in 50000 ppm $MgCl_2$ concentration solution loses more magnisium after frying than those that are sonicated and then VI.

The summary of mass transfer results including mass change (ΔM), water loss change (ΔWL), and magnesium uptake change (ΔMg) is presented in Table 25. According to ANOVA, there were significant differences ($p < 0.05$) between the two pretreatments (SVI and NSVI) on the percentage of magnesium uptake change at both concentrations (15000 and 50000 ppm). The effect of sonication treatment was in accordance with those reported by Karizaki (2013) that applying ultrasound to potato samples immersed in osmotic solutions increased the rate of diffusion and resulted in higher water loss and solid gain. $MgCl_2$ concentration significantly ($p < 0.05$) affected mass change, water loss, and magnesium uptake. Potato slices with higher ΔML and ΔWL were obtained when higher concentration of $MgCl_2$ was used. As expected, the changes in magnesium content (ΔMg) was higher for the ther higher concentration (50000 ppm) of $MgCl_2$ for the SVI and NSVI treated samples. SVI treated samples

absorbed 29% more magnesium than the NSVI samples for the 15000 ppm solution concentration. For the 50000 ppm solution, the SVI samples absorbed 15% more Mg than the NSVI ones. It can also be seen that the mass changes were consistent with the water loss values, suggesting potato slices gained or lost mass mainly due to the moisture migrations.

Table 23 Magnesium content (Mg^{2+}) of potato slices without pretreatment (control) or pre-treated with and without sonication in 15000 ppm and 50000 ppm $MgCl_2$ for 30 min, followed by VI.

$MgCl_2$ [10^3 ppm]	**Magnesium Content [mg/100g D.M.]**	
	SVI	**NSVI**
Control	75.67 ± 2.08	
15	$_{x}461.7 \pm 3.51^{a}$	$_{y}421.33 \pm 3.21^{a}$
50	$_{x}1261.33 \pm 4.51^{b}$	$_{y}1156.00 \pm 6.56^{b}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y} and column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$). Control = no treatment; SVI = sonication-assisted vacuum impregnation; NSVI = soaking without ultrasound treatment, followed by vacuum impregnation.

Table 24 Magnesium content (Mg^{2+}) of potato chips (fried for 4 min at 165 ℃) without pretreatment (control) or pre-treated with and without sonication in 15000 ppm and 50000 ppm $MgCl_2$ for 30 min, followed by VI.

$MgCl_2$ [10^3 ppm]	**Magnesium Content [mg/100g D.M.]**	
	SVI	**NSVI**
Control	66.67 ± 3.06	
15	$_{x}429.67 \pm 3.06^{a}$	$_{y}392.33 \pm 3.51^{a}$
50	$_{x}1165.33 \pm 5.51^{b}$	$_{y}954.67 \pm 6.11^{b}$

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y} and column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$). Control = no treatment; SVI = sonication-assisted vacuum impregnation; NSVI = soaking without ultrasound treatment, followed by vacuum impregnation.

Table 25 Mass change (ΔM), water loss (WL), and magnesium uptake (ΔMg) during the pretreatments (SVI and NSVI) in 15000 ppm and 50000 ppm $MgCl_2$ for the treatment time of 30 min.

	$MgCl_2$ [10^3 ppm]	**SVI**	**NSVI**
ΔM [%]	15	$_{x}$-1.30 ± 0.37^{a}	$_{y}$-7.78 ± 0.47^{a}
	50	$_{x}$-23.13 ± 0.44^{b}	$_{x}$-23.88 ± 0.49^{b}
ΔWL [%]	15	$_{x}$2.27 ± 0.37^{a}	$_{y}$5.99 ± 0.30^{a}
	50	$_{x}$26.02 ± 0.48^{b}	$_{x}$25.51 ± 0.79^{b}
ΔMg [%]	15	$_{x}$526.63 ± 5.72^{a}	$_{y}$373.34 ± 30.29^{a}
	50	$_{x}$1378.74 ± 49.48^{b}	$_{y}$1165.75 ± 24.89^{b}

Values are presented as means of three replicates, followed by standard deviations. Different letters within the same row^{x,y} and column^{a,b} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA test ($\alpha = 0.05$). SVI = sonication-assisted vacuum impregnation; NSVI = soaking without ultrasound treatment, followed by vacuum impregnation.

