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OPTIMIZATION OF THE OSMOTIC DEHYDRATION PROCESS FOR IMPROVING THE QUALITY OF VACUUM-FRIED "VINH CHAU" SHALLOTS AT THE PILOT STAGE

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ABSTRACT

Background. Osmotic treatment is necessary to reduce the oil on the vacuum-fried product. However, the nutrition of the treated sample is also affected, especially bioactive compounds.
Materials and methods. The Box-Behnken model was applied to evaluate the change in bioactive compounds of vacuum-fried shallots during osmotic dehydration in the pilot-scale plan. Three factors of osmotic treatment were examined, namely the concentration of maltodextrin (30–50%), concentration of citric acid (0.1–0.2%), and dehydration time (15–45 minutes). The frying conditions were constantly set at a temperature of 120 ±1°C with a vacuum of 680 mmHg for 8 minutes.
Results. The results showed that all factors significantly affected the content of phenolics and flavonoids in the sample. The optimal condition of the dehydration process was found to be 38.9% maltodextrin and 0.17% citric acid in the soaking medium, and the soaking time was 31.9 minutes. The sample's total phenolic and flavonoid content in optimal conditions were 12.44 mgGAE/g and 1841.73 µgQE/g, respectively.
Conclusion. The quality of vacuum-fried shallots was greatly affected by osmotic dehydration conditions. It was also found that under optimal conditions the biological compounds of the product are still maintained at a high level. Further studies on the techno-economic value of the product for commercialization should be considered.

Keywords: vacuum frying, shallot, osmotic, bioactive compound, Box-Behnken model

INTRODUCTION

In recent years, particularly during the Covid-19 pandemic, food with high biological activities, which might help to enhance the immune system and reduce the risk of disorders, has attracted public attention (Bellavite and Donzelli, 2020). The onion (*Allium ascalonicum*) is one such plant with high antioxidant compounds, including phenolics, flavonoids, kaempferols, and quercetin (Thuy et al., 2020). These compounds have health benefits, such as preventing cancer, anti-microbiological activities, strengthening cardiovascular health, reducing high blood pressure and insulin resistance, controlling weight, antioxidant activity, and fighting chronic bronchitis, infections, and fever (Donno et al., 2019; Paczkowska-Walendowska et al., 2021). However, in Vietnam, the shallot is not used effectively, and the price of these materials

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has fluctuated in recent years. The economic value of agricultural ingredients could be increased thanks to converting raw materials into processed products. One of the exciting techniques that might be applied is vacuum frying. Vacuum frying technology is widely used for many foods, especially fruits and vegetables (Moreira, 2014). Recent studies show that this technology has been applied to such foodstuffs as apples, bananas, jackfruit, kiwi, carrots, mushrooms, sweet potatoes, and tapioca (Belkova et al., 2018; Dueik et al., 2010; Mariscal and Bouchon, 2008). Vacuum frying is used for frying fruits and vegetables under atmospheric pressure, preferably below 50 Torr (6.65 kPa). This lowers both the boiling point of frying oil and the moisture level in the food and produces better-quality fried foods with a lower oil content when compared with atmospheric frying (Andrés-Bello et al., 2011). In general, one of the earliest and most widely used methods of cooking, deep fat frying, involves submerging the material under atmospheric pressure into hot oil (150–200°C). Meanwhile, vacuum frying works at a reduced temperature (100–144°C) (Manzoor et al., 2022; Moreira, 2014).

