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FULL PAPER

Optimization of the cold drying process of mango (*Mangifera indica L.*) Tu Quy variety by response surface methodology

[1,2,*]Tran, T.Y.N., [2]Dang, T.T, [3]Do, T.N., [3]Pham, V.T., [3]Do, V.L., [4]Chung T.P.T., [1,5]Nguyen, N.Q., [6]Tran, B.L., [1]Le, D.T. and [1,2]Dao, T.P.

[1]*Institute of Environmental Sciences, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam*

[2]*Faculty of Environmental and Food Engineering, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam*

[3]*Faculty of Food Science and Technology, Ho Chi Minh City University of Food Industry, Vietnam*

[4]*Department of Literature and Technology, Huyen Hoi Secondary School, Cang Long District, Tra Vinh Province, Viet Nam*

[5]*Faculty of Pharmacy, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam*

[6]*College of Agriculture, Can Tho University, Can Tho City, Vietnam*

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Abstract

Mango (*Mangifera india L.*) is a common fruit that has been found in a wide range of applications. However, the diversification of mango products has been limited. In this study, the manufacture of dried mango was attempted to process by adopting the cold drying technique. The response surface methodology was used to evaluate the simultaneous effects of cold drying temperature - CT (°C), ultrasonic power - UP (W) and slice thickness-ST (cm) on total polyphenol (TP) and total ascorbic acid (TA) content by using Box Behnken experimental design. The established model was considered suitable to describe the experimental data. The optimized drying parameters were determined at the temperature of 45.02°C, ultrasonic power of 312.22 W, and slice thickness of 0.96 cm. These conditions effectively resulted in the highest retention of TP values of 338.994 mg GAE/100 g and TA of 36.783 mg AA/100 g.

1. Introduction

Fruit is well known to be a rich source of many health benefits, notably tropical fruit (Li *et al*., 2016; Nguyen *et al.*, 2020). Mango (*Mangifera india L.)* belongs to the family of Anacadiaceae, genus Mangifera, firstly originated in the India-Burma-Malaysia region more than 4000 - 6000 years ago (Yadav and Singh, 2017). It has been widely cultivated around the world since the early XIX century. One cup of mango is highly recommended in the daily diet due to its beneficial effect in promoting health and preventing cardiovascular diseases (Siddiq *et al.,* 2017). Mango includes many types of vitamins and minerals (B_1, B_2, B_3, A, E, K, magnesium, iron,). The production of mango was reported to reach 839 thousand tons in Vietnam, which was up to 43 million metric tons in the world. (Jegede, 2019). Many innovative processing technologies were applied to promote diversified products from mango raw material to extend their shelf life or solving inventory problems. Recently, dried fruit products have been favoured for not only novelty of taste but also their convenience. However, the use of the conventional drying method was found to reduce the quality of the final product including texture, colour, taste, and nutritional values due to the high processed temperature and extended drying time (Erenturk *et al*., 2005). Hence, the alternative approach to minimize the nutritional loss of the dried products has been increasingly gained attention from researchers.

A variety range of technologies has been explored to tackle the problem of quality loss from dried fruit including vacuum drying, freeze-drying, or microwave drying which promoted a better effect in the retention of nutritional values from fried fruit (Carranza-Concha *et al*., 2012; Tian *et al*., 2016; Dereje and Abera, 2020). The pre-treatment process assisted with the drying process was found to speed up the drying rate by softening the food structure but inducing undesirable

*Corresponding author.
Email: *ttynhi@ntt.edu.vn*

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quality of products (Huang *et al*., 2020). Among current techniques, non-thermal processes such as ultrasound-assisted drying have been currently considered an effective method in reducing drying time. The application of ultrasound pre-treatment on the drying process was observed to decrease about 40% of the drying time of dried carrots (Wiktor *et al*., 2019). Huang *et al*., (2020) implied that ultrasound application can decrease water activity, improve the product colour as well as reducing nutritional loss. Besides, the response surface methodology (RSM) applied in the process has been usually used to optimize the operational conditions. Therefore, the aim of this study was to assess the effects and interactions of three independent variables: cold drying temperature (CT), ultrasound power (UP) and slice thickness (ST) on total polyphenol content and total acid content in mango fruit. The result can possibly provide the optimized conditions for the process of dried mango and a basic understanding of correlation among influencing factors.