5.8 Kinetics

5.8.1 Moisture Content

The kinetics of dehydration and oil uptake of pretreated potato chips during deep-fat frying were analyzed. Prior to frying, potato slices were sonicated in 15000 ppm $MgCl_2$ solution for 30 min, followed by vacuum impregnation. Pretreated samples were then fried at 165 ℃ from 0 s to 720 s. The moisture content of pretreated potato slices was 3.95 ±0.14 (d.b.). The moisture loss profile shown in Figure 21, exhibits a classical drying behavior described by Garayo and Moreira (2002). Based on the fact that the dehydration in food during frying follows the typical moisture diffusion mechanisms, the experimental data was fitted to the exponential model described by the following equation:

$$\mathrm{MC_{db}} = (\mathrm{M_0} - \mathrm{M_e})\exp(\frac{\pi^2 D_e t}{4a^2}) + \mathrm{M_e} \quad (9)$$

where MC_{db} is the moisture content in dry basis; M_0 and M_e are the initial and equilibrium moisture content (d.b), respectively; *t* is the frying time [s]; *a* is half of the thickness of potato slices [m], and D_e is the moisture diffusion coefficient [m^2/s] which describes the drying rate during deep-fat frying.

The value of moisture diffusion coefficient obtained from the predictive exponential model was $D_e = 2.856 \times 10^{-8}$ m^2/s. The typical dehydration during frying can be distinguished into three periods. The first period is the heat-up stage where the wet material absorbs heat from the surrounding hot oil until moisture starts to evaporate from the food. The second period is the constant rate stage, during which the rate of moisture loss is controlled by the rate of heat transfer. This period continues until the product surface is no longer wetted. The constant rate period was not detected in Figure 21, probably because the dehydration rate was very fast at the beginning. The last period is the falling rate stage. The drying process starts to slow down until reaches equilibrium. As expected, the moisture decreased drastically especially in the early stages of frying (0-60 s). This was in accordance with those observed by Gamble and others (1987) that moisture loss of potato chips frying under high temperature consists of an initial rapid fall, followed by a continuous drying period.