Osmotic dehydration is an essential step in vacuum-frying processing and is a pre-treatment measure to help raw materials reduce moisture and produce low-fat products (Su et al., 2021). Osmotic dehydration is the removal of water from a lower concentration of solute to a higher concentration through semi-permeable membrane and results in the equilibrium condition on both sides of the membrane (Nguyen et al., 2022). Furthermore, maltodextrin has been used in the soaking process, and applied in the production of vacuum-fried mango products, significantly improving the product structure (Diamante et al., 2011). This process can also be combined with citric acid to limit the color change in the fried products (Diamante et al., 2011). A preliminary study was conducted to find the effects of osmosis on the bioactive compounds in vacuum-fried shallots; optimal conditions have not been (Thủy et al., 2016). To optimize the pre-treatment condition of vacuum-fried shallots, the effects of the process parameters on the product quality must be clearly understood. Modelling can be one of the methods for expressing this relationship between process parameters and quality factors. With experimental data, empirical models can be developed (Thuy et al., 2021a). Although empirical models purely rely on data, it is suited for the simulation of food processes. One of the primary advantages of response surface methodology (RSM) is that a large amount of information is obtained from a limited number of experiments. Various studies have applied the Box-Behnken model as one of the model designs of RSM to determine the optimal value of different processing methods (Thuy et al., 2021b; Thuy et al., 2022a; Thuy et al., 2022b; Van Tai et al., 2021). Therefore, the study was conducted to determine the optimum parameters for the osmotic dehydration of raw materials to produce vacuum-fried shallot products with a high-content biological compound.

MATERIALS AND METHODS

Preparation of vacuum-fried shallot

Shallots were harvested at Vinh Chau district (Soc Trang, Vietnam), peeled and the bulk collected. Raw shallot bulk was initially sliced 2 mm, soaked in 0.05% $CaCl_2$ solution for 30 minutes, and blanched at 85 ±1°C for 1.5 minutes (Thủy et al., 2016). The sample was then soaked with the prepared solutions as an experimental setup. After soaking, the ingredients were frozen at –20°C for 24 hours before vacuum frying. Shallot is fried in a vacuum fryer (FS-251, South Africa with a maximum frying capacity of 25 kg/batch) with a vacuum of 680 mmHg at 120 ±1°C for 8 minutes (each treatment used 10 kg of material). Products were obtained and analyzed for quality (total phenolic and flavonoid content).

Chemicals and reagents

Gallic acid, quercetin and Folin-Ciocalteu were purchased from Sigma-Aldrich (USA). The deionized water and other chemicals used in this study were analytical reagent grade.

Experimental design

Based on the preliminary research by Thủy et al. (2016), the optimization experimental setup was conducted with three factors, including the concentration of maltodextrin (A, %), the concentration of citric acid (B, %), and soaking time (C, minute). The level of each factor ranged from –1 to +1; 0 was the central point, as shown in Table 1. The Box-Behnken model was

Table 1. Level of independent variables used in the Box-Behnken design for osmotic dehydration

Variables	Level of each factor		
	–1	0	+1
Concentration of maltodextrin (A), %	30	40	50
Concentration of citric acid (B), %	0.1	0.15	0.2
Soaking time (C), min	15	30	45

applied to find the optimal pre-treatment conditions, six repetitions at the center point were conducted, and 18 runs were investigated (Table 2). The sample was obtained from each run, which further analyzed the content of bioactive compounds.

In order to validate the adequacy of the model equation, a verification experiment was carried out under optimal conditions. A comparison between experimental and actual data of TPC and TPC was conducted.

Table 2. Osmotic dehydration operating variables based on the Box-Behnken model design

Run	Factor A	Factor B	Factor C	Run	Factor A	Factor B	Factor C
1	40	0.15	30	10	40	0.15	30
2	40	0.2	15	11	40	0.15	30
3	40	0.2	45	12	40	0.15	30
4	50	0.15	45	13	40	0.1	45
5	40	0.1	15	14	40	0.15	30
6	50	0.15	15	15	30	0.2	30
7	30	0.1	30	16	30	0.15	45
8	50	0.2	30	17	40	0.15	30
9	50	0.1	30	18	30	0.15	15

Quality analysis

Total phenolic content (TPC) was based on the absorbance of the reaction between polyphenols and the Folin-Ciocalteu assay (Hossain et al., 2013). The absorbance was measured at a wavelength of 738 nm. TPC was calculated as mg of gallic acid equivalents (GAE) per gram of dry matter [mg GAE/g dry weight (DW)] through the obtained standard curve: $y = 0.025x + 0.0632$ ($R^2 = 0.9957$), where: y is the absorbance value, and x is TPC.

