



Research article

Magnesium ion impregnation in potato slices to improve cell integrity and reduce oil absorption in potato chips during frying



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ABSTRACT

The effect of structural alteration of potato tissues by using divalent ions on oil uptake, texture, and color 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 $MgCl_2$.

SVI pretreated (sonicated by 50 min in a 15K magnesium solution) potato chips had 20% and 41% less oil content than the NSVI and control samples, respectively; and absorbed 29% more magnesium than the NSVI samples. The SVI pretreatment significantly affected product texture, color, shrinkage, and porosity.

Potato chips treated with combined $MgCl_2$ and $CaCl_2$ received a significant higher score than the other two treatments because of the improved sample's texture (crispness).

Microscopic analysis of SEM images showed a well-intact cellular structure and thicker middle lamellae after SVI treatment compared to the control samples.

1. Introduction

Although the use of metal ions, especially calcium ions, has been studied to improve the texture of foods, limited studies have provided experimental evidence relating the structure modification to oil reduction during deep-fat frying. Furthermore, most studies focus on the use of calcium ion (Ca^{2+}) (Morris, 1980). Although the forming of Ca^{2+} -pectin bridges improves the cell wall stability (Demarty et al., 1984; Koch et al., 2019), the difficulties obtaining the perfect balance between oil reduction and texture improvement have limited its application to fried products. The present study assessed the use of magnesium ion (Mg^{2+}) as an alternative to Ca^{2+} because of the similar divalent behavior in stabilizing cell structure, with expected lesser undesired hardness and/or off-taste of the fried products.

The health benefits of calcium and magnesium intake include a lower risk of type 2 diabetes (Liu et al., 2006; Villegas et al., 2009) and osteoporosis (Nieves, 2005). Increasing magnesium intake is recommended for those individuals susceptible to high blood pressure, kidney stones, and bladder stones (Bajaj, 1987). Based on that, the effect of a combination of Mg^{2+} and Ca^{2+} on oil content and texture of potato chips was also evaluated in the present study.

The use of metal ions is related to oil reduction in fried products by maintaining the cell integrity (Moreira et al., 2020). Kim and Moreira

(2013) found that by soaking potato strips in 3% w/w NaCl solution for 5 min, decreased oil absorption by 35% when the samples were de-oiled compared to the untreated samples. The use of $CaCl_2$ or $MgCl_2$ causes a firming effect in plant tissue by stabilizing its structure (Kasai et al., 1997; Mierczyńska et al., 2015; Moustacas et al., 1991; Poovaiah, 1986). The complexity of the firming mechanism rises when incorporating the cooking step, where starch gelatinization, protein denaturation, heat and mass transfer all take place simultaneously thus affecting the final product texture. It is generally accepted that the texture of cooked potato is attributed to (1) the retrogradation of starch, (2) the stabilization of the cell wall and middle lamellae by activating pectin methyl esterase (PME), (3) the leaching of amylose, and (4) the formation of salt bridges between pectin molecules (Andersson et al., 1994).

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 agree with those found by Khalil (1999). However, the mechanism of how impregnated divalent ions affect oil uptake in deep-fried potatoes is not yet fully understood.

One approach to enhance the delivery of metal ions into the potato tissue is the use of ultrasound, a technology widely used in food

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production to accelerate the mass transfer process (Gamboa-Santos et al., 2012; Lagnika et al., 2018; Rodríguez et al., 2018). In addition to ultrasound, vacuum impregnation has also been claimed as significant to introduce a desired solution into porous food materials, in a controlled way (Chiralt et al., 1999; Fito et al., 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. Therefore, this study also investigated the feasibility of using ultrasound-vacuum impregnation to enhance the texture and reduce oil content of potato chips.

2. Materials and methods

2.1. Raw material

The potatoes (*Solanum tuberosum*) used in this study were from CSS Farms (LLC, Dalhart, TX). The cultivar, Snowden, is used in the chips market because of its high specific gravity and uniform round to slightly flat shape. Potatoes were stored at 7 ± 1 °C and 90% RH. Before frying, potato reconditioning was done in a dark place at ambient temperature for 5–10 days to lower the reducing sugars content, which may cause undesirable browning during the frying process.

2.2. Sample preparation

Potatoes were selected based on a range of 1.078–1.080 specific gravity. Potatoes were washed, peeled, and sliced (thickness of 1.60 ± 0.10 mm) using a Mandolin slicer (Matfer model 2000, Matfer Bourgeat USA, Inc., Van Nuys, CA, USA). 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, rinsed with reverse osmosis (RO) water at room temperature (21.0 ± 1.0 °C) and then blotted dry gently with a paper towel.

2.3. Pre-treatments

Potato slices were immersed in soaking solutions (RO water, $MgCl_2$, or a combination of $CaCl_2$ and $MgCl_2$) at several concentrations, and simultaneously subjected to ultrasound followed by vacuum impregnation with 10,000 mg/L (1% w/v) $MgCl_2$. Six concentrations of $MgCl_2$ were used as soaking solutions during sonication, 0 mg/L (RO water), 5,000 mg/L, 10,000 mg/L, 15,000 mg/L, 20,000 mg/L, and 50,000 mg/L. For each of these concentrations, three sonication times were used: 10, 30, and 50 min. A mixture of 15,000 mg/L $MgCl_2$ and 15,000 mg/L $CaCl_2$ each (50% v/v) was 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 for specific assessments. 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.

2.4. Sonication process

A Branson ultrasonic tank (B52 model, Branson Co., Shelton, CT, USA), with capacity of 5 L and power of 240 W, frequency 47 kHz, 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 °C 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 airtight plastic bag, filled with soaking solution (100 ml) and sealed with a FoodSaver vacuum sealer, were submerged in the ultrasonic bath. Samples were submitted to ultrasonic

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

2.5. Vacuum impregnation (VI)

Each potato slice treated with ultrasound was placed in a glass container and then filled with 100 ml of impregnating solution (10000 mg/L $MgCl_2$). The concentration of 10,000 mg/L was sufficient to carry out vacuum impregnation at the ratio of 100 ml $MgCl_2$ /slice at a constant vacuum pressure of 600 mm Hg. The constant vacuum impregnation step was added to the study after a complete sonication process to evaluate whether adding such step would cause a synergistic effect. The VI treatment was carried out at ambient temperature (21.0 ± 1.0 °C) by applying vacuum for 10 min, and then restoring to atmospheric pressure for 10 min, while keeping the remaining potato slices immersed in the solution. The experiment was done in triplicate.

2.6. Frying process

The pretreated and untreated (control) samples were fried in a deep-fat fryer (Spin Frying Machine Model GSF026B, George Foreman, Beachwood, OH, USA) of capacity 2.6 L. 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. Once the temperature of the frying oil (canola oil – brought from local market, LouAnna, Brea, CA, USA.) reached 165 °C, the basket was lowered and potato slices were fried for 4 min to fully cooked potato chips, with a final the moisture content below 2% (w.b.). After frying, the basket with samples were raised from the hot oil and then centrifuged for 40 s. The chips were removed from the basket after the de-oiling step, cooled down for 2 min at room temperature, and the oil on chip's surface was then removed by gently blotting with paper towel. The samples were then stored in mason jars and then placed in a desiccator for additional analysis. Fresh oil was used for each experiment to avoid contamination by the $MgCl_2$.