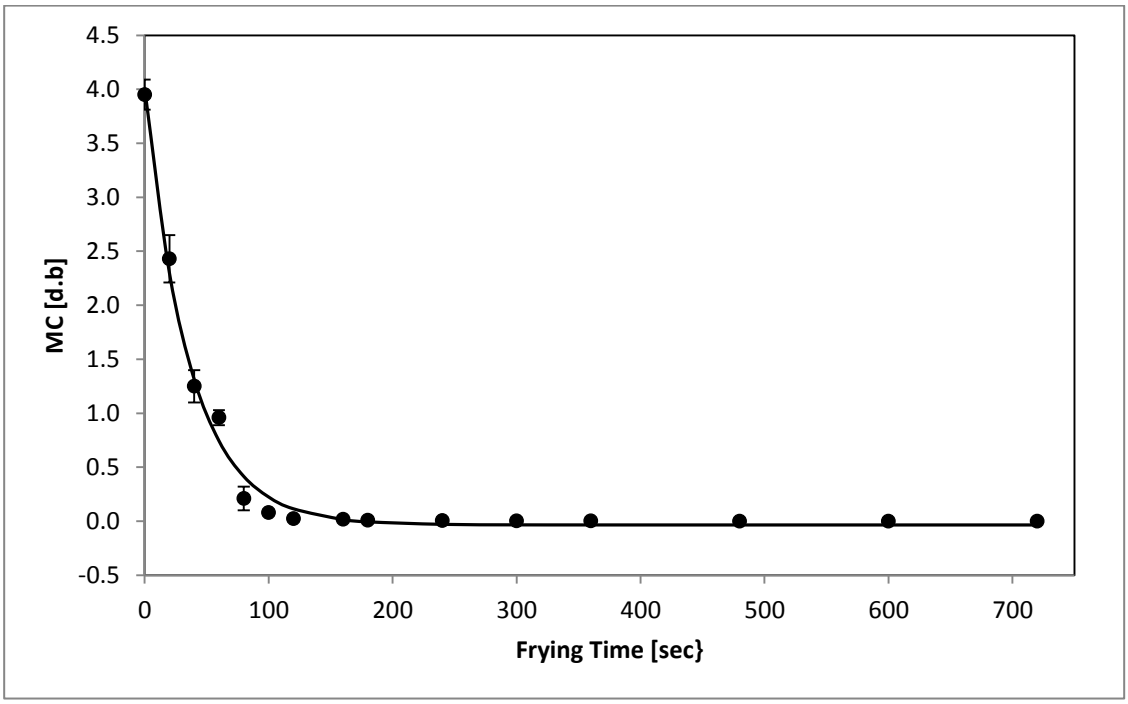

Figure 21 Moisture loss of potato chips fried at 165°C pretreated with SVI in 15000 ppm $MgCl_2$ solution for 30 min. (symbols are experimental values and line the predicted curve using Eq.(9)).

5.8.2 Oil Content

Figure 22 presented oil absorption profile of potato chips pretreated with sonication in 15000 ppm $MgCl_2$ solution for 30 min, followed by vacuum impregnation. Pretreated samples were fried at 165 ℃ from 0 s to 720 s. The fractional conversion kinetic model described by Chen & Ramaswamy (2002) was rearranged and used to fit the experimental data of oil content (Eq. 8):

$$\mathrm{OC_t} = \mathrm{A} \cdot \exp(-kt)(OC_0 - OC_e) + OC_e \qquad (10)$$

where OC_t is the oil content in dry basis; OC_0 and OC_e are the initial and equilibrium oil content (d.b), respectively; t is the frying time [s]; k is the rate constant [s^{-1}] and A is the regression coefficient.

The predictive model gave the values of constant rate k = 0.010 s^{-1} and A = 1.010 kg/kg solid. As demonstrated in Figure 22, the oil content increased exponentially in the early stages of frying. After that, the rate of oil uptake slowed down until a plateau was reached. This was closely related to the moisture loss behavior. Similar results were also reported by Gamble et al. (1987) and Moreira (2014). The pretreatment did not change the typical behavior of oil absorption during deep-fat frying, compared the curve to those obtained in similar kinetics studies (Moyano & Pedreschi, 2006; Pedreschi et al., 2007).