A yellow solution was obtained after flavonoid compounds reacted with aluminum chloride (10%) form (Mandal et al., 2013). Based on the level of absorbance at a wavelength of 415 nm, the content of flavonoids (TFC) in the extract was calculated as mg of quercetin equivalents (QE) per gram of dry matter (µgQE/g DW). The quercetin standard curve was obtained from the previous study (Thủy et al., 2016): $y = 0.0005x + 0.059$ ($R^2 = 0.99$), where: y is the absorbance value, and x is TFC.

The sample obtained under optimal conditions was analyzed for lipid content, TPC, TFC, and antioxidant activity. Then, the comparison between the optimal sample and the untreated sample was investigated. The antioxidant activity was measured as Mandal et al. (2013) described. Meanwhile, the lipid content was determined by the AOAC method (AOAC, 2005).

Statistical analysis

Statgraphics centurion XV.I and Design-Expert 13 application fit the model to the actual data using the response surface methodology. The effect of the independent variable on the model for response (Y), was shown in Equation 1.

$$Y = b_o + b_1A + b_2B + b_3C + b_4AB + b_5AC + b_6BC + b_7A^2 + b_8B^2 + b_9C^2 \quad (1)$$

where A, B, C are independent variables, b_o, b_1, b_2, ..., b_9 are intercept, interaction, and squared effects. The selection model was mainly based on the R^2 value obtained from the regression.

RESULTS AND DISCUSSION

Vacuum frying technology has been used commercially to produce fried food with a lower oil content and a better appearance than that obtained using traditional frying processes (Rosenthal et al., 2018). Pre-treatment through osmotic dehydration has been the subject of excellent studies outlining de-oiling procedures and mechanisms (Dehghannya and Abedpour, 2018). However, the bioactive compounds of vacuum-fried products also changes during osmotic dehydration.

Table 3. Analysis of variance for TPC and TFC

Source	Total phenolic content (mgGAE/g DW)			Total flavonoid content (µgQE/g DW)		
	mean square	*f*-ratio	*p*-value	mean square	*f*-ratio	*p*-value
A – maltodextrin concentration	0.2206	6.97	0.0460	23 111.4	8.53	0.0330
B – citric acid concentration	1.6415	51.84	0.0008	18 930.7	6.99	0.0458
C – soaking time	1.9781	62.46	0.0005	2 524.41	0.93	0.3786
A^2	2.6262	82.93	0.0003	21 122.5	7.80	0.0383
AB	0.6477	20.45	0.0063	2 307.36	0.85	0.3983
AC	0.1475	4.66	0.0834	1 346.16	0.50	0.5122
B^2	0.1225	3.87	0.1064	22 268.5	8.22	0.0351
BC	1.2255	38.70	0.0016	5 640.76	2.08	0.2085
C^2	0.8208	25.92	0.0038	12 445.1	4.60	0.0849
Lack-of-fit	0.0333	1.05	0.4460	444.965	0.16	0.9160
Pure error		0.0317			2 707.89	
R^2		97.47%			89.12%	

The Box-Behnken model was applied to describe the behavior of change in total phenolic and flavonoid content of vacuum-fried shallots. All factors, including maltodextrin, citric acid concentration and soaking time affected by the change of biological compounds in the product. The model for the evolution of TPC and TFC in products was established with a high correlation coefficient (R^2 > 89%). It proved that the fitted model could fully describe the change in TPC and TFC content under different conditions of osmotic treatment. From all the data obtained, the regression equations describing the relationship between TPC/TFC and independent variables for the Box-Behnken model were established (as mentioned above). The Analysis of Variance for TPC and TFC was analyzed (Table 3 and Figures 1–2).