2.7. Analytical methods

2.7.1. Moisture content

About 5 g of ground fried samples were placed into a forced air oven at 105 °C for 24 h (AACC, 1986). The moisture content of raw potato was determined using a conventional oven at 105 °C for 72 h (AOAC, 1990). The test was done in triplicate.

2.7.2. Oil content

The Soxtec System HT (Pertorp, Inc., Silver Spring, MD, USA) extraction unit with petroleum ether for 3 h (AACC, 1986) was used to measure total oil content of potato chips as described elsewhere (Moreira and Almohaimeed, 2018). The analysis was carried out in triplicate.

2.7.3. Magnesium (Mg^{2+}) content

Ground dry potato samples (0.5 g) was wet digested with 10 ml nitric acid using the MARS 6 microwave digestion system (CEM Corporation). The resultant solution was then diluted with deionized (DI) water to 50 ml, moved to centrifuge tubes and kept at refrigeration temperature. A plasma mass spectrometer (iCAP 7000 Plus Series ICP-OES, Thermo Fisher Scientific, Waltham, MA, USA) was used to quantify the magnesium concentration in the samples. To eliminate sample contamination and/or human error, the ASX-560 Autosampler (teledyn Cetac Technologies) was used in this study. All measurements were done in triplicate.

2.7.4. Specific gravity of potatoes

The method described by Da Silva (2018) was used to measure the specific gravity of the potatoes. It consists of a thin wire basket, which is placed inside a beaker containing water on top of an analytical scale

(0.01 g resolution, Sartorius, Wood Dale, IL, USA) was used to weight each potato. The potato weight in air (w_a) was measured directly from the scale and the potato weight in water (w_w) was determined by submerging the potato in the pre-tared basket under water.

2.8. Product quality attributes

2.8.1. Texture

Raw (pre-treated and untreated) and fried sample texture were evaluated with the puncture test using the Brookfield Texture Analyzer (TA-CT3 Texture Technologies Corporation, Scardale, NY, USA). The firmness of raw potato slices was defined as the maximum force of penetration (Steffe, 1996). One potato slice was placed on the middle of a hollow fixture plate (2-point support - 0.018 m hollow TA-DEC Pot) and penetrated with a cone-shaped probe with an angle of 30° (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. Hardness was used to characterize the mechanical strength of potato chips, identified as the peak force recorded to break the chips (Steffe, 1996). The probe used was a spherical ball with a 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.

2.8.2. Color

The color of potato chips was measured with a Hunter Lab Colorimeter LabScan XE (Hunter Associates Laboratory, Reston, VA, USA). 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.

2.8.3. Degree of shrinkage and porosity

Degree of diameter shrinkage and porosity of samples were measured as described in Da Silva and Moreira (2008).

2.8.4. Sensory evaluation

Fifty randomly selected 50 panelists from Texas A&M University (faculty, students, and staff) served as the consumer panel. Procedures followed were approved by and in accordance with the ethical standards of the Institutional Review Board (IRB) at Texas A&M University Division of research. Study Number: IRB2012-0019M. All participants consented the performed sensory tests.

The score of each sample was based on its appearance, color, odor, texture, flavor, and overall quality of the chips. A nine-hedonic scale (Da Silva and Moreira, 2008) was used, with "1" representing the lowest quality and "9" representing the highest quality. Scores higher or equal to "5" were considered acceptable.

The panelists evaluated three different samples: (1) Control (no treatment); (2) Chips sonicated (30 min) in 15000 mg/L MgCl₂ (food grade), followed by vacuum impregnation; (3) Chips sonicated (30 min) in a combination of MgCl₂ and CaCl₂ (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.

2.8.5. 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,

Wood Dale, IL) before and after the pretreatment (SVI or NSVI). Seven concentrations (0, 1000, 5000, 10000, 15000, 20000, 50000 mg/L) of soaking solutions (MgCl₂) were selected to analyze the mass transfer process. Five treatments were selected to do specific material balance, including control (no treatment), sonicated in 15000 mg/L MgCl₂ for 30 min, followed by VI (SVI15), non-sonicated (soaking) in 15000 mg/L MgCl₂ for 30 min, followed by VI (NSVI15), sonicated in 50000 mg/L MgCl₂ for 30 min, followed by VI (SVI50), and non-sonicated in 50000 mg/L MgCl₂ for 30 min, followed by VI (NSVI50).

a) Mass change (ΔM), [%] was calculated using Eq. (1):

$$\Delta M = \frac{m_t - m_0}{m_0} \times 100 \quad (1)$$

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

b) Water loss (ΔWL), [%] was determined using Eq. (2):

$$\Delta WL = \frac{w_{wo} \cdot m_o - w_{wt} \cdot m_t}{w_{wo} \cdot m_o} \times 100 \quad (2)$$

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 (Mg²⁺) change (ΔMg), [%] was calculated using Eq. (3):

$$\Delta Mg = \frac{w_{mg,t} \cdot m_t - w_{mg,o} \cdot m_o}{w_{mg,o} \cdot m_o} \times 100 \quad (3)$$

where m = mass in dry basis [g]; w_{mg} = mass fraction of Mg²⁺ (w/w).

2.8.6. Kinetics of moisture loss and oil absorption

The kinetics of moisture loss and oil absorption was determined by varying the frying time from 20 s to 720 s. Potato slices from control (without treatment) and pretreated samples in 15000 mg/L MgCl₂ with ultrasound simultaneously for 30 min, followed by VI (600 mm Hg pressure, vacuum time 10 min, restoration time 10 min) were used in this experiment. 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. Each assay was performed in triplicate.

2.8.7. Microstructural changes

The microstructural changes of potato samples were analyzed with scanning electron microscopy (SEM). Not treated potato slices (control) and treated samples with 15000 mg/L MgCl₂ for 30 min sonication, followed by VI were all 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 coated with gold using a sputter coater (TedPella Cressington 108). A scanning electron microscope (JEOL JCM-5000 Neoscope), at an accelerating voltage of 15 kV, was used to analyze the changes in the microstructure of the samples.

2.8.8. 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.