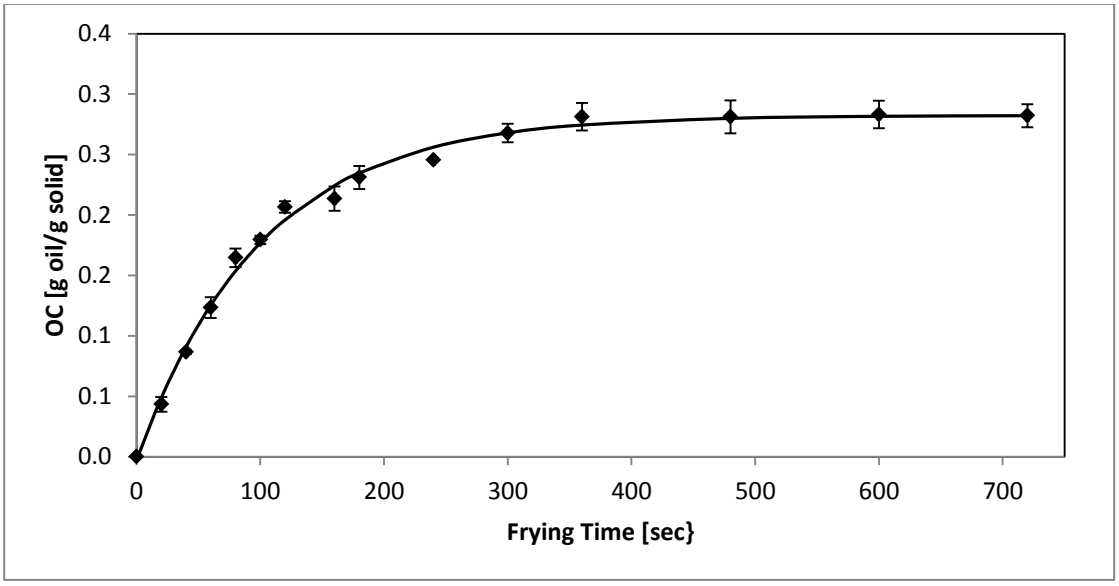

Figure 22 Oil absorption for potato chips fried at 165°C pretreated with SVI in 15000 ppm $MgCl_2$ solution for 30 min. (symbols are experimental values and line the predicted curve using Eq.(10)).

5.9 Microstructural Changes

5.9.1 Raw Potato Slices

The microstructure of fresh potato slices was examined by SEM, which revealed a normal morphology of potato cells. As shown in Figure 23a-d, the cell structure was well arranged in the control and pretreated (SVI – 15000 ppm of $MgCl_2$ and 30 min sonication) potato slices and cell walls, middle lamella, as well as starch granules were clearly presented. This kind of compact hexagonal cells in potato tissues has been previously observed by Bordoloi, Kaur, and Singh (2012) using light (LM) and confocal scanning electron microscopy (CSLM). A greater degree of cell separation and more collapsed cell structure were observed in untreated samples as compared to pretreated samples (Figure 23a), while the SEM micrograph of pretreated potato slices (Figure 23c) revealed a well-integrated cellular structure. No cell distortion was observed due to ultrasound treatment as described by Fernandes, Gallão, and Rodrigues (2009) for pineapples. Unevenly distributions of intact round-shape starch granules were observed in both untreated and treated samples. The SEM micrographs under higher magnification (Figure 23b, d) revealed more details concerning the structure of cell walls and the middle lamella. It is perceived that the thickness of the middle lamella was greater in samples pretreated with sonication-assisted vacuum impregnation (SVI) than control samples, evidencing the formation of cross-bridges between magnesium ions and pectic substances. The thicker middle lamella is responsible for stronger cell adhesions, thus reducing cell separation. Similar observations were reported by Moreno et al. (2004) for Chilean papaya treated with VI and OD. They attributed the thickening of middle

lamella to the formation of a polymeric compound due to interactions between middle lamella pectins and osmotic solutes. The presence of this polymeric compound or the concentrated solutes on sample's surface could help explain the firming effect brought by pretreatment (Moreno, Chiralt, Escriche, & Serra, 2000).