2. Materials and methods

2.1 Materials

Tu Quy mangos were purchased from Thanh Phu district, Ben Tre province, Vietnam. The fruit was selected with a fully characteristic aroma, uniform size, and non-waterlogged. Fruit weight was weighed at 0.7-0.8 kg with a total dissolved solids concentration of approximately 18-20ºBx. Harvested fruits were washed and peeled. Each fruit was sliced to the specimen of 150 g and was used for further experiments

Sugar was supplied from Bien Hoa Sugar Company, Dong Nai Province, Vietnam. Standard of Galic acid (99.99%), L-ascorbic acid (99.7%) were purchased from Sigma-Aldrich, Inc, St. Louis, Missouri, US. Methanol (99.5% purity), Na_2CO_3 (99.5% purity) were from Unionchem Co. Ltd, Quindao, China.

2.2 Box-Behnken design

The process of ultrasound-assisted cold drying to manufacture dried mango was optimized by using Design Expert software (Version 11, StatEase Inc., USA). The design was run with three independent variables including ultrasonic power, drying temperature, and slice thickness of mango specimen. Total polyphenol content and total ascorbic acid content were two dependent variables. The experimental design consisted of 18 runs (6 experiments at the center) with the central points of slice thickness 1cm, ultrasonic power W and drying temperature 45°C, shown in Table 1. The detailed experimental plan of 18 runs is presented in Table 2. After obtaining the predictive models to determine the TP and TA values, the validatory experiments to confirm the precision of these model was conducted at the optimized conditions from the models.

Table 1. Matrix for variables for ultrasound-assisted drying of mango fruit. Ultrasound power was applied to each 150 g of sample specimen.

Code	Independent factors	Low (-1)	Middle (0)	High (+1)
A	Drying temperature (°C)	40	45	50
B	Ultrasound power (W)	200	300	400
C	Slice Thick (cm)	0.5	1	1.5

Table 2. Experimental design for 3 factors. Ultrasound power was applied to each 150 g of sample specimen

Std	Run	A	B	C
1	8	40	200	1
2	4	50	200	1
3	2	40	400	1
4	3	50	400	1
5	14	40	300	0.5
6	15	50	300	0.5
7	18	40	300	1.5
8	12	50	300	1.5
9	13	45	200	0.5
10	9	45	400	0.5
11	1	45	200	1.5
12	10	45	400	1.5
13	7	45	300	1
14	11	45	300	1
15	17	45	300	1
16	6	45	300	1
17	16	45	300	1
18	5	45	300	1

2.3 Ultrasound-assisted cold drying process

Each slice of fruit was cut in the range of thickness from 0.5 to 1.5 cm. The as-prepared mango specimen was pre-treated by ultrasound process in 20 mins with applied ultrasonic power from 200 to 400 W per 150 g of sample (1.33-2.66 W/g sample). The drying temperature was varied from 40 to 50°C with a fixed air velocity of ~2 m/s. The drying process was complete when the moisture of dried mango was less than 16%. The total polyphenol content and total ascorbic acid content were determined to evaluate the effects of mango slice thickness, ultrasonic power, and drying temperature on dried mango products.

2.4 Quantitative analysis of total ascorbic acids

TA determination method was implemented and varied according to Spínola *et al.* (2014). Five grams of sample were crushed and 25 mL of metaphotphoric acid - acetic acid solution was added to a 50 mL volumetric flask. The mixture was shaken gently to homogenize the solution and was diluted to the mark with metaposphoric acid - acetic acid. The mixture was then centrifuged at 4000 rpm for 15 mins. An aliquot of 0.23 mL of 3%

bromine water was added to 4 mL of collected supernatant to oxidize the ascorbic acid to dehydroascorbic acid. Then, 0.13 mL of 10% thiourea was added to the mixture to reduce the excess bromine. 1 mL of 2,4-dinitrophenylhydrazine solution was then added to the mixture to form an osazone complex. The obtained solution was left at 37°C for 3 hrs in a water bath before refrigerating for 30 mins. Finally, 5mL of 85% H_2SO_4 was added to the mixture to obtain the coloured solution. The absorbance of the colored solution was recorded at 521 nm. The total vitamin C content in the sample was calculated based on the standard equation y = 0.0427x - 0.1466 (coefficient R^2 = 0.9859)

