



Development of fortified low-fat potato chips through Vacuum Impregnation and Microwave Vacuum Drying

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ABSTRACT

Potato chips, traditionally prepared by conventional frying, are the most widely consumed snack in the world; however, it has some health concerns associated. This work aims to produce potato chips fortified with calcium and vitamins C and E using Vacuum Impregnation and Microwave Vacuum Drying. The drying process conditions were optimized considering the power density and absolute pressure as independent variables, and some textural and nutritional properties, as dependent variables. The chips were evaluated for microstructure, sensory, and storage studies. The fortification process through Vacuum Impregnation significantly enhanced the content of fortifiers. A power density of $1.7 \text{ W} \cdot \text{g}^{-1}$ and an absolute pressure of 4.0 kPa were found to be optimum microwave vacuum drying conditions yielding an average retention higher than 90% of calcium, 53% of vitamin C and 72% of vitamin E. It is concluded that the combination of vacuum impregnation and vacuum microwave drying is an alternative to produce value-added low-fat snack with acceptable sensory quality.

Industrial relevance: Potato is a promising food for the development of innovative food products with health benefits, due to its high consumption worldwide. Potato chips could be used as an ideal strategy for targeted nutrient delivery e.g. vitamins and minerals. The findings of this study showed that the use of vacuum impregnation as a fortification technique, with the subsequent stabilization by microwave vacuum drying, are promising ways to develop fortified low-fat potato chips.

1. Introduction

The potato chip or crisp (PC) is considered one of the most popular snacks globally (Dhital et al., 2018; Tian et al., 2017). Some authors define snack “as a portion of food, smaller than a regular meal, generally eaten between meals” (Pedreschi et al., 2018). Snack food manufacturers produce mainly two kinds of potato crisps: the traditional crisps from fresh potatoes, which are produced from thinly slicing and deep frying of them, and the typical restructured potato crisps from a potato dough, which are molded into desired shapes by extruding or pressing before frying, such as Pringles® (Pedreschi et al., 2018).

Traditionally, potato crisps are produced by oil frying to a temperature well above the boiling point of water (e.g., 180 °C), causing evaporation of water inside the product, and the formation of a crust. Besides cooking foods very quickly, this unit operation also provides unique sensorial attributes attractive to the consumer such as colors,

aromas, flavors and textures that improve the overall palatability (Pedreschi et al., 2018). However, the health concerns of fried potatoes are primarily associated with their high oil content, the formation of hazardous chemical substances (e.g., acrylamide), and the rapid digestion of starch (Joshi et al., 2016a, b; Tian et al., 2017), which implies a high glycemic index. It has been found that excessive consumption of foods with high-fat content contributes to the development of diseases such as obesity, hypertension, diabetes and cardiovascular diseases (World Health Organization, 2018).

Therefore, instead of conventional frying methods, an alternative processing method to produce healthier products is urgently needed (Tian et al., 2017). Some of the methods for the production of healthy potato snack reported at industrial and laboratory level are the application of edible coatings before frying (Trujillo-Agudelo et al., 2019), frying at low pressure (Belkova et al., 2018), and air frying (Tian et al., 2017). Nevertheless, the snack can also be produced by drying. This food preservation method reduces both product moisture content and

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water activity, slowing down microbial growth, enzymatic activity, and chemical reaction, extending food shelf life and reducing transportation and storage costs (Barbosa-Cánovas and Ibarz, 2003; Monteiro et al., 2016). Many different drying processes have been developed, such as hot air-drying, freeze-drying, vacuum-drying, solar drying, and osmotic drying, finding that most of those processes can be assisted by microwaves (Schubert and Regier, 2005) and/or run under vacuum conditions.

For different consumption purposes, the dehydration process of potatoes can produce two kinds of products, namely, dehydrated vegetable and snack food (Wang et al., 2010). Dehydrated potato snack has been produced using several methods, such as drying with superheated steam (Leeratanarak et al., 2006), freeze-drying microwaves (Wang et al., 2010), microwaves (Joshi et al., 2016a), infrared radiation (Wu et al., 2018), and microwave vacuum drying (Lefort et al., 2003; Setiady et al., 2007). Snacks dried using different drying methods have a different flavor, texture, and taste; the combination drying technology can improve the quality of dried products by avoiding disadvantages of any single drying method. (Huang and Zhang, 2012).

The use of Microwave Vacuum Drying (MVD) permits a shorter drying time and a substantial improvement in the quality of dried materials (Bondaruk et al., 2007). MVD is a dehydration process that uses microwave radiation (frequencies ranging from 0.3 GHz to 300 GHz) to generate heat in a chamber with absolute pressure in the range from above the triple point of water to less than atmospheric pressure (101.33–0.61 kPa) (Scaman et al., 2014). When microwaves impinge on a material, part of the energy is transmitted, a part is reflected, and part is absorbed by the material, being dissipated as heat (Meda et al., 2016). Heating starts from inside of the product and it is faster than conventional heating (Monteiro et al., 2018).

On the other hand, Vacuum Impregnation (VI) has been described as a fast mass transfer process that occurs when porous structures are immersed in a liquid phase (Duarte-Correa et al., 2019). VI has emerged as a useful tool for incorporating physiologically active compounds into the porous structure of fruits and vegetables (Joshi et al., 2016b). VI consists of exchanging the internal gas, or liquid occluded in open pores, for an external liquid phase, due to the action of hydrodynamic mechanisms promoted by pressure changes (Fito et al., 2001). The application of reduced pressure to a solid-liquid system, followed by restoration of atmospheric pressure cause the incorporation of active compounds into the food by the impregnation of the liquid in which it is dissolved (Mújica-Paz et al., 2003).

Several works have reported that the fortification of potatoes using VI technique increases the content of ascorbic acid (Hironaka et al., 2011), zinc (Kazunori Hironaka and Koaze, 2015), iron (Erihemu et al., 2015), calcium and zinc (Joshi et al., 2016b), calcium (Tiware et al., 2018), phenolic compounds from beet (Moreira and Almohaimeed, 2018), calcium, vitamin C, and vitamin E (Duarte-Correa et al., 2019), and essential rosemary oil, as well (Luo et al., 2019). However, while some of the works intended to produce fortified snacks, some others aimed to produce a minimally processed product. Finally, other foods that have been fortified using vacuum impregnation are pineapple (Mateus de Lima et al., 2016), eggplants, carrot, and mushrooms (Gras et al., 2003), apple (Codoñer-franch et al., 2013), and gooseberry (Restrepo et al., 2009), among others.

The aim of this research was to produce potato chips fortified with calcium and vitamins C and E through Vacuum Impregnation and Microwave Vacuum Drying (MVD) techniques. MVD conditions were optimized considering the power density and absolute pressure of microwave vacuum drying as independent variables, and some physico-chemical properties (moisture and water activity), functionals (antioxidant capacity), textural (hardness and crispness work), and nutritional (available lysine), as well as the added active components (AC) (vitamin E, vitamin C, and calcium) contents, as dependent variables. Finally, potato chips produced under optimal drying conditions were submitted to sensory analysis. Moreover, the stability of added

components was evaluated during 3 months of storage at 25 ± 2 °C and $65 \pm 2\%$ of relative humidity. This work represents a proposal to increase the market of healthy products as fortified dehydrated potatoes, because of its availability, convenience, versatility, stability, and nutritional value.

2. Materials and methods

2.1. Materials

This research used the Diacol Capiro variety of potato (*Solanum tuberosum*) with medium caliber (45 mm–64 mm diameter) based on Colombian Technical Standard NTC 341 (ICONTEC, 1996a), and harvested in La Unión, located east of Medellín, Colombia. The potatoes were washed and cut into slices of 2.5 mm thickness. Afterward, they were blanched with a 1:10 potato: water ratio for 4 min at 80 ± 2 °C; cooled and dried with a paper towel to remove any excess moisture.