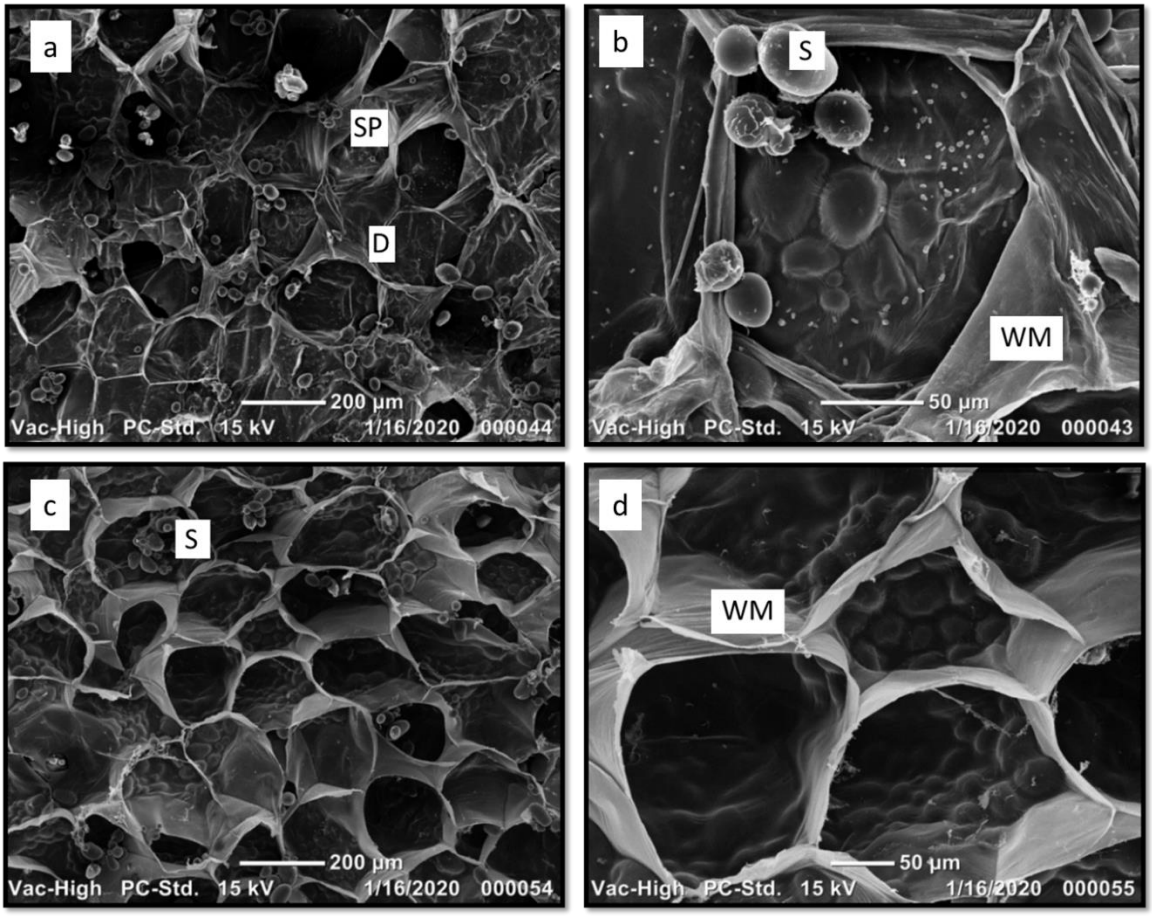

Figure 23 SEM images of (a) control (200 **µm**); (b) control (50 **µm**); (c) pretreated (200 **µm**); (d) pretreated (50 **µm**) potato slices. Pretreatment: SVI – 15000 ppm of $MgCl_2$ and 30 min sonication.
S = starch granule; D = disruption of cells; SP = separation of cells; WM = cell wall and middle lamella.

The formation of microscopic channels was not detected by SEM images. A possible explanation could be the "sponge effect" only took place during the application of ultrasonic waves, and no permanent disruption was caused. The applied acoustic waves resulted in continuous alternate cell flattening and elongation, thereby accelerating moisture loss and magnesium uptake during the pretreatment. This mechanism of ultrasound effect was also observed in melon, in which no breakdown of the tissue was observed (Fernandes, Gall ão, & Rodrigues, 2008).

5.9.2 Potato Chips

The microstructures of untreated (control) and pretreated potato chips deep-fat fried at 165 ℃ for 4 min are presented in Figure 24. It is interesting to note that the potato cells were not fully broken down after cooking, which was contrary to those observations from Burton (1948). However, our observations agreed with those obtained by Karizaki et al. (2013) that small changes were noticed for untreated potato samples after frying at 170 ℃ for 4 min. The SEM observations in Figure 24a and 24c show that the cell shape and integrity were well preserved for both untreated and pretreated potato chips. However, the size and shape of cell structure became less uniform in the control samples. The difference between untreated (Figure 24b) and treated fried (Figure 24d) samples in structure of cell wall and the middle lamella was in accordance with those described before frying, in which a thicker and stronger cellular connection was observed for the treated samples. This may help to explain the lower oil uptake and crisper texture for pretreated potato chips. Another possible reason for lower oil uptake could be that the metal ions uptake during pretreatment concentrated on potato surface,