In these cases, almost all effects have *p*-values less than 0.05, indicating that they are significantly at a 95.0% confidence level. The fitted models for TPC and TFC of vacuum-fried shallots are presented in Equations 2 and 3.

$$TPC = -2.04 + 0.44A + 19.11B + 0.21C - 0.008A^2 + 0.805 AB + 0.0013 AC - 67.01 B^2 - 0.738 BC - 0.002C^2 \quad (2)$$

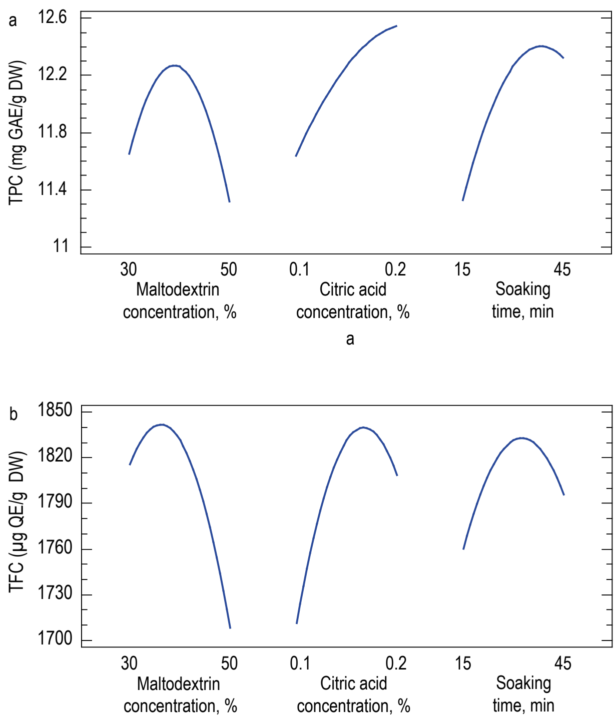


Fig. 1. Effect of main factors on the TPC (a) and TFC (b) of vacuum-fried shallots

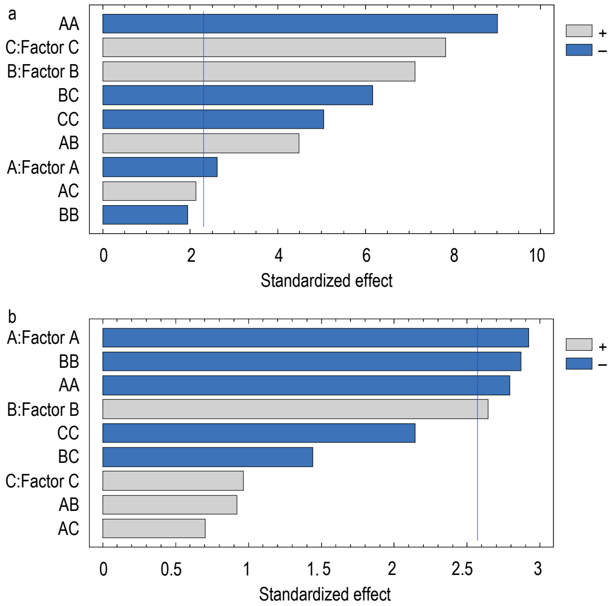


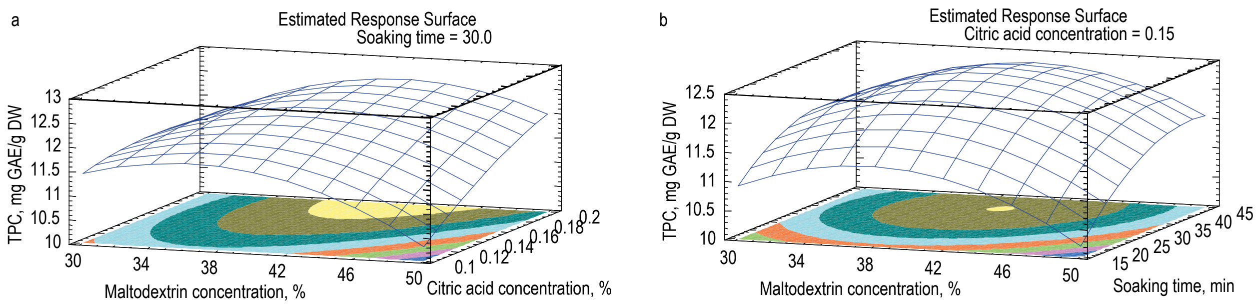
Fig. 2. Standardized pareto chart for TPC (a) and TFC (b)