2.5 Quantitative analysis of total phenolic content

The TP value, determined by the Folin-Ciocalteu method, is based on the phenol reduction of a complex called phosphowolframate-phosphomolybdate to a blue complex as described by Tran *et al.* (2020). Briefly, 0.25 g of sample was crushed in a cold jar and was placed in a 100 mL dark bottle. A total of 25 mL of 50% methanol was added to the sample and was shaken for 3 hrs, followed by incubation for 72 hrs at room temperature. The solution was then filtered through Whatman No.4 filter paper, centrifuged at 13000 rpm for 10 mins. An aliquot (1 mL) of supernatant extract placed in a test tube, was mixed with 5 mL of 10% Folin-Ciocalteu reagent and 4 mL of Na_2CO_3 7.5% solution. The obtained solution was kept in a thermostat bath at 45°C for 15 mins. The absorbance was measured at 765 nm. The TP content, expressed as mg equivalent of gallic acid per 100 grams of dry matter (DW), was calculated from the standard curve of gallic acid following the equation y = 0.0074x + 0.0178 (correlation coefficient R^2 = 0.9981).

2.6 Analysis of data

Data analysis were statistically processed by Excel and Design Expert software 11 Box-Behnken model to design experiments with the support of ANOVA in Design Expert 11 software with 18 experiments (6 experiments at the centre) were used to analyze the experimental plan and analyze data.

3. Results and discussion

3.1 Interaction between factor on total polyphenol content

With the design of 18 experiments (Table 2) including the central points (Table 1), the significance was recognized with a p-value < 0.05. In Figure 1, data points corresponding to actual TP values and predicted TP values were fairly gathered on the 45-degree line and were equally on both sides of the line which meant that the predicted level was considerably high accurate. On the other hand, the ratio of signal to noise measured by adequate precision showed a value of 5.895 which was believed to be desirable if the value is greater than 4. The correlation coefficient (R^2) obtained from Design Expert 11 was 0.8311, which described the compatibility of experimental values and the values obtained from the model. According to Samart Sai-Ut *et al* (2015), the coefficient of determination (R^2> 0.75) showed that the established model was compatible with the experimental values (Sai-Ut *et al.*, 2015).

From the results of data presented in Table 3 and Table 4, The regression analysis data were fitted to a quadratic model as followed by the equation:

$$TP\ (mg/100\ g\ DW) = -5773.18292 + 244.58465A + 3.13589B + 269.69217C – 2.71726A^2 – 0.005132B^2 – 140.43983C^2$$

"Lack of Fit" (1.32 with p > 0.05) indicated that the proposed model experienced a good fit with the experimental data. There was a 36.46% chance that a Lack of Fit F-value could occur due to noise. More importantly, there was only a 2.47% chance that a P-value could occur due to noise. P-values less than 0.05 indicated that the predicted model was statistically significant. In this case, two interaction coefficients: A^2,

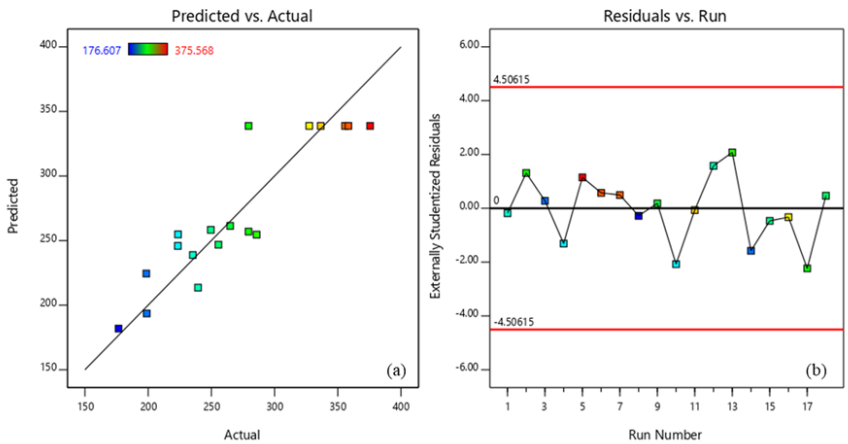

Figure 1. (a) Predicted and actual plot, (b) Studentized residuals versus Run number