obstructing the migration of oil into the structure (Oladejo et al., 2017). The observed microstructural differences between control and pretreated potato chips were also reflected on the measured hardness values, in which pretreated samples exhibited a significant harder texture as compared to untreated samples. No starch granules were observed in both control and treated potato chips, indicating starches were fully gelatinized during frying.

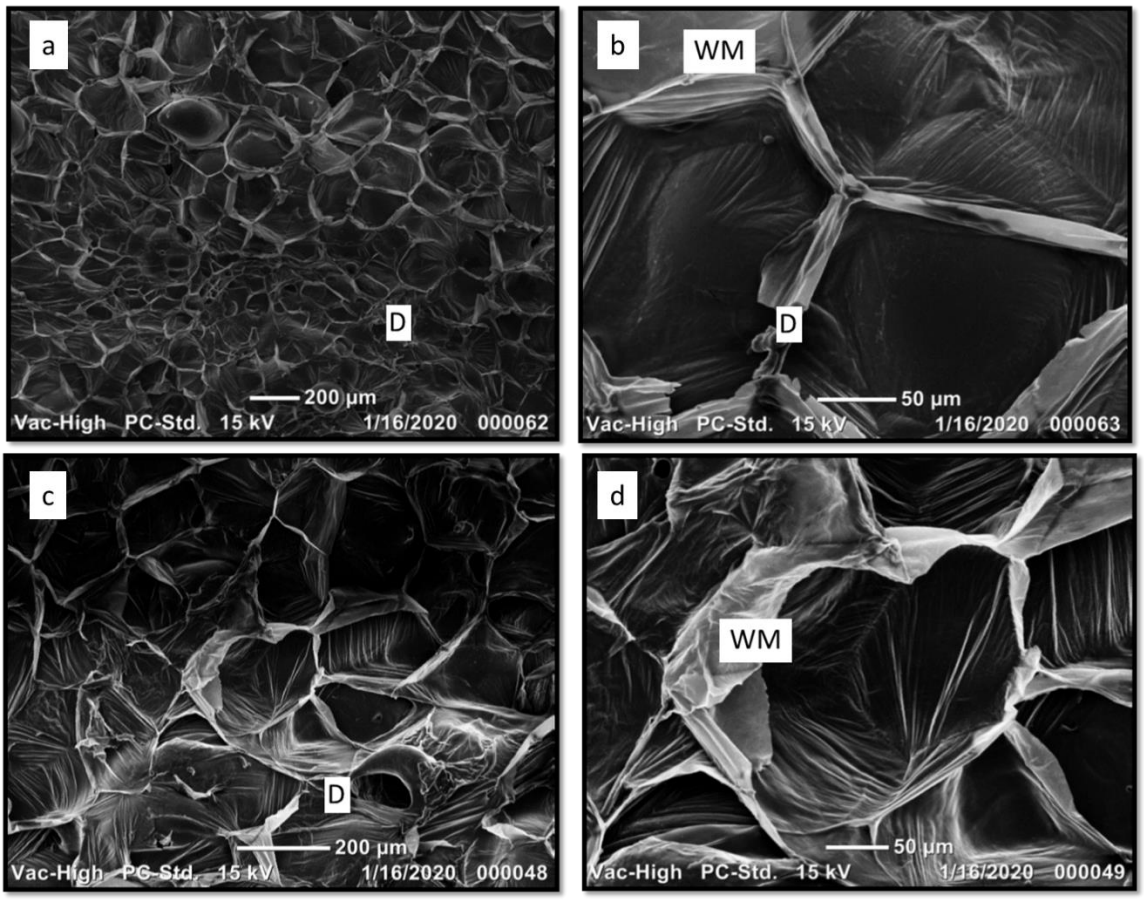

Figure 24 SEM images of (a) control (200 **µm**); (b) control (50 **µm**); (c) pretreated (200 **µm**); (d) pretreated (50 **µm**) potato chips fried at 165 ℃ for 4 min. D = disruption of cells; WM = cell wall and middle lamella.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