3. Results and discussion

3.1. Oil uptake

There was a 27%, 38%, and 41% decrease in oil uptake compared to the control for the SVI samples treated (15000 mg/L $MgCl_2$) for 10, 30, and 50 min, respectively $MgCl_2$ (Figure 1). The highest oil content value was obtained under NSVI treatment at 10 min (0.34 ± 0.02 g oil/g solid), while the lowest oil uptake was obtained with SVI samples at the treatment time of 50 min (0.23 ± 0.01 g oil/g solid). The sonication treatment significantly reduced oil uptake at all times ($p < 0.05$). In both SVI and NSVI treatments, statistical difference only exists between treatment time of 10/30 min, and 10/50 min. No significant difference ($p > 0.05$) was obtained between 30 min and 50 min because the concentration of soaking solution used (15000 mg/L) was not sufficient to cause further structural changes in the potato tissue, which, in turn, impacted oil absorption of potato chips (see Figure 2 where significant differences exist between 30 and 50 min of sonication times at the concentration of 20000 mg/L). The retention of metal ions by the plant cell wall depends not only on cell wall structure, but also on starch content, and type and concentration of treated metal ions, thus limitations exist in the absorption of metal ions (Beveridge and Murray, 1976; Fortuna et al., 2013; Lester and Grusak, 1999; Muschitz et al., 2015).

The significant oil reduction by the application of ultrasound is supported by previous studies (Karizaki et al. (2013), Dehghannya et al. (2016), Oladejo et al. (2017a, b), Da Silva (2018), and Dehghannya and Abedpour (2018)). The effect of sonication in the present study can be due to the cavitation or microchannels formed during the treatment, which enhances the delivery of magnesium ions into potato structure, thereby maintaining cell integrity, and impairing oil absorption during deep-fat frying.

Low concentration of $MgCl_2$ (5000 mg/L) did not affect oil uptake ($p > 0.05$) at all sonication times, while higher concentrations ($10, 15, 20 \times 10^3$ mg/L) impeded oil uptake significantly ($p < 0.05$) (Figure 2). The greatest oil reductions were observed from 10000 mg/L to 15000 mg/L, with the reduction of 23%, 25%, and 28% at the sonication time of 10, 30, and 50 min, respectively. At 15000 mg/L, significant changes on oil uptake were only noticed from 10 to 30 min, but not 30–50 min, probably due to the rupture of potato cell structure during longer sonication time. Same phenomenon was observed at 10000 mg/L. This finding

agrees with Karizaki et al. (2013) who showed that the longer osmotic treatment and ultrasound time caused damages to the potato cell structure.

Figure 3 shows the effect of vacuum impregnation on oil uptake by comparing the SVI samples 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. However, there was no significant difference between SVI and S samples at 10 min and 50 min, indicating that vacuum impregnation alone 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 cell structure, thus leaving less space for further ion impregnation. On the other hand, subjecting samples to ultrasound for too long might destroy the cell structure, thereby less effective on controlling oil absorption.

Compared with the NSVI samples, the S samples had significantly ($p < 0.05$) less oil content for 10- and 50-min soaking times. However, there was no significant difference ($p > 0.05$) between S and NSVI samples at 30 min. The experimental results affirm that the combination of sonication and vacuum impregnation (VI) has a synergistic effect on reducing oil uptake.

Oil content of SVI treated samples with $MgCl_2$ (0.2455 ± 0.0153^b) and those of SVI treated slices with a combination of $MgCl_2$ and $CaCl_2$ (0.2601 ± 0.0010^a) showed no evidence that significant difference exists ($p > 0.05$) on oil content between the two treatments.

3.2. Texture

3.2.1. Effect of SVI on potato slices firmness

Table 1 shows the texture results for SVI treated raw potatoes as function of $MgCl_2$ concentration. Raw potato slices without any treatment (control) had a firmness value of 0.96 ± 0.02 N. As concentration goes up, the firmness of pretreated potato slices initially 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). After reaching the highest firmness value at 10000 mg/L $MgCl_2$ concentration, the raw potato slices became very leathery, probably due to the osmotic gradient between the cell turgor and the outside solutions (Mauro et al., 2016). Thus, pressure difference acts as a driving force, pushing intra and inter-cellular fluid migrating out of the

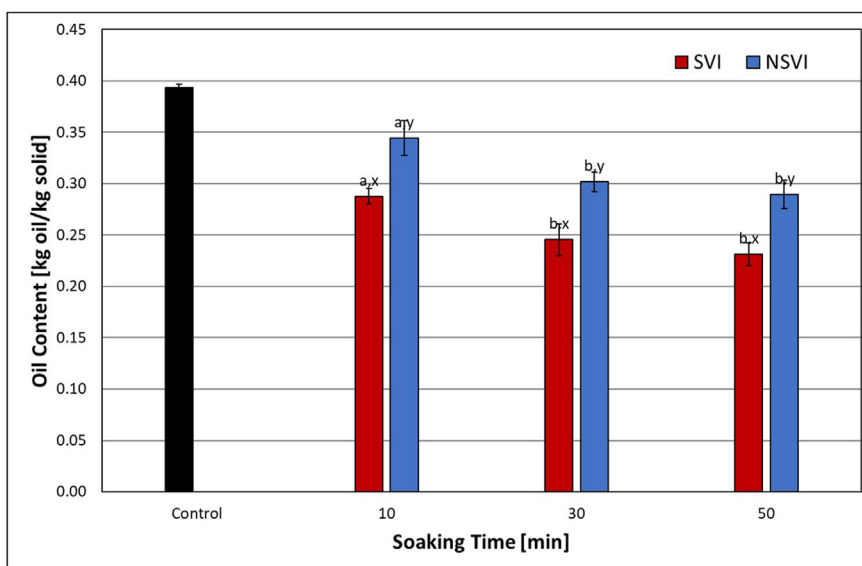


Figure 1. Oil content of potato chips treated with sonication (SVI) and without sonication (NSVI) in 15000 mg/L $MgCl_2$ for 10, 30 and 50 min, followed by VI (10000 mg/L). Different letters shown above each bar indicate significant difference exists, in which a-b represent differences between sonication times and pretreatments, whereas x-y represent differences between pretreatments within each sonication time. The control samples had an oil content of 0.3932 ± 0.0034 (g oil/g solid).

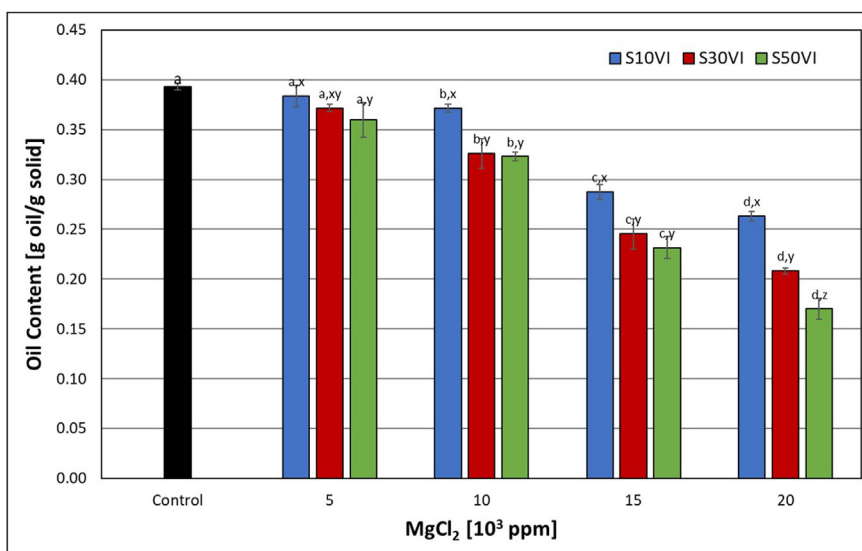


Figure 2. Oil content of SVI pretreat potato chips under different sonication conditions (sonication time and concentration). Different letters shown above each bar indicate significant difference exists, in which a-d represent differences between concentrations within each sonication time, whereas x-z represent differences between sonication times within each concentration.

slices to the surrounding solution. This behavior was also observed by Da Silva (2018).