$$TFC = 105.01 + 39.41A + 9126B + 18.04C - 0.696A^2 + 48.035AB + 0.122AC - 28574.7B^2 - 50.07BC - 0.24C^2 \quad (3)$$

where A, B, and C are maltodextrin concentration, citric acid concentration and soaking time, respectively, as independent values.

The high R^2 values of these models indicated a good fit for the data. This implied that TPC and TFC of vacuum-fried shallots could be estimated using the pre-treatment condition. The TPC and TFC affected by osmotic dehydration treatment are shown in Figures 3 and 4.

In this study, the osmotic agent is maltodextrin. Under hypertonic medium during osmotic treatments, plant tissue becomes soft and shrinks because water is removed from the symplasma, called "protoplasts" (Spiess and Behsnilian, 2006). It leads to excellent mass transfer and satisfactory sensory properties of the final product when employed for osmotic dehydration (Belkova et al., 2018). However, the great difference in pressure between the medium and material led to the loss of some water-soluble components such as anthocyanin in the shallots. Figures 1, 3, and 4 show the effects of osmotic dehydration at different times (15, 30, 45 min), maltodextrin concentrations (30%, 40%, 50% w/v), and citric acid concentrations (0.1, 0.15, 0.2%w/v) on TPC and TFC of shallots vacuum-fried at 120 ±1°C (680 mmHg) for 8 minutes. A change in TPC and TFC was found, and TPC and TFC increased at a concentration of maltodextrin from 30% to 40%. An explanation could be that the hypertonic medium made the cell wall soft, and some phenolic compounds inside the food matrix were extractable, leading to an



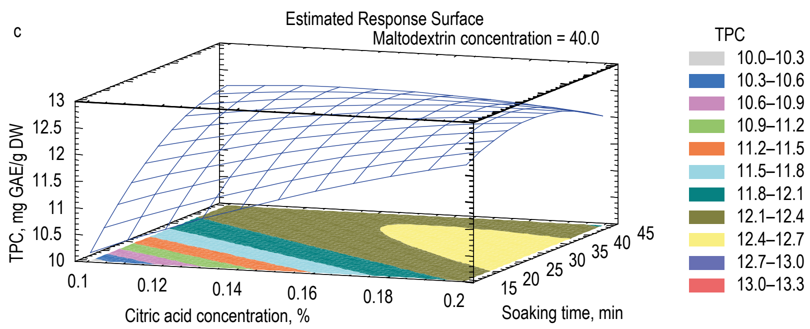


Fig. 3. Response surface of TPC under different pre-treatment conditions: interaction effect of maltodextrin and citric acid in 30-min soaking (a), interaction effect of maltodextrin and soaking time at 0.15% of citric acid (b), interaction effect of citric acid and soaking time at 40% of maltodextrin (c)