Table 3. Fit Statistic predictions for a model of TP content in mangos affected by processing conditions

Std. Dev.	Mean	C.V. %	R^2	Adjusted R^2	Predicted R^2	Adeq. Precision
35.72	270.24	13.22	0.8311	0.6411	-0.332	5.8951

Table 4. Effects of independent variables on TP

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	50228.5	9	5580.94	4.37	0.0247	significant
A-Drying temp	0.1972	1	0.1972	0.0002	0.9904	
B-Ultrasound power	258.74	1	258.74	0.2028	0.6644	
C-Slice thick	250.32	1	250.32	0.1962	0.6695	
AB	4058.9	1	4058.9	3.18	0.1123	
AC	1126.37	1	1126.37	0.8829	0.3749	
BC	21.09	1	21.09	0.0165	0.9009	
A^2	20136.8	1	20136.8	15.78	0.0041	
B^2	11491.3	1	11491.3	9.01	0.017	
C^2	5379.09	1	5379.09	4.22	0.0741	
Residual	10206.7	8	1275.83			
Lack of Fit	4519.31	3	1506.44	1.32	0.3646	not significant
Pure Error	5687.35	5	1137.47			
Cor Total	60435.1	17				

$P < 0.01$ highly significant, $0.01 < P < 0.05$ significant, $P > 0.05$ not significant. Values obtained from DE11

B^2 were observed to be significant The occurring noise variables were likely to be dominated by the unrelated cross-interactions A, B, C variables.

Figure 2 shows the surface reaction of TP when changing the ST, heat treatment parameters during ultrasonic osmotic and CT in mango slices. The results showed that the lowest TP value at drying temperature of 40°C and ultrasonic power of 1.33 W/g was 176.607 mg/100 g DW and was found to increase when increasing the influencing factors. The repetition of the 6 central point experiments also revealed the interaction between the A and B variables in the model on the TP content, where the highest value was 375.568 mg/100 g DW (at 45°C, 2 W/g, 1cm of thickness). The observed trend was generally positive, in which an increase in any of the three examined factors resulted in an increase of TP value. However, the measured value decreased with increasing the cold drying temperature greater than 45°C and 2 W/g (ultrasonic power). At temperatures lower than 45°C, there was the possibility that individual compounds in the phenolic group were degraded to form H^+ free radical scavenging derivatives in reaction with GAE reagents that increased the TP content. When both CT and UP were increased simultaneously, the increased ultrasonic frequency created more vibrations and increased the internal temperature of the mango slices, causing a decrease in the TP-value of heat-sensitive and environmental-sensitive compound groups (Qureshi *et al*., 2020). Figure 2B with an inverse interaction at a significant level ($p > 0.05$) between CT and UP, the TP value increased when the size of the mango slice was in the range of 0.5-1.5 cm. The size of thin or thick slices has a direct effect on the rate of heat transfer from the surface to the centre and is related to the area exposed to the environment (Kohayakawa *et al.*, 2004). The adjustment of a slice size of mango by 1cm showed the interaction with UP, resulting in the highest TP value (Figure 2C). The ultrasound-assisted drying was found to achieve higher retention in carotenoid content (80.7%) compared to un-pretreated carrots (53%). The effect of ultrasound might induce higher shrinkage and smaller porosity in the structure of food material, hindering air penetration. Besides, the pre-treatment of ultrasound could partially contribute to the inactivation of peroxidases responsible for the decay of bioactive compounds (Wiktor *et al*., 2019).