Reagents: calcium lactate, DL- α -tocopherol acetate and L-ascorbic acid were acquired from Bellchem®. Vitamin E standard was acquired from Sigma-Aldrich®.

2.2. Fortification process

An impregnation emulsion containing 0.2% vitamin E, 0.6% vitamin C and 0.9% Ca^{2+} was prepared. These compounds were emulsified over an isotonic aqueous base (NaCl: 0.8%). Surfactants Tween 80 (0.051%) and Span 60 (0.049%) were also used (Duarte-Correa et al., 2019). The impregnating emulsion was prepared using a homogenizer (Ultraturrax Microdistech, MDT 1000, Estados Unidos) at 20,000 rpm for four 3-min cycles followed by a 3-min rest to control the system's temperature.

The particle size of the emulsion droplet was analyzed by dynamic light scattering using a Zetasizer Nano-ZS90 equipped with zetasizer software (version 7.11, Malvern Instruments Ltd., UK) and was found that the droplet had an average diameter of 234.9 nm, with particle sizes from 137.9 nm to 331.9 nm (Duarte-Correa et al., 2019).

The fortification process was carried out using the vacuum impregnation (VI) technique (Fig. 1a). Potato slices were placed in a desiccator and the potato to emulsion ratio was maintained to 1:5 to ensure adequate immersion. A vacuum pump was attached to the desiccator (Welch, Gardner Denver Thomas, Inc. Sheboygan, WI USA) and the system was subjected to a vacuum pressure (VP): 77.3 KPa, for 3 min. Subsequently, the atmospheric pressure was restored and the slices remained submerged for an additional time (4 min) without stirring (Duarte-Correa et al., 2019). Finally, the impregnated samples were taken out from the desiccator, drained, and the excess liquid on the sample's surface was removed lightly with absorbent paper (Luo et al., 2019; Setiady et al., 2007). Part of the impregnated samples was used to analyze calcium, vitamin E, vitamin C; and another part of the impregnated samples was packed in bags flex up, pet metallic/flexible (117 μm) (Alico SA, Medellin, Colombia) and stored at 25 ± 2 °C before MVD process.

2.3. Microwave vacuum drying (MVD)

The fortified potato slices were subsequently dehydrated using a batch vacuum microwave equipment of semi-industrial scale (Fig. 1b), with magnetrons (CW 2450 MHz, Toshiba, China) and vacuum pump (Lanphan 2XZ-4, Zhengzhou, China). The potato slices were placed in two round trays (50 cm diameter), rotating at a speed of 6 rpm and the output power was 0.6 kW (2.45 GHz). The microwaves were applied under a Temperature-Controlled System (Gaukel et al., 2016), that automatically regulates the power of the introduced microwaves (on-off cycles) depending on the temperature, which was set at 60 ± 2 °C (Duarte-Correa et al., 2019; Setiady et al., 2009b). The vacuum chamber pressure was reduced until a value defined of absolute

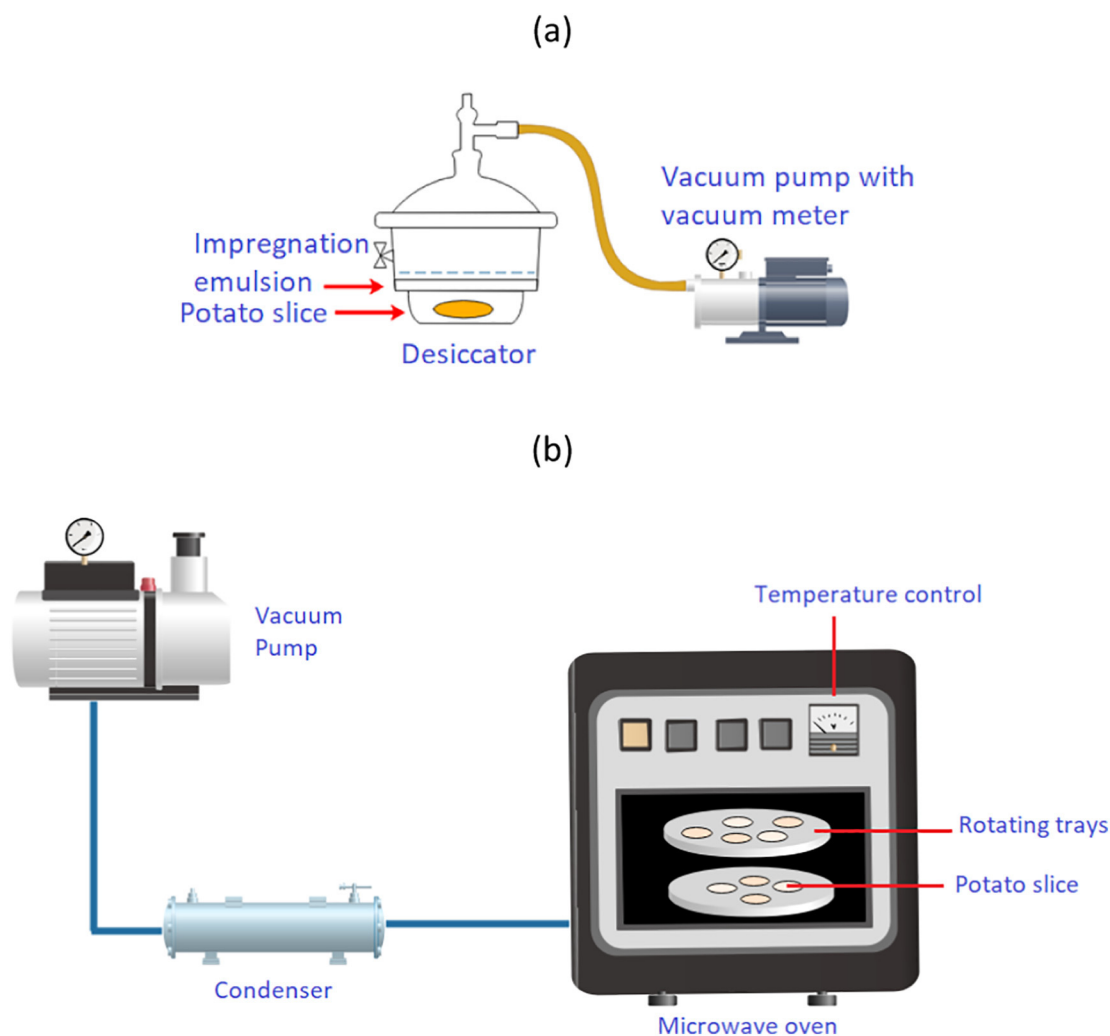


Fig. 1. Schematic representation of (a) vacuum impregnation system and (b) microwave vacuum drying system.

Table 1

Moisture, water activity (A_w), drying time, and vitamin E and C and calcium content of potato chips after microwave vacuum drying.