Table 2 shows the comparison between SVI treated samples in MgCl₂ solution and SVI treated samples with a combination of MgCl₂ and CaCl₂. There was a significant difference ($p < 0.05$) in the firmness of samples pretreated with both Mg²⁺ and Ca²⁺, which showed a significant higher value on firmness, owing to the better performance of Ca²⁺ on “firming effect”.

3.2.2. Effect of SVI on potato chips hardness

The texture results of SVI treated potato chips as function of MgCl₂ concentration is shown in Table 1. The control sample had a hardness value of 1.75 ± 0.14 N. The concentration of MgCl₂ started to make significant difference ($p < 0.05$) at 10000 mg/L, compared to the control. Sonication time impacted ($p < 0.05$) the hardness of potato chips pretreated with 15000 and 20000 mg/L MgCl₂. With the Mg²⁺ contributing

to the improvement in texture. This finding is supported by with the work of Tajner-Czopek (2003). The firming effect is due to a decrease in pectin solubility, thus stabilizing the middle lamellae and cell walls.

Table 2 shows that the use of a combination of Mg and Ca yield significantly harder potato chips, mainly because Ca²⁺ is a more effective firming agent than other divalent ions (Mauro et al., 2016; Murayama et al., 2017; Tajner-Czopek, 2003).

3.3. Color

Pretreated potato chips had significant difference in lightness ($p < 0.05$) compared to the control samples (Table 3). Both concentration and sonication time affected ($p < 0.05$) the color parameter L^* . Samples sonicated for longer time appeared lighter, while impregnation with MgCl₂ made the chips darker. Pretreated potato chips showed significant difference in a^* value, compared to the control samples, except for

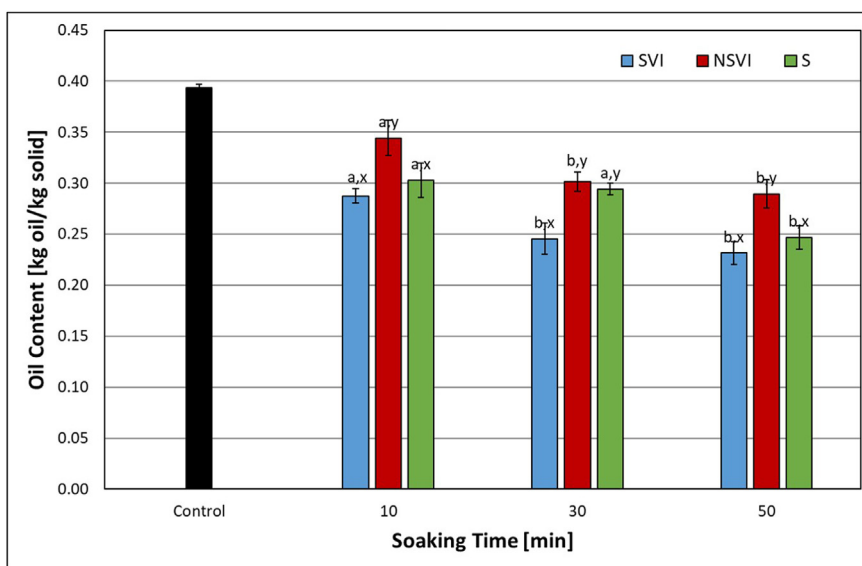


Figure 3. Comparisons of effects among SVI, NSVI, and S pretreatments on potato chips' oil content (fried for 4 min at 165 °C) sonicated in 15000 ppm MgCl₂ for 10, 30, and 50 min. Different letters shown above each bar indicate significant difference exists, in which a-c represent differences between sonication times within each pretreatment, whereas x-z represent differences between pretreatments within each sonication times.

Table 1. Firmness of potato slices and hardness of potato chips pretreated with SVI* for each sonication time and MgCl₂ concentration.

MgCl ₂ [10 ³ mg/L]	Sonication Time [min]		
	10	30	50
Firmness [N] of raw potato slices			
Control	0.96 ± 0.02		
5	_x 1.02 ± 0.01 ^b	_y 1.21 ± 0.03 ^b	_y 1.22 ± 0.03 ^b
10	_x 1.19 ± 0.02 ^c	_y 1.25 ± 0.01 ^b	_x 1.20 ± 0.02 ^b
15	_x 0.49 ± 0.00 ^d	_y 0.45 ± 0.02 ^c	_y 0.43 ± 0.0 ^c
20	_x 0.40 ± 0.00 ^e	_y 0.31 ± 0.02 ^d	_y 0.29 ± 0.02 ^d
Hardness [N] of potato chips			
Control	1.75 ± 0.14		
5	_x 1.75 ± 0.12 ^a	_x 1.78 ± 0.03 ^a	_x 1.88 ± 0.25 ^a
10	_x 2.11 ± 0.04 ^b	_x 2.08 ± 0.12 ^b	_x 2.09 ± 0.06 ^a
15	_x 2.26 ± 0.06 ^c	_y 2.12 ± 0.03 ^b	_x 2.28 ± 0.14 ^b
20	_x 2.19 ± 0.04 ^c	_y 2.49 ± 0.03 ^c	_y 2.56 ± 0.07 ^c
50	_x 2.91 ± 0.05 ^d	_x 2.82 ± 0.17 ^d	_x 2.89 ± 0.14 ^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} for each component indicate significant difference ($p < 0.05$) according to one-way ANOVA and Tukey's HSD test ($\alpha = 0.05$). *Sonication assisted-Vacuum Impregnation.

samples sonicated in 10000 mg/L MgCl₂ for 10 min (Table 3). Samples sonicated for a longer time and with higher MgCl₂ concentration appeared more red in color. Color b^* values of potato chips treated under different conditions showed significant differences ($p < 0.05$) for both factors (sonication time and concentration) (Table 3). The samples became more yellow when sonicated for a longer and with higher MgCl₂ concentration. Potato chips treated with 20000 mg/L MgCl₂ for 30 min were darker, redder, and yellower ($p < 0.05$) in comparison with other samples, resulting in an unacceptable color for consumers (Figure 4). This finding may be due to the presence of divalent Mg ions that stabilized the cellular structure of potato tissues, thus obstructing browning reactants from leaching out to the surroundings (Patton, 1948). The catalytic properties of magnesium during Maillard reaction are well known (Rizzi, 2008, 2010; O'Brien, 1997). Omari et al. (2019) showed that the catalytic effect of magnesium in beer color development is significant, persisting for about 20 min.