This study evaluated the effect of structural alteration of potato tissues by using divalent ions on oil uptake and some other quality attributes of deep-fat fried potato chips. The structure modification was achieved by sonication-assisted vacuum impregnation (SVI), with varying sonication times and soaking concentrations of $MgCl_2$. Non-sonication-assisted vacuum impregnation (NSVI) and sonicated without vacuum impregnation (S), were also carried out separately and compared with SVI pretreatment to evaluate if the combination of sonication and vacuum impregnation (VI) has a synergistic effect on reducing oil uptake. In addition, the effect of combined ions (Mg^{2+} and Ca^{2+}) was also evaluated based on product quality attributes such as oil content, texture, color, shrinkage, porosity, and sensory scores. Moreover, the mass transfer during SVI pretreatment, kinetics on moisture loss and oil uptake of pretreated samples during deep-fat frying, as well as microstructural changes were also analyzed in the study.

The main results obtained from this study were as follows:

- The combination of sonication and vacuum impregnation (SVI) has a synergistic effect on reducing oil uptake, with the oil reduction values of 20.06% at 50 min treatment time and 16.58% at 30 min sonication time, in comparison to samples pretreated with NSVI and S, respectively.

- Within the SVI pretreatment, the greatest oil reductions were observed from 10000 ppm to 15000 ppm of $MgCl_2$, with the reduction of 22.56%, 24.72%, and 28.37% at the sonication time of 10, 30, and 50 min, respectively. The lowest oil content of potato chips was obtained at the highest $MgCl_2$ concentration (20000 ppm) and longest sonication time (50 min), with a value of 0.1702 ± 0.0103 (d.b.), compared to the control sample of 0.3932 ± 0.0034 (d.b.).
- The firmness of pretreated potato slices was found increasing significantly ($p < 0.05$) with $MgCl_2$ concentration until reaching the maximum value of 1.25 ± 0.01 N at 10000 ppm for 30 min, then decreasing due to loss of cell turgor. Potato slices treated with combined ions (Mg^{2+} and Ca^{2+}) showed a significant higher firmness value.
- SVI pretreatment led to increased hardness of potato chips and it was significantly affected by the concentration of $MgCl_2$ solution. Potato chips pretreated with combined ions (Mg^{2+} and Ca^{2+}) showed a significant harder texture than only treated with Mg^{2+}.
- Both $MgCl_2$ concentration and sonication time were found significantly influencing the color parameters L^*, a^*, and b^*. Samples sonicated for longer time appeared lighter, while the impregnated $MgCl_2$ made the chips darker. Samples sonicated for a longer and with higher $MgCl_2$ concentration appeared redder and yellower.

Significant differences ($p < 0.05$) exist in L^* and b^* values between samples treated with combined ions (Mg^{2+} and Ca^{2+}) and only Mg^{2+}.

- The diameter shrinkage of potato chips was found increasing ($p < 0.05$) with $MgCl_2$ concentration and sonication time.
- The results showed that there was statistically significant ($p < 0.05$) effect of $MgCl_2$ concentration on bulk density and solid density. Samples treated with $MgCl_2$ at concentrations above 10000 ppm showed a significant ($p < 0.05$) lower value on porosity.
- No significant difference exists ($p > 0.05$) between potato chips treated with only $MgCl_2$ and those treated with a combination of $MgCl_2$ and $CaCl_2$ on oil content, color parameter a* (greenness-redness), shrinkage, bulk density, solid density, and porosity. They were significantly different ($p < 0.05$) in term of texture (hardness) as the addition of Ca^{2+} improved the sample's texture.
- In sensory analysis, no significant difference ($p < 0.05$) was detected between control, $MgCl_2$, and $MgCl_2$ + $CaCl_2$ treatments, except for the sensory attribute "texture". Potato chips treated with combined $MgCl_2$ and $CaCl_2$ received a significant higher score on "texture" than the other two treatments.
- The pretreatments (SVI and NSVI) were proven to effectively delivering magnesium ions into potato structure. The applications of ultrasound and higher concentration $MgCl_2$ resulted in higher water