3.2 Interaction between factor on total ascorbic acid content

The quadratic model of the TA content is determined by the multivariate regression method:

$$TA\ (mg/100\ g\ DW) = -0.221490A^2 - 0.000986B^2 - 27.54413C^2 - 1.6557AC + 21.54206A + 0.626361B + 127.67717C - 609.15564$$

Predictive models for the TA content of mango are shown in Table 5. A negative Predicted R^2 revealed a better predictor of response than the current model. The

Table 5. The prediction coefficients for the model of TA content in mango

Std. Dev.	Mean	C.V. %	R^2	Adjusted R^2	Predicted R^2	Adeq. Precision
3.55	26.57	13.35	0.9181	0.8261	-0.0132	8.1160

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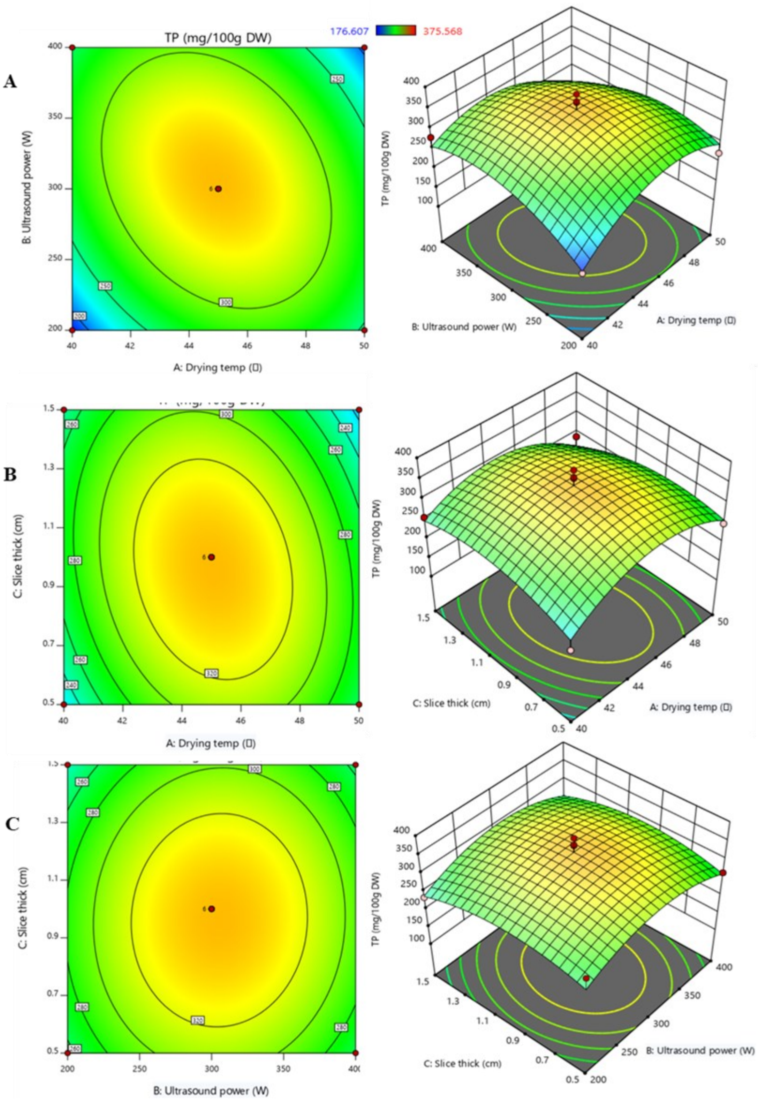

Figure 2. 3D and 2D response surface plots of interaction relationship of TP with (A) cold drying temperature and ultrasound power, (B) cold drying temperature and slice thick, (C) ultrasound power and slice thick

established model could be used to navigate the design space through Adeq Precision display which was 8.116 (> 4 is desirable). There is only about a 0.09 difference between the predicted and actual values (Figure 3). This implied the significance level in the model.

The model with an F-value of 9.97 implied the significance of the model (Table 6). There is only a 0.18% chance that an F-value could occur due to noise. P -values less than 0.05 indicated the model was significant. In this case, interaction coefficients of B, AC, A^2, B^2, C^2 were significant model terms. Values greater than 0.1000 indicated that the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve this model.

