

Optimization of Heat Pump Drying Process for Schefflera heptaphylla Leaves by the Response Surface Methodology

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ABSTRACT

Background: Schefflera heptaphylla is a species of plant living in the forest, containing numerous bioactive compounds beneficial for human health. The efficient extraction of these bioactive compounds from the plant while minimizing losses is a currently highlighted issue.

Methods: In this research, the impact of three variables: drying time (min), drying air velocity (m/s) and drying air temperature (°C) on the reactions, including loss of total phenolic (TPCL), total flavonoid content loss (TFCL) and moisture content (MC) investigation of Schefflera heptaphylla leaves was conducted using the central complex design (CCD) within the framework of the methodology reaction surface method (RSM).

Result: The optimal conditions obtained from the response surface method were 48°C, 1.9 m/s and 600 min. The experimental values at the optimal conditions of TPCL, TFCL and MC are 17.22±0.20%, 14.83±0.15% and 8.47±0.12%, respectively. Crude fiber content, moisture content, total ash and total fat content of raw materials have been identified.

Key words: Heat pump drying, Response surface methodology, Schefflera heptaphylla, Total flavonoid content loss, Total phenolic content loss.

INTRODUCTION

Schefflera heptaphylla (L) Frolin is a plant species commonly found in forest and woodland habitats, particularly in mountainous regions. These plants are distinguished by their lofty, woody trees, leaving with digitately compound structures. The leaves are accompanied by sheathing petiolar bases, which are frequently arranged in compact rosette formations. Typically, these plants produce terminal (or pseudo-lateral) inflorescences in the form of panicles or compound umbels. These inflorescences bear smaller secondary umbels known as umbellules (Wang et al., 2021). A plant native to Southeast Asia, it has been traditionally employed as a medicinal remedy for alleviating fever, inflammation and rheumatism (Li et al., 2009; Maeda et al., 1994). Polyacetylenes, triterpene glycosides, caffeic acid and saponins derivatives are the secondary metabolites that are characteristic of the Araliaceae family (Mthembu et al., 2010). Other research studies have indicated that leaves have medicinal properties for the treatment of indigestion, while roots have been traditionally employed for the treatment of fevers and venereal disease, as well as in emetics to alleviate nausea. Additionally, cold infusions have been utilized to soothe skin irritation in newborn babies (Frodin et al., 2010; Wu et al., 2011). The plant's bark is extensively employed in folk medicine due to its diuretic properties and tonic effects (Wu et al., 2013). Sometimes, ashes are used to treat dropsy. Earlier investigations on S. Heptaphylla's phytochemical properties have indicated that saponins and triterpenoids are the primary bioactive components found in the plant (Li et al., 2007).

By adjusting the compressor capacity and carefully selecting the right air and refrigerant flows, the heat pump ¹School of Chemistry, Biology and Environment, Vinh University, Vinh City, Vietnam.

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Submitted: 17-08-2023 Accepted: 20-12-2023 Online: 31-01-2024 drying process can be controlled from -20°C to 70°C. This feature makes it an effective method for conducting lowtemperature drying operations (Patel and Kar, 2011). Consisting of a conventional drying chamber equipped with an air circulation system, the heat pump dehumidifier also incorporates standard components commonly found in aircooled systems. During the refrigeration cycle, the heat pump condenser heats the air and the evaporator acts as the cooler and dehumidifying the dry air. The condensing temperature of the refrigerant used determines the maximum achievable drying temperature (Colak, Hepbasli, 2009). After the 1970s, heat pump systems gained popularity due to their cost-effective operation, which led to their proposal for food

drying processes (Shi et al., 2008; Hepbasli et al., 2009). The response surface method (RSM) proves to be a powerful statistical technique for optimizing test conditions,

examining critical processes and minimizing the need for extensive testing (Bas and Boyacı, 2007). By using quantitative data obtained through a suitable experimental design, it is possible to identify and solve multivariate problems simultaneously (Khuri and Mukhopadhyay, 2010). Verifying the predicted values experimentally is one of the most important aspects of implementing this method. Therefore, RSM has been frequently used in the optimization of food processes (Silva et al., 2007; Rajha et al., 2014; Chi et al., 2022). we aimed to optimize the drying conditions of Schefflera heptaphylla leaves to minimize the loss of total flavonoid content, total phenolic content and moisture content.

MATERIALS AND METHODS

Material and equipment

Material

Leaves of Schefflera heptaphylla were collected in Anh Son district, Nghe An province, Vietnam in July 2022 and identified by the Institute of Ecology and Biological Resources, Vietnam Institute of Science and Technology. Leaves collected are processed within 24 hours. The material is cut to a size of 2 mm.

Equipment experimental and measurements

The drying process of *Schefflera heptaphylla* leaves was carried out using a heat pump dryer model CYF-EL040 of Chin Ying Fa, Taiwan (Fig 1). The system used in this dryer consists of a heat pump system consisting of a compressor, two condensers (one located inside the drying chamber and the other outside the chamber, switched by a 3-way solenoid valve), one expansion valve, an evaporator, a heat recovery unit and an auxiliary heater in the form of resistance. The recycled refrigerant in the heat pump drying system is R22.

The temperatures and velocities of drying air were controlled to satisfy the setpoint of designed technology conditions. All process parameters were sampled and recorded. The drying air temperatures were controlled through the automatic coordination of the compressor, auxiliary heater and the 3-way solenoid valve. The velocities of drying air are regulated by an inverter to drive the axial fan at different values of rotational speed.

Methods

Proximate analysis

Proximate analysis of *Schefflera heptaphylla* leaves in the whole experiment involved assessment of crude fiber, moisture content, total ash and total fat content.