loss and magnesium uptake. The result also suggests that the mass change during pretreatment was mainly due to the mositure migrations.

- The moisture loss as a function of frying time of pretreated (SVI) potato chips fried at 165 ℃ followed a typical drying behavior. The fitting of experimental data to the predictive exponential model gave a moisture diffusion coefficient value $D_e = 2.856 \times 10^{-8}$ m^2/s.
- The oil absorption as a function of frying time of pretreated (SVI) potato chips fried at 165 ℃ also showed a typical behavior in deep-fat frying, with the values of predicted constant rate $k = 0.010$ s^{-1} and regression coefficient $A = 1.010$ kg/kg solid.
- The SEM micrographs of pretreated (SVI) samples revealed a well-integrated cellular structure and thicker middle lamella, evidencing the texture improvement of pretreatment.

Recommendations for future research on sonication-assisted vacuum impregnation pretreatment of potato chips include:

- To quantify changes of dry matter, starch, pectic substances, and other chemical compositions during the pretreatment which may help understand the mechanisms behind the treatment and their correlations to oil uptake and other quality attributes of potato chips.

- To measure each fractions of the total oil, including structural oil (STO), penetrated surface oil (PSO), and surface oil (SO), in order to get a clearer picture of oil distributions in the crisps.
- To implement other microscopy techniques to better understand the microstructural changes, such as using atomic force microscopy (AFM) to study surface roughness, using transmission electron microscopy (TEM) to study the solutes (ions) distributions in potato tissues.
- To perform studies on sonication and vacuum impregnation process separately.

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APPENDIX

A - 1. Mass change (%) of potato slices pretreated (SVI and NSVI) in different concentrations of MgCl2 with the treatment time of 30 min.

Treatment	**SVI**	**NSVI**
$MgCl_2$ [10^3 ppm]	**ΔM [%]**	
0	5.60 ± 0.03	7.01 ± 0.03
1	5.25 ± 0.02	6.78 ± 0.02
5	4.85 ± 0.04	5.97 ± 0.03
10	1.99 ± 0.03	1.52 ± 0.02
15	-1.30 ± 0.37	-7.79 ± 0.47
20	-19.07 ± 0.04	-11.13 ± 0.13
50	-23.14 ± 0.44	-23.88 ± 0.49

A - 2. Moisture loss and oil absorption of potato chips fried at 165 ℃ pretreated with SVI in 15000 ppm $MgCl_2$ solution for 30 min.

Frying Time [s]	**MC [d.b.]**	**OC [d.b.]**
0	3.95 ± 0.01	0.00 ± 0.00
20	2.43 ± 0.02	0.04 ± 0.01
40	1.25 ± 0.02	0.09 ± 0.00
60	0.96 ± 0.01	0.12 ± 0.01
80	0.21 ± 0.01	0.16 ± 0.01
100	0.08 ± 0.01	0.18 ± 0.00
120	0.02 ± 0.01	0.21 ± 0.00
160	0.02 ± 0.01	0.21 ± 0.01
180	0.01 ± 0.01	0.23 ± 0.01
240	0.01 ± 0.01	0.25 ± 0.00
300	0.00 ± 0.00	0.27 ± 0.01
360	0.00 ± 0.00	0.28 ± 0.01
480	0.00 ± 0.00	0.28 ± 0.01
600	0.00 ± 0.00	0.28 ± 0.01
720	0.00 ± 0.00	0.28 ± 0.01