The multivariate interactions on the TA content after treatment are shown in Figure 4. The maximum TA value of 38.8897 mg/100 g DW at the centre point was processed at CT about 44-46°C, and the UP increases from 2 W/g to 2.33 W/g. TA value tended to decrease when increasing variables (Figure 4A). In a previous report, a continuous ultrasound process could greatly destroy tissues and structural compounds (including vitamins and phytochemicals). Besides, the increased cold-drying temperature could induce an increase in the rate and type of oxidation reactions, leading to a declination in TA value (Nowacka *et al*., 2018). This was also consistent with the claims for heat sensitivity of the antioxidant compounds (Nguyen *et al.*, 2019). Dereje and Abera (2020) reported that the vitamin C content of dried mangoes was in the range of 28.45 - 78.87 mg AA/100 g. It showed that the results of the fluctuation of Vitamin C content in the model were consistent. The interaction between CT-ST (Figure 4B) and UP-ST (Figure 4C) showed similar figures to conclude: the relevance of CT selection at 45.02°C, UP at 312.22 W/150 g, ST at 0.963 cm as the optimal parameter.

3.3 Model validation

The TP and TA values were experimental collected based on the optimized process parameters from the predictive models, shown in Table 7. The experimental result of TP and TA values were 341.798 mg GAE/g

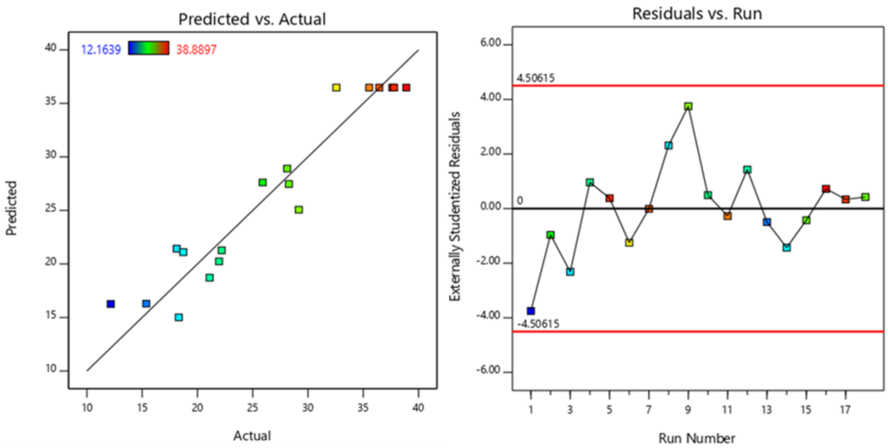

Figure 3. Estimation of Model Precision for TA in mango

Table 6. Effects of independent variables on TA

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1129.83	9	125.54	9.97	0.0018	significant
A-Drying temp	0.454	1	0.454	0.0361	0.8541	
B-Ultrasound power	95.03	1	95.03	7.55	0.0252	
C-Slice thick	7.31	1	7.31	0.5806	0.468	
AB	32.59	1	32.59	2.59	0.1463	
AC	68.52	1	68.52	5.44	0.0479	
BC	3.55	1	3.55	0.282	0.6098	
A^2	133.79	1	133.79	10.63	0.0115	
B^2	424.65	1	424.65	33.73	0.0004	
C^2	206.91	1	206.91	16.43	0.0037	
Residual	100.73	8	12.59			
Lack of Fit	75.67	3	25.22	5.03	0.057	not significant
Pure Error	25.05	5	5.01			
Cor Total	1230.56	17				

Table 7. Total polyphenol content and total ascorbic acid content at optimized conditions on Design expert 11. Ultrasound power was presented to be applied to 150 g of sample specimen.

		CT	UP	ST	TP (mg GAE/g DW)	TC (mg/100 g DW)
Predict		44.88	312.89	0.965	338.994	36.783
Experiment	Run 1	45	310	1	344.968	37.459
	Run 2	45	310	1	338.248	33.134
	Run 3	45	310	1	344.231	37.988
	Medium	45	310	1	341.798	36.193
				Error	0.604	0.867

DW and 36.193 mg AA/100 g DW, respectively which was compatible with the predicted value from the models of 338.994 mg GAE/g DW and 36.783 mg AA/100 g DW, respectively. This demonstrated that the conditions of the model were consistent with the experimental values. Applying the RSM and ANOVA analysis of Design expert 11 software, this study determined the optimal parameters of the ultrasound-assisted cold drying was found to obtain the highest retention of polyphenol and acid content at the temperature of 45°C, ultrasonic power of 2.07 W/g, and the mango slice thickness of 1 cm.