Run	Power density (PD)(W/g)	Absolute pressure (AP) (kPa)	Moisture (%)	A_w	Drying time (min)	Vitamin E (mg·100 g ⁻¹ ds)	Vitamin C (mg·100 g ⁻¹ ds)	Calcium (mg·100 g ⁻¹ ds)
1	1.4	7	4.50 ± 0.03	0.253 ± 0.003	21 ± 1	33.81 ± 7.37	295.13 ± 18.09	829.33 ± 1.85
2	1.4	7	6.91 ± 0.19	0.414 ± 0.003	21 ± 1	33.47 ± 1.84	293.75 ± 3.72	807.54 ± 0.38
3	1.1	4	7.13 ± 0.11	0.431 ± 0.003	28 ± 1	32.29 ± 3.49	209.20 ± 5.23	866.84 ± 3.92
4	1.4	4	7.79 ± 0.02	0.480 ± 0.001	20 ± 1	32.68 ± 1.24	236.90 ± 14.63	780.63 ± 2.21
5	1.7	7	3.09 ± 0.02	0.203 ± 0.024	16 ± 1	34.06 ± 2.80	256.82 ± 3.63	784.64 ± 1.02
6	1.4	10	5.95 ± 0.33	0.379 ± 0.003	21 ± 1	37.64 ± 2.88	255.69 ± 3.91	852.91 ± 2.26
7	1.4	7	2.56 ± 0.01	0.194 ± 0.009	21 ± 1	34.25 ± 4.82	288.64 ± 1.90	778.83 ± 7.58
8	1.7	10	8.86 ± 0.03	0.523 ± 0.001	15 ± 1	36.79 ± 3.73	319.30 ± 11.21	812.07 ± 4.17
9	1.4	7	4.93 ± 0.04	0.110 ± 0.001	21 ± 1	31.28 ± 1.50	285.22 ± 7.33	809.08 ± 8.82
10	1.1	10	5.67 ± 0.22	0.384 ± 0.001	28 ± 1	37.08 ± 0.98	281.99 ± 2.07	847.79 ± 2.09
11	1.7	4	8.49 ± 0.19	0.524 ± 0.002	15 ± 1	35.02 ± 8.76	232.76 ± 3.97	840.10 ± 1.15
12	1.1	7	2.90 ± 0.11	0.122 ± 0.01	28 ± 1	34.60 ± 2.06	234.84 ± 13.21	821.49 ± 4.87
13	1.4	7	6.69 ± 0.23	0.415 ± 0.001	21 ± 1	31.70 ± 0.68	237.06 ± 3.85	780.20 ± 7.62
14	1.4	7	4.00 ± 0.08	0.290 ± 0.001	21 ± 1	31.36 ± 2.37	284.68 ± 13.70	818.82 ± 8.94

pressure in the experiment design (Table 1) and the end of the process was determined by a preliminary approach attempting to reach a constant mass. In that sense, the end of the process was fixed when on-off cycles lasted less than 5 s, which implies that the samples' surface temperature was rising rapidly.

The fortified and dehydrated potato chips were packed in black flexible bags of PET/foil/LDPE caliber 78, suitable for contact with food (Alico SA, Medellin, Colombia), for further analysis.

2.3.1. Moisture content and water activity of chips

The moisture content of potato chips was determined according to the AOAC method, and the water activity was measured at 25 °C using a dewpoint hygrometer (Equipo AQUALAB-PRE) (AOAC, 2000).

2.3.2. Determination of calcium (Ca), vitamin C (VitC), and vitamin E (VitE) in fresh potato, impregnated potato, and dehydrated potato chips

The calcium analysis was performed by atomic absorption (AOAC,

2000) where samples were previously calcined for 4 h at 550 °C and digested with nitric and hydrochloric acid. Vitamin C was determined following the method used by Contreras-Calderón et al. (2011), based on titration of the analyzed sample with a 2,6-dichlorophenolindophenol standard solution in an acid environment. Finally, vitamin E was determined by high-performance liquid chromatography (HPLC, Knauer Azura®). The HPLC system was equipped with an autosampler and UV/Vis detector set to 285 nm. The separation was carried out at 30 °C using a reverse-phase Eclipse Plus C₁₈ column (4.6 mm × 250 mm × 5 µm), a mobile phase composed by methanol (53%), acetonitrile (45%), water (2%) and a flow rate of 0.8 ml min⁻¹. The quantification of Vitamin E was carried out from the calibration of Vitamin E standard (DL-α-tocopherol acetate, Sigma-Aldrich®).

2.3.3. Determination of antioxidant capacity of raw potato and dehydrated potato chips

The antioxidant capacity was evaluated for raw potato and potato chips by two methods: FRAP and ORAC. Firstly, the extraction of antioxidant compounds was performed following the method described by Duarte et al. (2017). 1 g of sample was dissolved in 8 mL of a methanol/water (50:50) solution. The sample was vortexed gently with a BV1000 Vortex Mixer (Benchmark Scientific, Edison, NJ) for 1 min and then placed in a shaker (Incubator 1000, Heidolph Instrument GmbH&Co. KG, Schwabach, Germany) at 25 °C for 1 h. The mixture was centrifuged at 5000 rpm for 10 min and the supernatant was recovered and stored in the dark. The precipitate was mixed with an acetone-water solution (70:30) and treated by the same process as before. All the supernatant was mixed in a 25 mL volumetric flask and stored at -20 °C, overnight, before analysis.

- FRAP (Ferric Reducing Ability of Plasma) assay

FRAP assay was made under the methodology described by Contreras-Calderón et al. (2016). FRAP reagent 900 µl (containing TPTZ, FeCl₃, and acetate buffer), freshly prepared and warmed at 37 °C in a water bath (Mettler), was mixed with 90 µl distilled water and 30 µl of the test sample, diluted appropriately with water, or Trolox standard and incubated at 37 °C/30 min. Maximum absorbance values at 595 nm were measured after 30 min (UV-3300 Mapada Instruments, Shanghai, China). Aqueous solutions of Trolox concentrations (between 0 and 500 µM) were used for making a calibration curve. Results were expressed as micromol of Trolox equivalents (TE) per gram of dry solids (µmol TE g⁻¹ ds), as the mean values ± standard deviation (SD) of three measurements.

- ORAC (Oxygen Radical Absorbance Capacity) assay

The ORAC assay was carried out according to the methodology described by Jiménez et al. (2015). 2,2'-azobis(2-amidino-propane) dihydrochloride (AAPH) was used as a peroxy radical generator, and Trolox was used as a standard. Moreover, fluorescein was used as the fluorescent probe. Fluorescein, AAPH, and the samples were prepared in a 75 mM phosphate buffer at pH 7.4. The samples were mixed with 1 M fluorescein (1:6), in black 96-well microplates for 30 min at 37 °C. Subsequently, 250 mM AAPH in PBS (Phosphate Buffer Solution) was added at a 1:7 ratio. Fluorescein intensity was measured every minute for 120 min at excitation and emission wavelengths of 485 and 520 nm, respectively, in a Spectra Max Gemini EM – Molecular devices – (Orleans, USA). The relative ORAC values were calculated using the differences of areas under the decay curves and the results were expressed as micromol of Trolox equivalents (TE) per gram of dry solids (µmol TE g⁻¹ ds), as the mean values ± standard deviation (SD) of three measurements.

2.3.4. Determination of available lysine in raw potato and dehydrated potato chips

The available lysine (lys) was determined by HPLC as the N-ε-dinitrophenyl-lysine (N-ε-DNP-lysine) obtained after acid hydrolysis of potato proteins and formed by the reaction of the lysine free amino group with the fluorodinitrobenzene (FDNB), according to Gómez-Narváez et al. (2019) methodology. Briefly, a sample of potato equivalent to 4 mg of protein was transformed through derivatization by adding 1-fluoro-2,4-dinitrobenzene (FDNB). Hydrolysis of FDNB derivative was conducted using HCl 7.95 N. The analysis was performed using an HPLC (Shimadzu, Kyoto, Japan), with a quaternary gradient pump (LC-20 CE), a diode array detector (SPD-M20A), and autosampler (SIL-20A HT). A Merck LiChrospher 100 RP-18 (5 µm) in LiChroCART 250-4 chromatographic column was used; the injection volume was 20 µL and the mobile phase Methanol – Sodium Acetate 0.01 M pH 5 (1:1), with a flow rate of 1.0 ml min⁻¹. The wavelength of the detector was 360 nm. Working standard solutions (2–10 mg ml⁻¹) were prepared by diluting stock standard solutions in 0.01 M sodium acetate (pH 5). Triplicate analyses of each sample were carried out (*n* = 3). Results were expressed as mg lys-100 g⁻¹ ds.