Significant differences ($p < 0.05$) exist in L^* and b^* values of SVI pretreated potato chips when using only Mg²⁺ and using combined Mg²⁺ and Ca²⁺. No difference ($p > 0.05$) was detected for a^* values between two pretreatments (Table 4).

3.4. Shrinkage and porosity

The diameter shrinkage of potato chips increased with MgCl₂ concentration (data not showing) and sonication time (control = 5.05 ± 0.20

Table 2. Texture of potato slices and potato chips sonicated in MgCl₂ and in a combination of MgCl₂ and CaCl₂ (750 mg/50 ml of each) for 30 min, followed by vacuum impregnation (VI).

Soaking solution	Firmness [N]
Raw Potato Texture	
MgCl ₂	0.45 ± 0.02 ^a
MgCl ₂ + CaCl ₂	1.16 ± 0.01 ^b
Potato Chips Texture	
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.

and SVI/1500 mg/L/30 min = 7.34 ± 0.24). Samples treated with MgCl₂ at concentrations above 10000 mg/L showed a significant lower value on porosity (control = 0.74 ± 0.01 and SVI/15000 mg/L/30 min = 0.69 ± 0.01). As expected, the samples shrunk as moisture is diffused out and the sample becomes more compacted as porosity decreases.

Results of combined Mg²⁺ and Ca²⁺ (12.03 ± 0.77) SVI treatment did not show differences ($p > 0.05$) with the samples treated with MgCl₂ (12.35 ± 0.6) only. There was no significant difference ($p > 0.05$) on porosity between potato chips treated with Mg²⁺ (0.69 ± 0.0) and a combination of Mg²⁺ and Ca²⁺ (0.69 ± 0.00).

3.5. Sensory evaluation

Significant difference ($p < 0.05$) was only observed in the sensory attribute "texture" (Table 5). Potato chips treated with combined MgCl₂ and CaCl₂ received a significant higher score. These samples were considered "crispier" by some panelists, which is in agreement with the statistical results regarding to "hardness" (see Table 1). 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 that they can be considered acceptable. No bitterness was sensed for pretreated potato chips, which was contrary with those obtained by Yang and Lawless (2005) who found that the use of CaCl₂ and MgCl₂ brought adverse effect of bitterness and off flavor. In summary, the pretreatments produced potato chips with improved texture and retained sensory quality.

3.6. Mass transfer during pre-treatment

Table 6 presents the experimental data of magnesium content of untreated (control) and pretreated (SVI and NSVI) potato slices and fried potato chips in 15000 and 50000 mg/L MgCl₂ for 30 min treatment time. Pretreated samples showed a significant ($p < 0.05$) higher amount of magnesium content than the control samples. This affirms the efficiency of the pretreatment as the presence of Mg²⁺ helped the retention of pectic substances by binding with them in the cell walls and the middle lamellae Tajner-Czopek (2003). The statistical results also show that magnesium content was significantly ($p < 0.05$) higher when treated with higher concentration of MgCl₂. As expected, the sonicated potato slices and chips showed higher magnesium contents than non-sonicated samples, indicating the ultrasonic waves facilitated the delivery of Mg²⁺.

Table 3. Effect of MgCl₂ concentration and sonication time during pretreatment on color parameter L*, a*, and b*.

MgCl ₂ [10 ³ mg/L]	10 min	30min	50min
	L*		
Control	62.21 ± 0.22		
5	_x 61.28 ± 0.49 ^a	_y 63.94 ± 1.12 ^a	_z 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
	a*		
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
	b*		
Control	15.48 ± 0.07		
5	_x 14.85 ± 0.27 ^a	_x 15.04 ± 0.07 ^a	_x 14.58 ± 0.23 ^a
10	_x 16.09 ± 0.21 ^b	_y 17.30 ± 0.04 ^b	_y 17.44 ± 0.19 ^b
15	_x 14.43 ± 0.25 ^a	_y 16.59 ± 0.21 ^c	_z 17.19 ± 0.18 ^b
20	_x 17.90 ± 0.62 ^c	_{xy} 18.33 ± 0.32 ^d	_y 19.18 ± 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 ($\alpha = 0.05$).

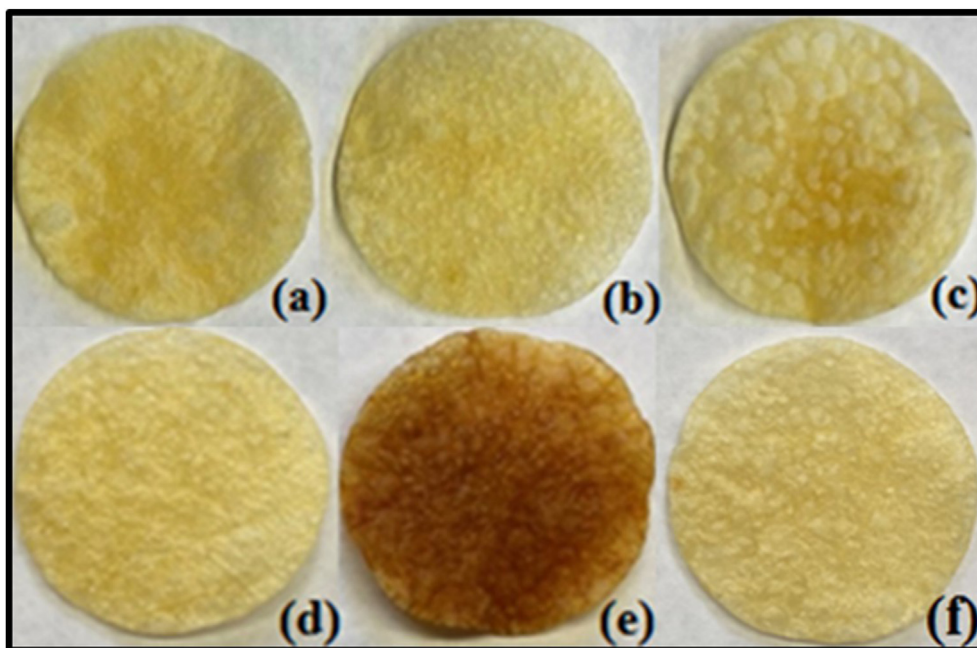


Figure 4. Potato chips sonicated in: (a) control (no treatment); (b) 5000 ppm; (c) 10000 mg/L; (d) 15000 mg/L; (e) 20000 mg/L MgCl₂; (f) MgCl₂ + CaCl₂ (750/50 mg/ml of each) for 30 min, followed by VI.

Table 4. Color parameters of potato chips sonicated in only MgCl₂ and in a combination of MgCl₂ (750 mg/50 ml of each) and CaCl₂ 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.

Table 5. Sensory evaluation results for potato chips processed under different pretreatments.