4. Conclusion

A food product was manufactured based on mango raw materials by ultrasound-assisted cold drying. Optimization of the drying process following optimal parameters: temperature of 45°C, ultrasonic power of 2.07 W/g, the material thickness of 1cm. These conditions corresponded with TP and TA of 338.994 mg GAE/100 g and 36.783 mg AA/100 g respectively (TA) in the resulted product. Validatory experiments confirmed the optimal conditions of the ultrasound-assisted cold drying process. The use of low temperature in drying was observed to effectively preserve the TP and TA content in the product. However, the study

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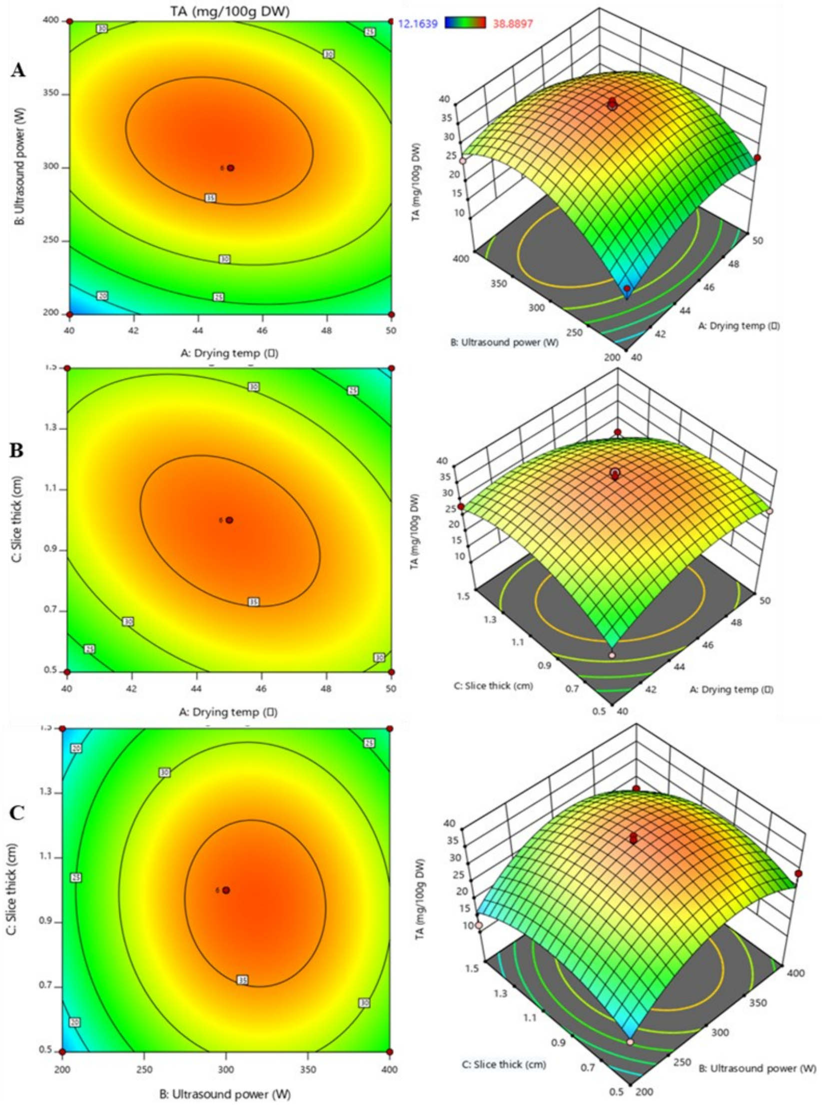

Figure 4. 2D and 3D response surface plots of interaction relationship of TA with (A) cold drying temperature and ultrasound power, (B) cold drying temperature and slice thick, (C) ultrasound power and slice thick

showed that non-significant interactions between the variables. Therefore, it is necessary to improve all the variables involved in each stage of product processing.

Conflict of interest

The authors declare no conflict of interest.

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