2.3.5. Textural analysis of dehydrated potato chips

A TA.XT-plus Texture Analyzer (Stable Micro Systems Ltd., Vienna Court, Surrey, UK) was used to measure the hardness and crispness work of potato chips. The analysis was carried out according to the method described by Huang et al. (2011) with the following modifications: a slice of dried potatoes was punctured with a 0.25 in. (6.35 mm) diameter spherical probe. The pre-speed, post-speed, and the test speed were set as 2 mm/s. The penetration distance was set to 10 mm and the trigger force was 20 g. In the penetration test, hardness was the maximum force required to break the sample. Crispness work (Wc) was calculated according to Azzollini et al. (2018) and can be interpreted as the sensory parameter of fracturability that describes the work required to fracture one pore or a group of pores. These measurements were made at room temperature (25 ± 2 °C) and eight samples were used in each treatment.

2.4. Scanning Electron microscopy (SEM)

A Scanning Electron Microscopy (JEOL JSM-64990LV) was used to investigate the microstructure of potato chips. The samples were fixed to stubs through adhesion with double-sided graphite tape and then coated with gold to improve conductivity (Duarte-Correa et al., 2019).

2.5. Stability of added components in chips produced under optimal conditions

A batch of potato chips was prepared according to the optimal drying conditions and was packed in black flexible bags of PET/foil/LDPE caliber 78, with two internal atmospheres: without (0% N) and with Nitrogen (100% N). In the case of 0% N, the potato chips were packed in normal air conditions (Brimblecombe, 1996). All bags were stored at 25 ± 2 °C. The content of calcium, vitamin E and vitamin C was evaluated every 3 weeks during 3 months, according to the methodology previously described in Section 2.4.2. A different bag was used for each analysis.

2.6. Sensory analysis

The sensory study of potato chips was conducted in the Food Sensory Analysis Laboratory at the Universidad de Antioquia, Colombia, by a multidimensional profile approach (ICONTEC, 1996b; ISO 11035:1994) (ICONTEC, 1996a) with an expert panel of five judges. They were able to associate descriptions of odor, taste, and texture, to give the maximum information about the sensory attributes of the samples. The scale used to qualify intensities of the descriptors

was from 0 to 5, where 0 is absent, 1 is very weak, 2 is weak, 3 is moderate, 4 is strong, and 5 is intense. The evaluation was carried out in individual cubicles with an ambient temperature of $24.0 \pm 0.2^\circ\text{C}$ and relative humidity $60 \pm 1.0\%$. The overall quality was also evaluated on a scale from 1 to 3, where 1 is low, 2 is medium and 3 is high. The authors declare that, in Colombia, an Ethics Committee does not need approval for Sensory Analysis. Nevertheless, these analyses were carried out following international tenets and informed consents were obtained.

2.7. Statistical analysis

The evaluation of the Microwave Vacuum Drying was carried out using a response surface methodology and a Central Composite experimental design with six central points (14 experiments).

(Table 1). The independent variables were Power Density (PD) (1.1–1.7 W/g) and Absolute Pressure (AP) (4–10 KPa). On the other hand, the dependent variables were: drying time, moisture, water activity, available lysine, hardness and crispness work, antioxidant capacity; and the content of added components (calcium and vitamins C and E). The response variables were investigated by using a response surface methodology (RSM) and fitting the experimental values to the following second-order equation (Eq. 1):

$$Y = b_0 + \sum_{i=1}^2 b_i X_i + \sum_{i=1}^2 b_{ii} X_i^2 + \sum_{i < j=1}^2 b_{ij} X_i X_j \quad (1)$$

where Y , is the response variable, b_0 is a constant, b_i are model coefficients linked to a linear effect, b_{ii} are the coefficients related to the quadratic effect, b_{ij} are the constants for the interaction effect, and X_i and X_j are the independent variables.

The analysis of variance (ANOVA) was developed with a 95% confidence level, which includes the statistical significance of each of the terms of the adjusted model (p -value), the coefficients estimated in each term (α_i), the model's coefficient of determination (R^2), and the lack of fit in order to establish the accuracy of the model to represent the data. The data and RMS were analyzed and performed using STATGRAPHICS Centurion XVI software (Statistical Graphics Corporation, Version 16.0.07, Rockville, USA).

3. Results and discussion

3.1. Fortification process

The analysis in the fresh potato allowed observing that the content of vitamin E is null in the raw material (Fig. 2). On the other hand, the

potato has a good content of calcium (45.9 mg/100 g⁻¹ ds) and vitamin C (26.4 mg/100 g⁻¹ ds). These values are similar to reported by Andre et al. (2007), who found calcium levels between 27.1 and 109.3 mg Ca/100 g⁻¹ ds and Vitamin C levels between 21.7 and 68.94 mg VitC/100 g⁻¹ ds, for different Andean cultivars. On the other hand, these authors reported vitamin E values between 0.27 and 2.08 mg/100 g⁻¹ ds for 23 native Andean potato cultivars.

Regarding impregnated products, the fortification through the vacuum impregnation process led to obtaining 854.4 mg of Ca/100 g⁻¹ ds, 472.8 mg of Vit C/100 g⁻¹ ds and 47.0 mg of VitE/100 g⁻¹ ds, which demonstrates the feasibility of the technique. Likewise, Hironaka et al. (2011), fortified whole potatoes with ascorbic acid using vacuum impregnation (VI) and observed that these samples consisted of ascorbic acid content ten times higher than samples without fortification. Tiwari et al., (2018) fortified potato with calcium and found that fortified potato chips had 4.5 and 7.1 times higher calcium content than that of control and commercial counterparts, respectively. Erihemu et al. (2015), impregnated two cultivars of potatoes with iron using the same technique, observed that fortified potatoes attained 6.4 and 6.6 times more iron content, for the two cultivars used. Also, Hironaka and Koaze (2015) fortified two cultivars of potato with zinc through vacuum impregnation, attaining 61 and 45 times more zinc content than the control, for the two cultivars.

3.2. Microwave vacuum drying results

3.2.1. Moisture content, water activity and drying time

The value obtained for the chips moisture ranged between 2.56% and 8.86% (Table 1), being greater than the one reported for potato chips obtained by conventional frying, where humidity is generally 2% (Pedreschi et al., 2018). However, Huang et al. (2011) found that for restructured mixed potato with apple chips obtained by four different drying methods the final moisture content about 6%. Also, a large variation of the final moisture content was found by Kingcam et al. (2008) working with potato dried chips with an initial thickness of 3.5 mm. Therefore, it can be explained by the thickness but also may be due to the location of the food in the microwave oven that can lead to no uniform heating, allowing for less than ideal interaction of food components (Schubert and Regier, 2005).

On the other hand, water activity was in the range of 0.110 to 0.524. This range covers a condition of enzymatic activity where hydrolytic and oxidation reactions could also occur. However, since the enzymes were inactivated during the blanching process and due to the low-fat content of potato (Singh and Kaur, 2016), it is unlikely that these phenomena will occur, resulting in products with good stability (Barbosa-Cánovas and Ibarz, 2003). Finally, moisture and water

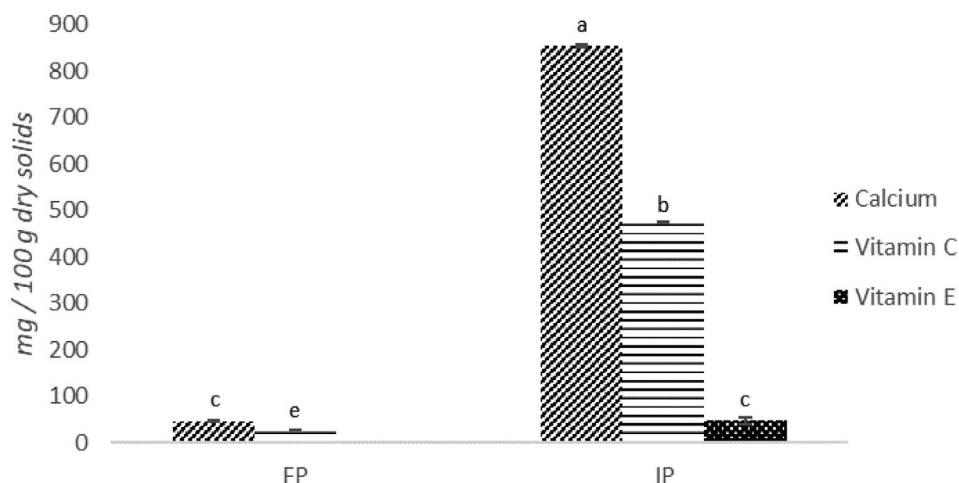


Fig. 2. Calcium, Vitamin C and Vitamin E contents in fresh (FP) and impregnated (IP) potato.