Sample	Appearance	Color	Odor	Texture	Flavor	Overall Quality
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
SVI-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
SVI- 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 ($\alpha = 0.05$). SVI = Sonication-assisted-Vacuum Impregnation.

There was a small percentage loss of magnesium content after frying (Table 6). For the SVI process, potato slices impregnated with 15000 mg/L or 50000 MgCl₂ solution lost around 8% of magnesium during frying. However, for the slices that were not sonicated (NSVI), soaking the slices at higher concentration (50000 mg/L) resulted in 17% loss of magnesium after frying. This is an indication that sonication should be used to properly impregnate raw potato with metal ions.

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 mg/L) (Table 7). Sonication of potato slices in osmotic solution increased the diffusion rate and consequently amount the water loss and solid gain (Karizaki et al., 2013). MgCl₂ 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 MgCl₂ was used. As expected, the changes in magnesium content (ΔMg) was higher for the ther higher concentration (50000 mg/L) of MgCl₂ for the SVI and NSVI treated samples. SVI treated samples absorbed 29% more magnesium than the NSVI samples for the 15000 mg/L solution concentration. For the 50000 mg/L solution, the SVI samples absorbed 15% more Mg than the NSVI ones.

Sonication alone (with no VI) was not as effective to impregnate potato slices with Mg ions compared with the SVI or NSVI treatment. The ΔMg values for the S treated samples were $144.66 \pm 15.51\%$ for the lower concantration and $430.19 \pm 26.84\%$ for the higher one, i.e., it absorbed 3–4 times less Mg than the SVI or NSVI samples. These results clear indicated the synergistic effect of vacuum impregnation and ultrasound techniques to impregnated potato slices with Mg Cl₂.

Table 6. Magnesium content (Mg²⁺) of potato slices and potato chips (fried for 4 min at 165 °C) without pretreatment (control) or pre-treated with (SVI) and without (NSVI) sonication in 15000 mg/L and 50000 mg/L MgCl₂ for 30 min, followed by VI.

MgCl ₂ [10 ³ mg/L]	Magnesium Content [mg/100g D.M.]	
	SVI	NSVI
Raw Potato Slices		
Control	75.67 ± 2.08	
15	$\bar{x}461.7 \pm 3.5^a$	$\bar{y}421.33 \pm 3.21^a$
50	$\bar{x}1261.33 \pm 4.5^b$	$\bar{y}1156.00 \pm 6.56^b$
Potato Chips		
Control	66.67 ± 3.06	
15	$\bar{x}429.67 \pm 3.06^a$	$\bar{y}392.33 \pm 3.5^a$
50	$\bar{x}1165.33 \pm 5.5^b$	$\bar{y}954.67 \pm 6.1^b$

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.

3.7. Kinetics of dehydration and oil uptake

3.7.1. Moisture content

The kinetics of dehydration and oil uptake of pretreated potato chips (SVI) during deep-fat frying were analyzed. The moisture content of pretreated potato slices was 3.95 ± 0.14 (d.b.). The moisture loss profile

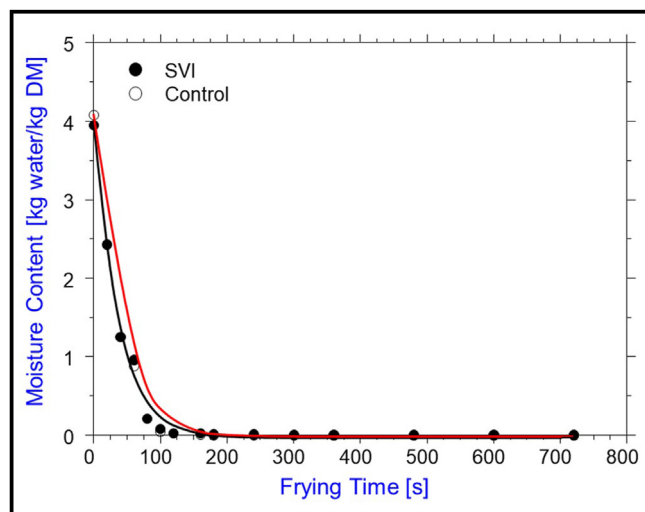


Figure 5. Moisture loss of potato chips fried at 165 °C pretreated with SVI in 15000 mg/L MgCl₂ solution for 30 min and control. (symbols are experimental values and line the predicted curve using Eq. (4)).

shown in Figure 5, exhibits a classical drying behavior described by

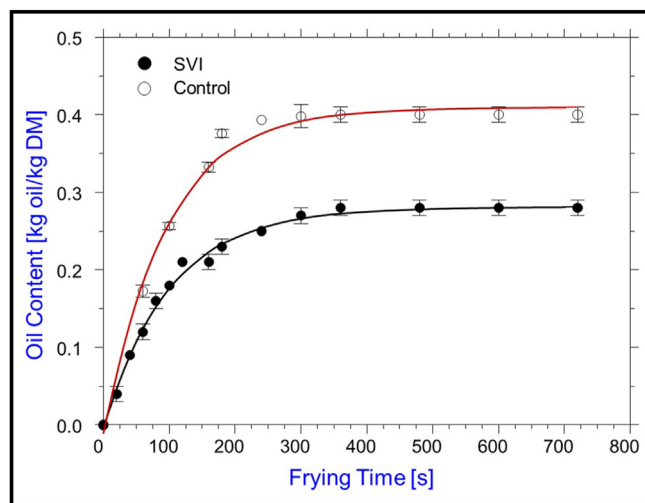


Figure 6. Oil absorption for potato chips fried at 165 °C pretreated with SVI in 15000 mg/L MgCl₂ solution for 30 min and control. (symbols are experimental values and line the predicted curve using Eq. (5)).

Table 7. Mass change (ΔM), water loss (WL), and magnesium uptake (ΔMg) during the pretreatments (SVI and NSVI) in 15000 mg/L and 50000 mg/L $MgCl_2$ for the treatment time of 30 min.

	$MgCl_2$ [10^3 mg/L]	SVI	NSVI
ΔM [%]	15	$x-1.30 \pm 0.37^a$	$y-7.78 \pm 0.47^a$
	50	$x-23.13 \pm 0.44^b$	$x-23.88 \pm 0.49^b$
ΔWL [%]	15	$x2.27 \pm 0.37^a$	$y5.99 \pm 0.30^a$
	50	$x26.02 \pm 0.48^b$	$x25.51 \pm 0.79^b$
ΔMg [%]	15	$x526.63 \pm 5.72^a$	$y373.34 \pm 30.29^a$
	50	$x1378.74 \pm 49.48^b$	$y1165.75 \pm 24.89^b$

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.

Garayo and Moreira (2002). The experimental data was fitted to the exponential model described by Eq. (4):

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

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 values of the moisture diffusion coefficient obtained from the predictive exponential model were $D_e = 7.125 \times 10^{-9} m^2/s$ (Predreschi, Hernandez, Figueroa, & Moyano, 2005; Granda, 2005) and $7.146 \times 10^{-9} m^2/s$ and for the SVI and control samples, respectively. The SVI treated samples dried slightly faster than the control samples indicating the

effect of sonication on the moisture loss. As expected, the moisture decreased drastically especially in the early stages of frying (0–60 s).