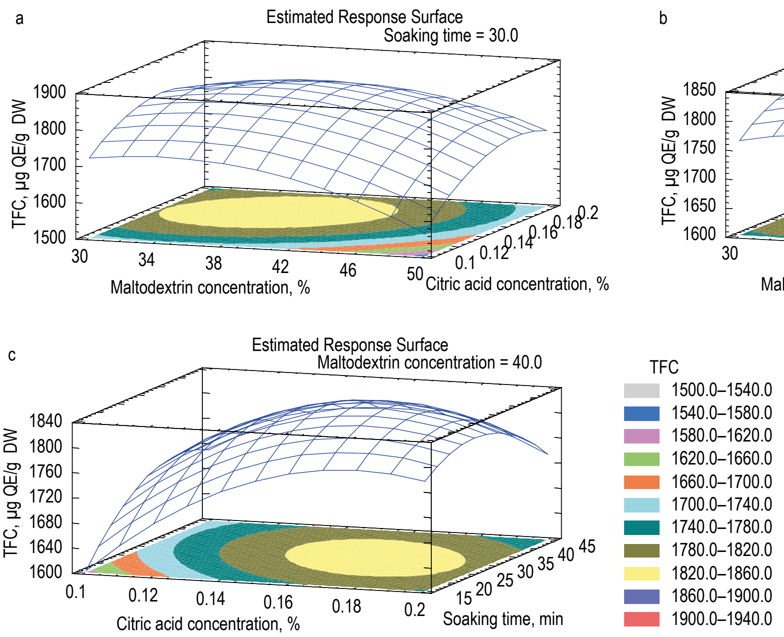


Fig. 4. Response surface of TFC under different pre-treatment conditions: interaction effect of maltodextrin and citric acid in 30-min soaking (a), interaction effect of maltodextrin and soaking time at 0.15% of citric acid (b), interaction effect of citric acid and soaking time at 40% of maltodextrin (c)

upward trend in the product. Maltodextrin also contributed as a coating agent; it covered the outer material and protected the nutritional components. However, when maltodextrin was over 40%, a decrease in TPC and TFC was found. As mentioned above, several phenolic and flavonoid compounds are water-soluble. When the fast mass transfer occurred at the first stage due to the significant difference in pressure between medium and material, antioxidant compounds also came to the soaking medium and the oxidation process might occur, leading to reduced TPC and TFC in the product. During osmotic dehydration, sugar uptake takes place, which is increased when more aggressive conditions are used (solution concentration and temperature, and osmotic dehydration time). Some biological compounds are soluble in water. Therefore, the high reductions in TPC and TFC reported could be due to the increase in sample water loss, as an increase in osmotic pressure occurs during osmotic dehydration, resulting in the bioactive compounds also being leached into the osmotic medium (Moreira, 2014; Xiao et al., 2017).

Dehydration time also affected the quality of the final products. The pressure difference affected the change in water content in the sample, as mentioned in the difference in maltodextrin conditions. The maintenance of TPC and TFC content found in the early stages of osmosis is because at this time the water loss mainly occurs at the surface of the material, the components in the tissue are less affected at this time. When materials were immersed for a long time, the tissue of the sample was soft and the compounds were leached out, which led to the oxidation of some water-soluble phenolic compounds, it resulted in a downward trend in total phenolic and flavonoids in the product (Xiao et al., 2017). Citric acid is the agent to preserve the antioxidant compounds of vacuum-fried shallots. Citric acid acts as a preservative in many processed foods, keeping them fresh. It does this by slowing or helping prevent the formation of browning compounds. It retains a food's color, flavor and texture (Robles-Sánchez et al., 2013). Vacuum-fried shallots maintained the TPC and TFC in the product under the protection of citric acid during pre-treatment processing. The decrease in TFC content when increasing the citric acid concentration to 0.2% could be due to the influence of the osmotic rate during osmosis. As can be seen from the response graph, a marked decrease in TFC at the citric acid concentration used (0.2%) was found in the sample treated in an osmotic medium containing 50% maltodextrin. In