Table 2
Results of ANOVA of response variables determined after microwave vacuum drying process.

Response variable	A:power density, PD (W/g)	B:Absolute pressure, AP (kPa)	AA	P-value	BB	R ²	Lack of fit
				AB			
Moisture	0.296	0.504	0.502	0.605	0.024	63.45	0.533
Water activity	0.340	0.637	0.560	0.857	0.027	64.58	0.741
ORAC	0.296	0.587	0.122	0.245	0.049	48.60	0.212
FRAP	0.430	0.147	0.016	0.574	0.357	62.62	0.283
Hardness	0.130	0.206	0.844	0.614	0.008	65.69	0.113
Crispness work	0.669	0.477	0.908	0.020	0.036	76.87	0.652
Drying time	0.000	0.195	0.000	1.000	0.019	99.72	0.222
Vitamin E	0.587	0.017	0.283	0.311	0.075	77.61	0.601
Vitamin C	0.182	0.021	0.410	0.765	0.427	59.07	0.220
Calcium	0.106	0.636	0.398	0.836	0.096	47.71	0.149
Available lysine	0.045	0.871	0.123	0.851	0.054	65.90	0.184

AA, AB and BB are the interactions of the factors on the analyzed responses.

ORAC (Oxygen Radical Absorbance Capacity) and FRAP (Ferric Reducing Ability of Plasma) are the antioxidant capacities.

content were correlated significantly positive ($p < .05$), through Pearson's correlation with $R^2 = 91\%$.

Meanwhile, drying time varied between 15 and 28 min, significantly affected by power density (PD) ($p < .05$) (Table 2). The longest drying time was obtained with the lowest power density, attributable to the higher material load and therefore higher water content to be removed. The same behavior has been reported previously for potato slices by Darvishi (2012), who reported drying times between 2.5 and 23 min, using power densities that varied from 5 to 20 W·g⁻¹, respectively. This behavior was observed also for many other food-stuffs, such as vegetable soybean (Hu and Zhang, 2006) and lotus seeds (Jiang et al., 2017).

Various power density values have been used in microwave drying processes, in the range of 0.75–20 W·g⁻¹, where, in general, it is reported that higher power density leads to faster water removal, with the consequent less drying time; since, the evaporation rate is a function of the microwave power density (W·g⁻¹) (Ambros et al., 2018; Darvishi, 2012; Huang et al., 2011; Jiang et al., 2017; Scaman et al., 2014; Zielinska et al., 2017).

3.2.2. Calcium, vitamin C and vitamin E contents

Calcium, vitamin C, and vitamin E contents (mg·100 g⁻¹ ds) for all microwave vacuum drying conditions evaluated were in the range of 778–866 mg, 209–295 mg, and 31–37 mg, respectively (Table 1). For calcium, in all experiments, it was obtained a retention percentage higher than 90%, similar to that obtained by Oladele and Abonsade (2009), who reported retention between 91 and 95% in spinach drying through sun and convective drying, and higher than other reported by Lima et al. (2019) (~51%) working on microwave drying of mustard greens. These authors attributed these results to the fact that during microwave radiation, leaf cells are more affected, thus releasing a higher mineral content.

In the case of vitamin C, a lower retention percentage was obtained (44–62%). This occurred because Vitamin C is the least stable of all vitamins and it can be easily degraded during processing (Herbig and Renard, 2017). Water content is of great importance in the reduction of vitamin C (Khraisheh et al., 2004), as the moisture content decreases, the degree of dilution decreases and, thus, the reaction rate increases to give lower vitamin C retention. In addition, Khraisheh et al., (2004) found that such phenomena are more apparent during microwave processing as a wider range of moisture levels can be achieved, as we found in this work. Likewise, the thermolabile nature of vitamin E is previously documented (Badui Dergal, 2006; Eitenmiller and Lee, 2004; Litwack, 2007), although the esterification process improves α -tocopherol stability, the molecule is still liable to degradation due to environmental sensitivity to oxygen and temperature (Pereira et al., 2015). In this case, the retention percentages for Vitamin E varied

between 66 and 79%.

Vitamins E and C contents were significantly affected by absolute pressure (AP) ($p < .05$) (Table 2), the higher absolute pressure (i.e. lower vacuum applied), the greater retention of vitamins. Although it has been reported that the use of vacuum helps to preserve these compounds (Barbosa-Cánovas and Ibarz, 2003), the impact of chamber pressure in microwave drying is really discussed controversially and may be system-specific (Ambros et al., 2018; Bondaruk et al., 2007; Cui et al., 2004; Kraus et al., 2013; Scaman et al., 2014). The lower the pressure in the drying chamber, the greater the driving force for water vapor to diffuse from the product. However, as pressure decreases, the latent heat of vaporization increases slightly, which contributes to a slight decrease in the rate of drying (Scaman et al., 2014). Thus, we hypothesize that the second effect may be predominant, which could conduce to longer process time and probably greater loss of compounds. However, the vitamin losses can also be explained from the fact that higher vacuum degrees normally would give rise to glow discharges or electrical arcing in the cavity, and cause local overheating and irreversible damages to the product (Hu and Zhang, 2006). Therefore, relatively higher vacuum degrees (less absolute pressure) could cause conditions that significantly affect product quality. Assis et al. (2019), working with fortified apple snacks, found that vacuum impregnation followed by vacuum pulse applications reduced to some extent the calcium concentration, but all processes raised the calcium content into the dried apple. A wide range of absolute pressure has been used in microwave drying processes (1–20 kPa), however, its effect is less evaluated in relation to other process variables such as power and temperature (Ambros et al., 2018; Huang et al., 2011; Jiang et al., 2017; Kowalska et al., 2018; Lefort et al., 2003; Setiady et al., 2009a, b; Zielinska et al., 2017). Some studies have attributed the conservation of compounds, during MVD, to the use of low temperatures, rather than the pressure effect.

3.2.3. Antioxidant capacity of raw potato and dehydrated potato chips

Two different methods were used in order to evaluate the antioxidant capacity of raw potato and potato chips. FRAP method is based on Single Electron Transfer (SET) mechanism, while ORAC method is based on Hydrogen Atom Transfer (HAT) reactions (López-Cobo et al., 2014). The FRAP values found were $12.26 \pm 1.84 \mu\text{mol TE}\cdot\text{g}^{-1}$ ds for fresh potato and $14\text{--}19 \mu\text{mol TE}\cdot\text{g}^{-1}$ ds for potato chips (Table 3). Kita et al. (2013) reported FRAP values for raw potatoes of $16.7\text{--}35.7 \mu\text{mol TE}\cdot\text{g}^{-1}$ ds and for chips $7.6\text{--}24.1 \mu\text{mol TE}\cdot\text{g}^{-1}$ ds, indicating that the degree of reduction was cultivar dependent. In the present case, the FRAP values obtained for the chips were higher than the fresh potato, which can be attributed to the increase in vitamin C achieved with impregnation. A multivariate analysis (data not shown) allowed to establish a positive correlation between FRAP and Vitamin C ($p < .05$).

Table 3

Antioxidant capacities, available lysine, hardness and crispness work of potato chips after microwave vacuum drying.