3.7.2. Oil content

Figure 6 presents the oil absorption profile of potato chips pretreated (SVI) in 15000 mg/L $MgCl_2$ solution for 30 min. The kinetic model described by Yagua and Moreira (2011) was used to fit the experimental data of oil content (Eq. 5):

$$OC_t = A \cdot \exp(-kt)(OC_0 - OC_e) + OC_e \quad (5)$$

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 $k = 0.01002 s^{-1}$ and $A = 1.0000 kg/kg$ solid for the SVI samples and $k = 0.01076 s^{-1}$ and $A = 0.99929 kg/kg$ solid for the control samples. There were no changes

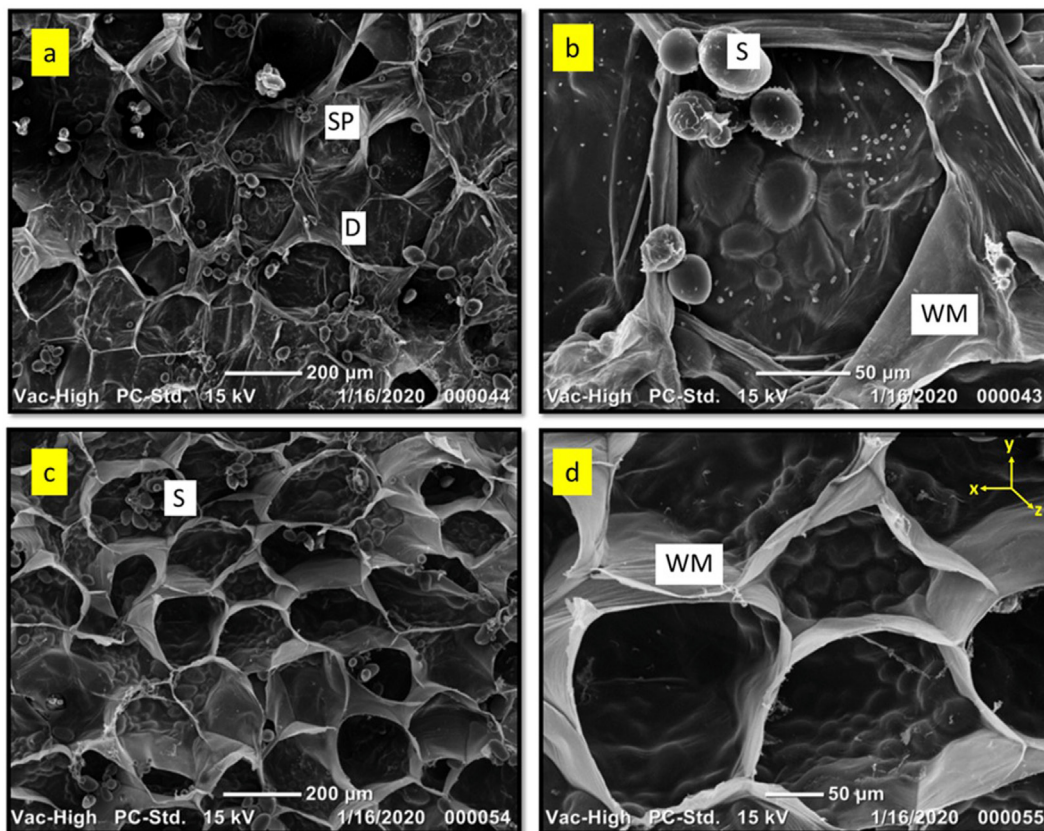


Figure 7. SEM images of raw potato slices: (a) control (200 μm); (b) control (50 μm); (c) pretreated (200 μm); (d) pretreated (50 μm) potato slices. Pretreatment: SVI – 15000 mg/L of $MgCl_2$ and 30 min sonication. S = starch granule; D = disruption of cells; SP = separation of cells; WM = cell wall and middle lamella.

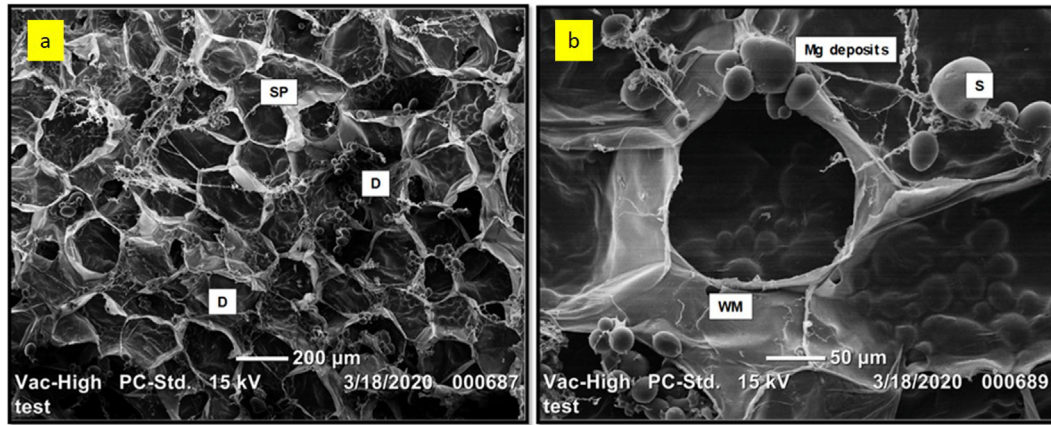


Figure 8. SEM images of raw potato slices: (a) NSVI (200 μm); (b) NSVI (50 μm) Pretreatment: NSVI – 15000 mg/L of MgCl_2 and 30 min soaking. D = disruption of cells; WM = cell wall and middle lamella.

in the oil kinetic behaviors for the control and SVI samples. However, the final oil content values were very different between control (0.40 kg/kg solid) and SVI (0.28 kg/kg solid). 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 and Pedreschi, 2006; Pedreschi et al., 2007).