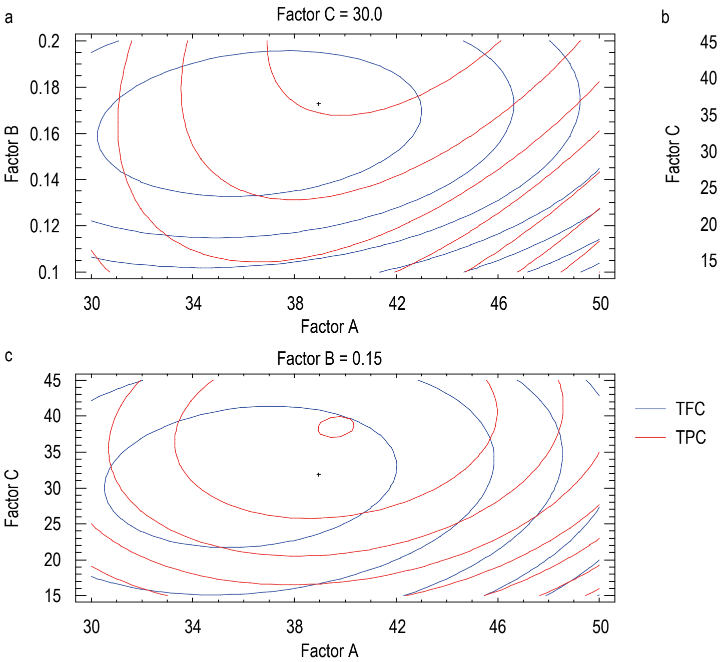


Fig. 5. Overlay plot of two responses (total phenolic and flavonoid content) of vacuum-fried shallots. Factor A: maltodextrin concentration (%), factor B: citric acid concentration (%), factor C: soaking time (min), TPC: Total phenolic content (mg GAE/g DW), TFC: Total flavonoid content (µg QE/g DW)

addition, the effective reducing acrylamide formation was also found in the fried shallots. Lowering the pH with citric acid before frying was effective in diminishing acrylamide formation (by approximately 73%) in French fries when fried for 6 min at 190°C in an atmospheric fryer (Jung et al., 2003).

Overlay plots in multiple regression statistics help to show and divide the level of desirability area based on the response surface plots that have been observed. Moreover, the advantage of using an overlay plot is that it is possible to find the optimal point and region containing the optimal point. The results showed that the highest concentrations of bioactive compounds were obtained when maltodextrin, citric acid concentration and soaking time were 38.9%, 0.17%, and 31.9 min, respectively (Fig. 5). In these processing conditions, the TPC and TFC in the sample were 12.44 mgGAE/g DW and 1841.7 µgQE/g DW, respectively.

In order to validate the adequacy of the model equations (2) and (3), a verification experiment was carried out under the optimal conditions mentioned above. The good correlation between experimental and actual results ($R^2 > 0.9$) undoubtedly confirmed that the model was suitable for reflecting the predicted optimization.

The final quality of the product with/without osmotic dehydration was also analyzed and is shown in Table 4. The vacuum-fried treated shallots have a high content of antioxidant compounds, and high antioxidant activity and is low in fat than an untreated sample. With a low moisture content, the sample could be stored for more than three months with no significant change in nutritional value (data not shown) and maintain its color (Fig. 6).

Table 4. Quality of vacuum-fried shallots processed under optimal pre-treatment conditions

Quality parameter	Optimal treated sample	Untreated sample
Lipid, %	12.12 ±0.13	16.23 ±0.14
Total phenolic content mgGAE/g DW	12.34 ±0.24	5.64 ±0.12
Total flavonoid content µgQE/g DW	1 823.45 ±1.24	654 ±4.35
Antioxidant activity % inhibition DPPH	70.45 ±1.24	45.34 ±0.34
Moisture content, %	3.43 ±0.04	3.47 ±0.09

Fig. 6. Vacuum-fried shallots under optimal osmotic dehydration conditions

CONCLUSIONS

The Box-Behnken model was successful in describing the relationship between pre-treatment conditions and the content of antioxidant compounds in vacuum-fried shallots. The high correlation coefficients were found that proved that the fitted model can be used to predict the phenolic and flavonoid content of fried shallots under different osmotic dehydration. Under the optimal treatment conditions, vacuum-fried shallots still retained a high content of antioxidant compounds, were high in antioxidant activity, and low in fat. With these results, the larger scale could apply to expand the product into the commercial market, which not only utilizes the material but also create incomes for farmers, especially in Vinh Chau town in Soc Trang province (Vietnam).

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