Run	Power density (PD) (W/g)	Absolute pressure (AP) (kPa)	ORAC ($\mu\text{mol TE/g ds}$)	FRAP ($\mu\text{mol TE/g ds}$)	Available lysine ($\text{mg} \cdot 100 \text{ g}^{-1} \text{ ds}$)	Hardness (N)	Crispness work ($\text{N} \cdot \text{mm}$)
1	1.4	7	24.48 $\pm$ 0.12	19.86 $\pm$ 3.04	259.08 $\pm$ 4.87	8.06 $\pm$ 1.71	0.42 $\pm$ 0.12
2	1.4	7	20.81 $\pm$ 0.33	19.08 $\pm$ 0.69	200.70 $\pm$ 0.70	8.08 $\pm$ 1.51	0.67 $\pm$ 0.18
3	1.1	4	20.67 $\pm$ 0.18	14.39 $\pm$ 0.26	258.34 $\pm$ 2.85	9.46 $\pm$ 2.14	0.89 $\pm$ 0.28
4	1.4	4	13.22 $\pm$ 0.17	17.76 $\pm$ 1.82	279.66 $\pm$ 8.15	7.99 $\pm$ 2.19	0.56 $\pm$ 0.17
5	1.7	7	23.78 $\pm$ 0.23	14.58 $\pm$ 0.07	268.56 $\pm$ 2.14	7.44 $\pm$ 2.00	0.51 $\pm$ 0.22
6	1.4	10	17.29 $\pm$ 0.00	17.77 $\pm$ 1.18	299.84 $\pm$ 4.74	9.57 $\pm$ 2.63	0.75 $\pm$ 0.12
7	1.4	7	18.36 $\pm$ 0.16	18.47 $\pm$ 2.70	240.76 $\pm$ 5.52	8.02 $\pm$ 1.52	0.47 $\pm$ 0.11
8	1.7	10	14.91 $\pm$ 0.18	18.11 $\pm$ 1.69	353.73 $\pm$ 7.93	8.97 $\pm$ 2.13	0.93 $\pm$ 0.28
9	1.4	7	21.73 $\pm$ 0.29	17.22 $\pm$ 0.56	270.39 $\pm$ 3.38	8.80 $\pm$ 2.07	0.41 $\pm$ 0.13
10	1.1	10	23.70 $\pm$ 0.15	17.69 $\pm$ 1.11	259.37 $\pm$ 7.49	9.13 $\pm$ 1.79	0.53 $\pm$ 0.15
11	1.7	4	18.46 $\pm$ 0.07	16.28 $\pm$ 0.19	363.53 $\pm$ 3.15	8.92 $\pm$ 1.40	0.55 $\pm$ 0.21
12	1.1	7	19.93 $\pm$ 0.18	14.32 $\pm$ 1.21	289.79 $\pm$ 3.20	8.36 $\pm$ 2.19	0.44 $\pm$ 0.12
13	1.4	7	18.09 $\pm$ 0.09	16.53 $\pm$ 0.33	251.60 $\pm$ 2.14	7.70 $\pm$ 1.87	0.55 $\pm$ 0.10
14	1.4	7	18.61 $\pm$ 0.08	18.63 $\pm$ 2.14	211.92 $\pm$ 1.35	8.06 $\pm$ 1.96	0.62 $\pm$ 0.18

This correlation has been previously observed by [Du et al. \(2009\)](#). Additionally, FRAP values of potato chips were higher than those obtained by [Nemš et al. \(2015\)](#): 1.74–13.31 $\mu\text{mol TE} \cdot \text{g}^{-1} \text{ ds}$ for snacks made with colored potato.

Regarding ORAC values, the raw potato and potato chips presented values of $20.04 \pm 1.65 \mu\text{mol TE} \cdot \text{g}^{-1} \text{ ds}$ and $13\text{--}24 \mu\text{mol TE} \cdot \text{g}^{-1} \text{ ds}$, respectively ([Table 3](#)). [Andre et al. \(2007\)](#) reported higher ORAC values ($28.25\text{--}250.67 \mu\text{mol TE} \cdot \text{g}^{-1}$) for different raw Andean cultivars. This was due to the edaphoclimatic differences of each variety, which in turn induce variable contents of natural phytochemicals, such as phenolic compounds, anthocyanins, carotenoids and vitamins which are related to antioxidant capacities ([Yamdeu Galani et al., 2017](#)). In general, for ORAC values, there was no specific pattern and no significant correlation was found (data not shown). [Yang et al. \(2016\)](#) stipulated that the different heat treatments reduced or, in some cases, enhanced the antioxidant capacity of the potato with respect to the uncooked samples.

3.2.4. Available lysine in raw potato and dehydrated potato chips

The available lysine content present in raw potato was $415.5 \pm 1.5 \text{ mg lys} \cdot 100 \text{ g}^{-1} \text{ ds}$. It has been reported that potato proteins are composed, in a high proportion, of peptides rich in lysine ([Bradshaw and Gebhardt, 2007](#); [Singh and Kaur, 2016](#)), presenting values between 310 and 760 $\text{mg lys} \cdot 100 \text{ g}^{-1}$ potato powder ([Peksa et al., 2013](#); [Woolife, 1987](#)). And, the content obtained for potato chips was in the range of $200\text{--}363 \text{ mg lys} \cdot 100 \text{ g}^{-1} \text{ ds}$, which corresponds to losses between 51 and 12%. Several reports have previously linked the loss of lysine with the Maillard reaction ([Contreras-Calderón et al., 2009](#); [Gómez-Narváez et al., 2019](#); [Jouquand et al., 2015](#); [Mogol et al., 2013](#)), since lysine reacts easily with reducing sugars, being one of the oldest known nutritional implications. [Jaswal \(1972\)](#) reported losses of available lysine between 38 and 67% in the preparation of potato chips, whilst [Gorinstein et al. \(1988\)](#) reported losses between 21 and 59%. On the other hand, [Žilić et al. \(2006\)](#) found a decrease in lysine bioavailability corresponding to 69.5% in microwave-heated soybean.

3.2.5. Textural properties of dehydrated potato chips

The texture is often described in terms of crispness and hardness. The characteristic crispy texture is one of the most important quality indicators of the finished product. The hardness values range from 7 to 9 N, whilst Crispness work was between 0.4 and 0.9 $\text{N} \cdot \text{mm}$ ([Table 3](#)). There were no significant differences ($p > .05$) ([Table 2](#)) for the levels of analyzed factors. [Salvador et al. \(2009\)](#) reported that, in addition to the processing conditions, raw potato properties, starch content and final moisture played an important role in the texture formation of the product. However, significant positive correlations were evidenced (data not shown) between hardness and calcium content ($p < .05$) and between the Crispness work and moisture and water activity ($p < .05$).

The first correlation was due to the increase of calcium content through VI process since the pectin is particularly abundant in potato cell walls ([Singh and Kaur, 2016](#)), and it has been reported that calcium establishes bonds with pectins, forming calcium pectates that increase the stiffness of the wall, making the tissue more resistant ([Badui Dergal, 2006](#); [Gras et al., 2003](#)). Similar behavior has been previously described for other food matrices like mango, pineapple, carrots, and eggplant ([Gras et al., 2003](#); [Mateus de Lima et al., 2016](#); [Torres et al., 2008](#)). On the other hand, the crispness work values increasing with moisture and water content indicated that more work was needed to deform/fracture the cells ([Sun, 2012](#)).

3.2.6. Optimization results

Considering that the lack of fit had p -values $> .05$, according to ANOVA results ([Table 2](#)), all dependent variables could be used to adjust to the general model (Eq. (1)). Only the dependent variables with $R^2 > .70$ were considered for model calculations. In that sense, the models that describe the behavior of the crispness work (W_c) ($R^2 = 76.87$), drying time (D_t) ($R^2 = 99.72$) and vitamin E content ($VitE$) ($R^2 = 77.59$) are presented as the Eqs. (2), (3), and (4), respectively.

$$W_c = 3.52 + 0.20 \times PD \times AP + 0.02 \times AP^2 \quad (2)$$

$$D_t = 69.22 - 52.22 \times PD + 11.11 \times PD^2 - 0.05 \times AP^2 \quad (3)$$

$$VitE = 49.18 - 0.96 \times AP \quad (4)$$

where PD is the power density and AP is the absolute pressure in the microwave chamber.