3.8. Microstructural changes

3.8.1. Raw potato slices

The microstructure of fresh potato slices examined by SEM revealed a characteristic morphology of potato cells. As shown in Figure 7a-d, the

cell structure was well arranged in the control and pretreated (SVI – 15000 mg/L of MgCl_2 and 30 min sonication) potato slices and cell walls, middle lamellae, as well as starch granules were clearly presented. A greater degree of cell separation and more collapsed cell structure were seen in the control samples (Figures 7a and 7b) compared to the pretreated samples (Figures 7c and 7d), which showed a well-integrated cellular structure. No cell distortion was observed due to ultrasound pretreatment (Oladejo, Ma, Qu, Zhou, Wu, Yang and Onwude, 2017a, b) for sweet potato chips. Unevenly distributions of intact round-shape starch granules were observed in both control and treated samples. The SEM micrographs under higher magnification (Figure 7b, d) revealed more details concerning the structure of cell walls and the middle

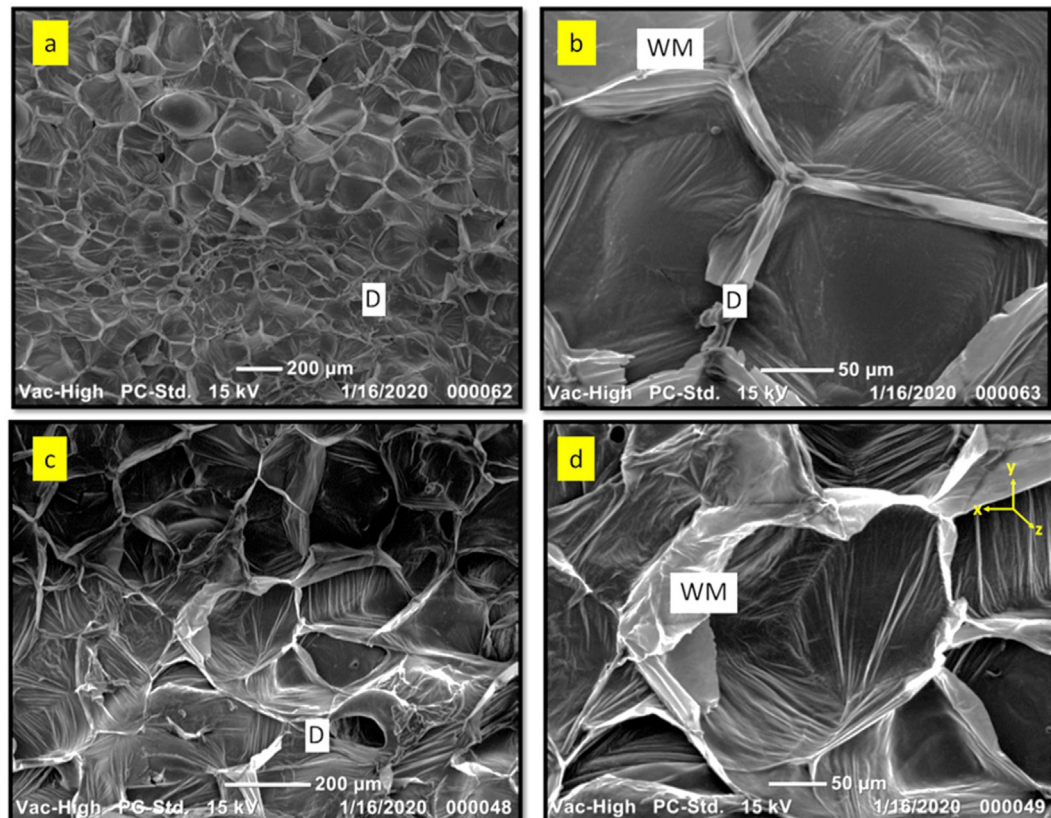


Figure 9. SEM images of potato chips: (a) control (200 μm); (b) control (50 μm); (c) pretreated (200 μm); (d) pretreated (50 μm) potato chips fried at 165 $^{\circ}\text{C}$ for 4 min. D = disruption of cells; WM = cell wall and middle lamella. Pretreatment: SVI – 15000 mg/L of MgCl_2 and 30 min sonication.

lamellae. It is observed that the middle lamellae was thicker (z-coordinate) in the SVI than in the control samples, evidencing the formation of cross-bridges between magnesium ions and pectic substances. The thicker the middle lamellae the stronger the cell adhesions and less cell separation. Similar results were reported by [Moreno et al. \(2004\)](#) for Chilean papaya treated with VI and OD. They attributed the thickening of middle lamellae to the formation of a polymeric compound due to interactions between the middle lamellae 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 et al., 2000](#)).

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 et al., 2008](#)).

[Figure 8](#) shows the effect of vacuum impregnation only (NSVI) for the samples soaked for 30 min in a 15000 MgCl₂ solution before VI was applied. When comparing these images with those of the SVI samples ([Figures 7c and 7d](#)), the NSVI microstructures seem to be more disorganized, with more cell ruptures and separations. The middle lamellae appears to be less thick than that of the SVI samples. It was also noticed that the NSVI images showed more deposits of Mg (strings) at the surface of the slices.

The structure of the final product is greatly affected using sonication assisted impregnation of di-valent ions in the potato-tissue. MgCl₂ ions help by creating MgCl₂-pectate bridges, which firm the cell walls, increase the middle lamellae-cell wall rigidity, and reduce additional degradation during the frying process. As a result, less pores would be formed as less cells would rupture; additionally, as the chemical composition of the formed pores would be different, the surface tension would change and affect the pore capillary pressure responsible for oil uptake ([Marle, 1997](#)).

3.8.2. Potato chips

The microstructures of control and SVI pretreated potato chips deep-fat fried at 165 °C for 4 min are presented in [Figure 9](#). It was noticed that the potato cells were not fully broken down after frying, 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 °C for 4 min. The SEM observations in [Figures 9a and 9c](#) 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. Comparing the control ([Figure 9b](#)) with the SVI treated samples ([Figure 9d](#)), a thicker and stronger cellular connection was observed for the treated samples, which have resulted in lower oil uptake and crispier texture for the SVI pretreated potato chips. Another possible reason for the lower oil uptake could be that the metal ions uptake during the pretreatment concentrated on the potato surface, obstructing the migration of oil into the structure ([Oladejo et al., 2017a, b](#)). The observed microstructural differences between the control and SVI pretreated potato chips were also reflected on the measured hardness values, in which pretreated samples exhibited a significant harder texture as compared to the control samples. No starch granules were observed in both control and treated potato chips, indicating starches were fully gelatinized during frying.

4. Conclusions

The combination of sonication and vacuum impregnation (SVI) has a synergistic effect on oil uptake, with oil reduction values of 20% at 50 min treatment time and 17% at 30 min sonication time, in comparison to

samples pretreated with NSVI and S only, respectively. Within the SVI pretreatment, the greatest oil reductions were observed from 10K mg/L to 15K mg/L of MgCl₂, with the reduction of 23%, 25%, and 28% at the sonication time of 10, 30, and 50 min, respectively. The lowest oil content of potato chips was obtained at the highest MgCl₂ concentration (20K mg/L) and longest sonication time (50 min).

Potato slices treated with combined ions (Mg²⁺ and Ca²⁺) showed a significant higher firmness. The hardness of the SVI pretreated potato chips increased with the increased concentration of MgCl₂ solution.

Potato chips treated with combined MgCl₂ and CaCl₂ received a significant higher score on "texture" than the control and NSVI treatments.

The pretreatments (SVI and NSVI) were effective in delivering magnesium ions into the potato structure. Applications of ultrasound and higher concentration MgCl₂ yielded higher water loss and magnesium uptake in the fried product.

Declarations

Author contribution statement

Tianyan Zheng: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Rosana G. Moreira: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data availability statement

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Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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