Lastly, these models were used for the optimization process with a multi-response optimization methodology. The maximum desirability (0.87) was achieved with Treatment 11 (see [Table 1](#), Run 11). The optimization results were confirmed by doing an independent experiment using the optimal conditions obtained. Thus, the best treatment was selected as $PD = 1.7 \text{ W} \cdot \text{g}^{-1}$; $AP = 4.0 \text{ kPa}$ to be used in the subsequent drying processes, in order to study the stability of added components and sensory analysis.

3.3. Scanning electron microscopy (SEM)

The Scanning Electron Microscopy of fortified potato chips allowed to observe that the microstructure of product suffered some few changes during fortifying and/or dehydration, observed as the opening of some cells, and loosening of the microfibrils ([Fig. 3a](#)), as reported by [Bordoloi et al. \(2012\)](#). According to [Wang et al. \(2010\)](#), this is more noticeable in blanched potatoes ([Wang et al., 2010](#)). However, most of the potato tuber parenchyma showed retained cell wall layout ([Fig. 3b](#)) and some calcium crystals were observed on the surface of the tissue

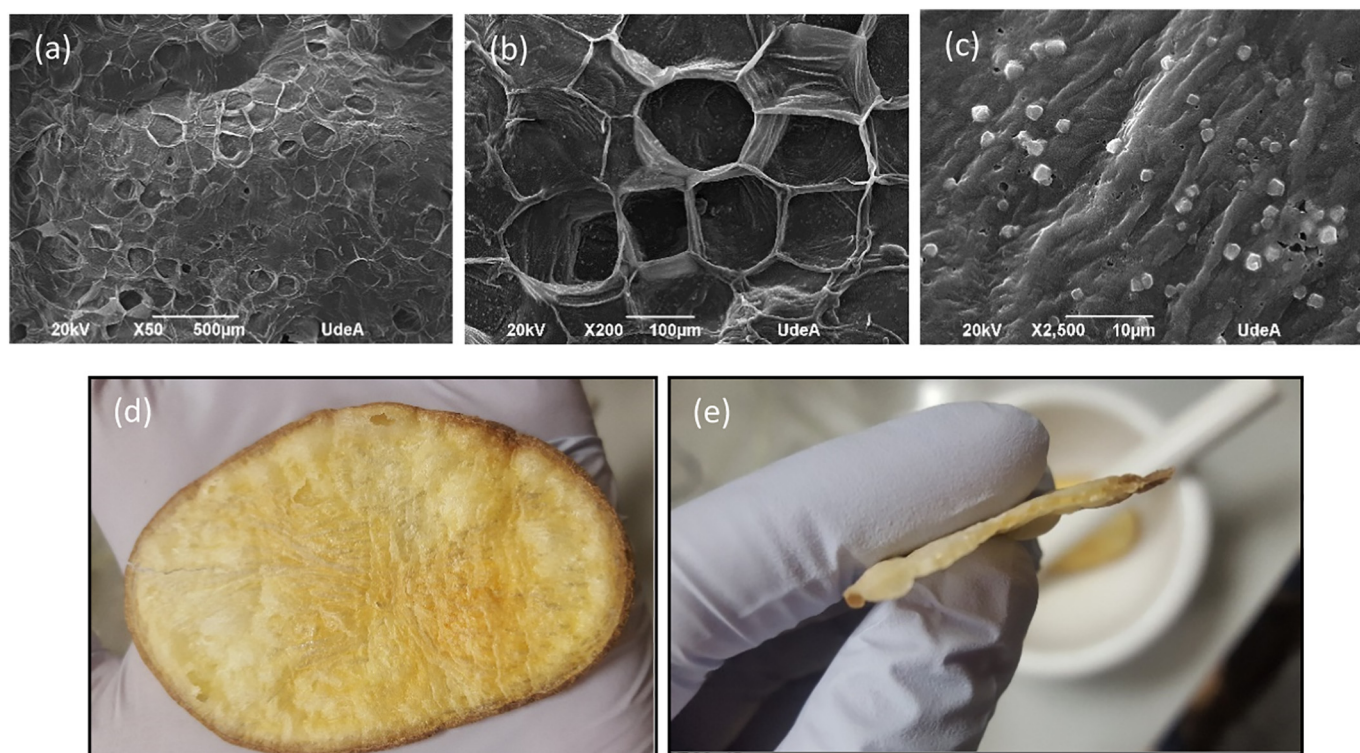


Fig. 3. Scanning electronic micrographs of fortified potato chips at 50 $\times$ (a), 200 $\times$ (b), 2500 $\times$ (c) and photographs of chips produced by microwave vacuum drying under optimal conditions, entire chip (d) and thickness (e).

(Mimouni et al., 2007) (Fig. 3c). According to Barreto et al. (2019), the use of vacuum impregnation combined with blanching can result in very porous chips, which is a very important attribute for the potato texture. That microstructure was not observed in these micrographs (Fig. 3a,b,c), it was possible to observe only a few micrometric porous.

Nevertheless, macroscopically, the microwave vacuum drying produced an effect called “puffing”, which occurs when the evaporation rate inside the product is higher than the transportation rate of steam to the outside the product (Scaman et al., 2014). The resulting over-pressure leads to product expansion (Duarte-Correa et al., 2019). This effect can be seen in some pictures taken of fortified potato chips (Fig. 3d,e).

3.4. Stability of added components

The behavior of the added components during 12 weeks of storage of potato chips packed in an atmosphere with (100% N) and without (0% N) Nitrogen can be observed in Fig. 4. As expected, for both treatments, the calcium content (Fig. 4a) remained practically constant throughout the storage period. The differences found in calcium content can be attributed to the impregnation process itself, more than to storage time. Regarding the Vitamin C content (Fig. 4b), no significant differences ($p > .05$) between both treatments (100% N and 0% N) were found, which implies that it is not necessary to use a Nitrogen atmosphere to reduce the ascorbic acid oxidation. On the other hand, the higher levels obtained at the end of the storage may be the reflection of variations in the impregnation levels, as in the case of calcium content. Thus, we can suggest that the vitamin C content remained constant during the 3 months, around 275 mg/100 g⁻¹ ds. Hironaka et al. (2011) impregnated whole potatoes with ascorbic acid through VI, and reported losses of approximately 37% after 14 days of storage, estimating that potatoes with a high content of ascorbic acid can result in a marked loss of this vitamin. However, the results obtained in this study might suggest that subjecting fortified potatoes to stabilization after impregnation (i.e. drying), could result in more effective

protection for the added components.

Finally, vitamin E content (Fig. 4c) decreased during 9 weeks, but showing a slightly higher value at week 12, resulting in losses of 29 and 32% ($p < .05$) for the 100% N and 0% N treatments, respectively. As in previous cases, these differences can be attributed mainly to the impregnation process. Cortés et al. (2007) evaluated the stability of vitamin E content in fortified and dehydrated apple and reported losses of 28 and 37% during 180 days of storage at 20 and 30 °C, respectively. They verified high variability, attributing this behavior to differences in impregnation levels, fluctuations in oxidation processes and the tocopherol extraction process.

Finally, it is worth to mention that the calcium and vitamins C and E contents at the end of the storage period were even higher than those found in fresh potatoes; thus, vacuum impregnation along with microwave vacuum drying are effective methods to produce fortified low-fat potato snack.

3.5. Sensory analysis

Fig. 5 shows the descriptors found for texture: *hard* (THD), *crispy* (TCP), *dry* (TDR), *fibrous* (TFB), *chewable* (TCW), and *lumpy* (TLM); for flavor: *saline* (SSL), *sweet* (SSW), *starchy* (SST), *fresh* (SFS), *vegetable* (SVG), *acid* (SAC), and *astringent* (SAS), and for odor: *starchy* (OST), *sweet* (OSW), *fresh* (OFS), *saline* (OSL), and *vegetable* (OVG) of potato chips. Regarding flavor, the *astringent* descriptor can be associated with the presence of polyphenolic compounds in the potato (López-Cobo et al., 2014), while *acid* and *saline* descriptors are attributed to the addition in the impregnation of vitamin C and NaCl, respectively. Regarding the odor, potato tubers release a wide range of volatile compounds, and the cooking method dictates the type and extent (Singh and Kaur, 2016). Besides, since both flavor and odor are processed and interpreted in the same region of the brain (Singh and Kaur, 2016) is not surprising that presented similar attributes. Regarding texture, the *crispy* attribute is highlighted because it is a major determinant of potato chip quality (Lefort et al., 2003). Salvador et al. (2009) indicated

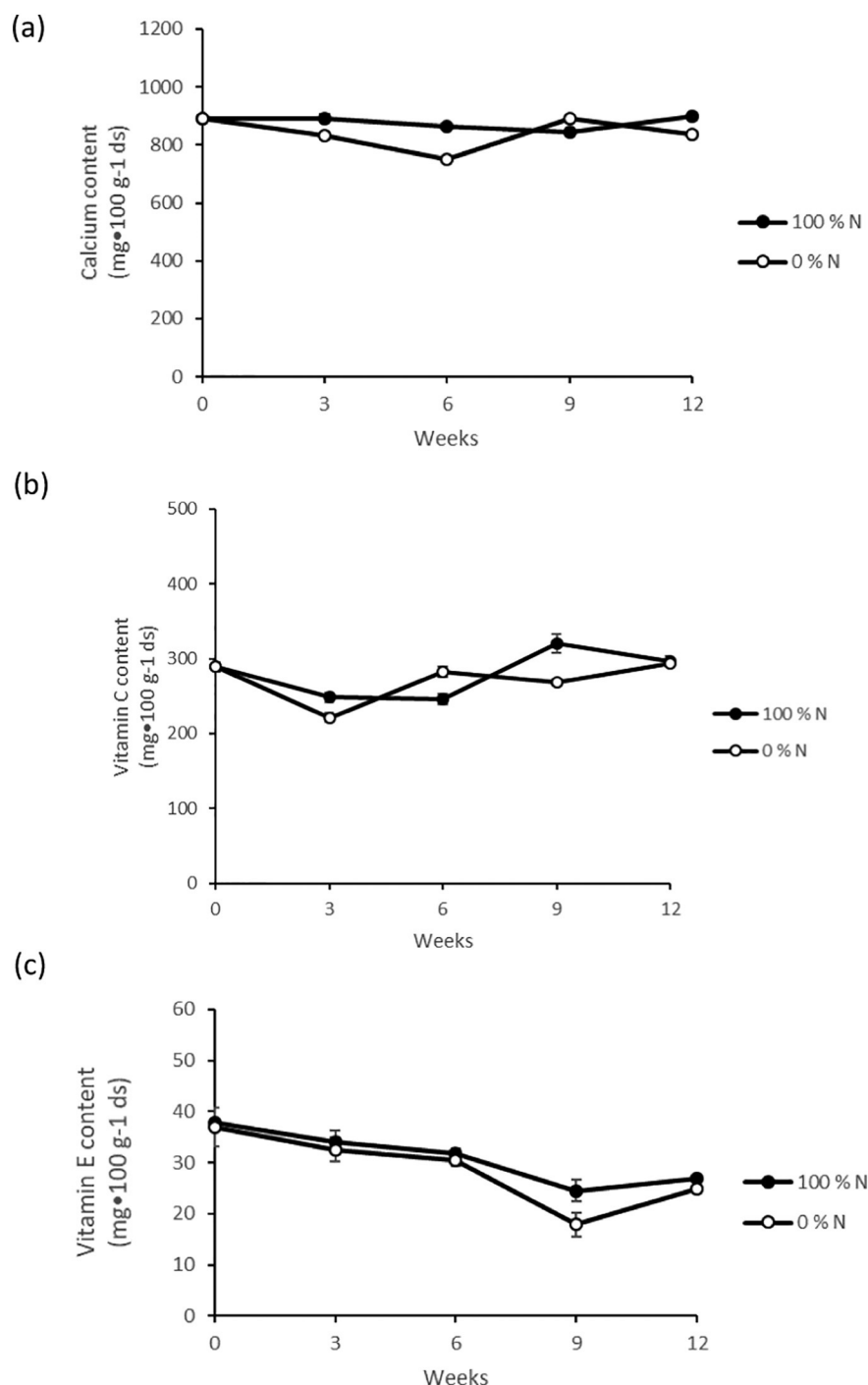


Fig. 4. Stability of calcium (a), vitamin C (b) and vitamin E (c) in potato chips, during 12 weeks of storage at 25 °C.

that a certain degree of sensory hardness is necessary for crispness perception, and in this study, both descriptors *hard* and *crispy* were perceived. In addition, the *hard* descriptor can be partially attributed to the addition of calcium in the impregnation emulsion.

Finally, the overall quality was evaluated as *high*. Likewise, Tiwari et al. (2018) could fortify potato chips at 700 mg·100 g⁻¹ of calcium level with acceptable sensory attributes. Joshi et al. (2016a, b) found that microwave processing was suitable to process the potato chips with acceptable sensory and textural scores (7.6 and 8.0, respectively on a 9.0 point on hedonic scale). Setiady et al. (2009b) dehydrated potatoes with different drying techniques and found that there was a tendency of panelists to prefer the MVD process. Other dehydrated foods produced using MVD and that have been generally acceptable to the consumers

are re-structured mixed potato with apple chips (Huang et al., 2011) and strawberries (Kowalska et al., 2018).

4. Conclusions

It was possible to produce potato chips fortified with calcium, vitamin C, and vitamin E, by using microwave vacuum drying, allowing us to have low-fat snacks. Additionally, the potato snack produced had high sensory acceptance. The optimal microwave vacuum drying conditions for the potato chips production were a power density of 1.7 W·g⁻¹ and absolute pressure of 4.0 kPa, with the desirability of 0.87. The power density had a significant effect on the drying time: the longest drying time was observed with the lowest power density, as

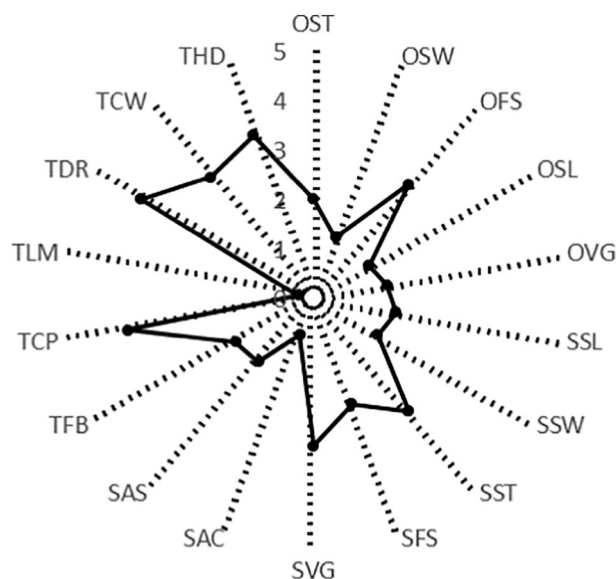


Fig. 5. Sensorial multidimensional profile of potato chips produced by microwave vacuum drying under optimal conditions.

expected. For its part, the absolute pressure had an important effect on vitamin C and vitamin E contents: higher absolute pressure, higher the retention of the vitamins. Although the use of a higher vacuum caused more vitamin losses, all processes elevated the vitamin content into the chips. However, it means than the MVD of vitamin-fortified potatoes can be done to a relatively higher absolute pressure to prevent quality damage, with the consequent energy savings. In conclusion, the coupled vacuum impregnation and microwave vacuum drying processes were able to produce fortified dried potato snacks.

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