Moisture content (MC)

For MC determination, a measured amount of sample was placed in a temperature-controlled oven at 105°C for 5-6 hours until a stable weight was reached (Khan *et al.*, 2013). Using an electronic balance, the dry weight of each sample

is then measured. The MC percentage is determined using the following equation (1):

MC (%) =
$$\frac{W_o - W_f}{W_o} \times 100$$
 ...(1)

W.: Final weight of a sample.

W_o: Original weight of sample.

Total fat estimation

Crude fat was estimated by cleaning dry ornaments with diethyl ether bleach. The detergent was removed using a rotary evaporator to dissolve it (Khan *et al.*, 2013). The crude fat content, expressed as a percentage, is calculated using the equation (2):

Crude fat (%) =
$$\frac{W_e}{W_d} \times 100$$
 ...(2)

W_e: Weight of ether extract.

W_d: Weight of dried sample.

Determination of crude fiber

A 10 g moisture-free sample was heated at 80° C for about 30 min in the presence of 200 mL of $\rm H_2SO_4$ 0.25N. To ensure a constant volume of boiling medium, hot water is regularly added. After filtering the mixture, the residue is washed with hot water to remove residual acid. The residue was then treated with 200 mL of NaOH 0.32N and washed again with hot water, ether and alcohol to obtain an alkaline extract.

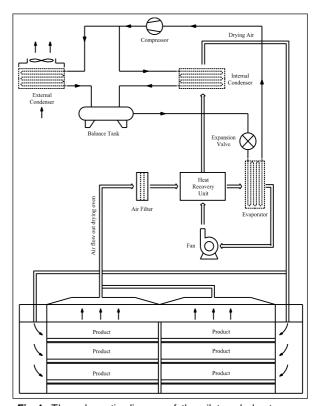


Fig 1: The schematic diagram of the pilot-scale heat pump conveyor dryer used in this study.

The alkaline extract was then transferred to a crucible and dried overnight at about 90-100°C and weigh (Wi) with an electronic balance. The crucible is put into a muffle furnace and heated at 600°C for 5.5-6 hours. After cooling, the crucible was weighed again (Wa) (Khan N.A *et al.*, 2013). The difference in mass (W_i - W_a) corresponding to the crude fiber content (3)

Crude fiber (%) =
$$\frac{W_i - W_a}{W_o} \times 100$$
 ...(3)

W: Dry weight after digestion.

Wa: Weight of ash.

W.: Weight of moisture and fat free sample.

Determination of total ash

To determine the ash content, the dried samples underwent combustion within a muffle furnace set at 600°C for a period lasting 5 to 6 hours (Khan *et al.*, 2013). The following equation was used to calculate the ash content (4).

Ash (%) =
$$\frac{W_a}{W_d} \times 100$$
 ...(4)

W_a: Weight of ash.

W_d: Weight of dried sample.

Total phenolic content loss (TPCL)

The specified extraction procedure was employed for the extraction of phenolics. Samples and the extractant (ethanol 50%) were placed in a beaker, mixed in 5 h at 40°C then filtrated and stored at -20°C for further experiments. Total phenolic content loss (TPCL) was calculated by (5)

$$TPCL = \frac{TPC_{raw} - TPC_{p}}{TPC_{raw}} \times 100$$
 ...5

The modified Folin-Ciocalteu procedure, as reported by Singleton et al. (1999), was used to determine the total phenolic content (TPC) of the samples. The solution of Folin-Ciocalteu was mixed with 1 mL of the sample in a ratio of 5:1. After a period of 3 min, 4 mL of Na₂CO₃ 7.5% (w/v) solution was added to the mixture and the volume was adjusted to 10 mL with deionized water. The mixture was then placed in the dark at room temperature for one hour before measuring its absorbance at 765 nm using an Agilent 8453 UV Spectrophotometer. TPC of S. heptaphylla leaf extract was determined in milligrams of gallic acid equivalent (GAE) using a standard curve prepared with different concentrations of gallic acid. TPC values are reported as milligrams of GAE per gram of dry weight (DW). Three measurements were taken and the average value was recorded, TPCL values, according to raw S, heptaphylla leave, were then calculated using equation (5):

Total flavonoid content loss (TFCL)

The extraction method was employed for flavonoid extraction. Samples and the extractant (ethanol 50%) were placed in a beaker, mixed in 5 h at 40°C then filtrated and

kept in storage at -20°C for further experiments. Total flavonoid content loss (TFCL) was calculated by (6):

$$TFCL = \frac{TFC_{raw} - TFC_{p}}{TFC_{raw}} \times 100 \qquad ...(6)$$

The TFCs of the samples were analyzed using the $AICI_3$ method, modified by Marinova *et al.* (2005). Prepare 10 ml volumetric flasks, each containing 4 ml of H_2O , to which add 1 ml of catechin extract or standard solution (0.01÷ 0.07 mg/mL). Then, 0.3 mL of $NaNO_2$ 5% was added to each flask. After an interval of 5 minutes, add 0.3 mL of $AICI_3$ 10%. Then, 2 mL of NaOH 1M was added after 6 min and the final volume of each flask was adjusted to 10 mL with H_2O . The solution was mixed thoroughly and its absorbance was then measured at 510 nm using an Agilent 8453 spectrophotometer.

Measurement was made against a previously prepared reagent blank. TFC of leaves of *S. Heptaphylla* was quantified as mg Catechin equivalent (mgCE) per gram dry weight (DW). Three replicates were measured and the mean was recorded. Then, TFCL values were calculated based on equation (6), using raw *S. heptaphylla* leaves.

Experimental design

Using the central composite design (CCD) of the response surface method (RSM), the study examined the main effects of process variables on total phenolic loss (TPCL), total loss of flavonoid content (TFCL) and moisture (MC) during production. drying process of S. Heptaphylla leaves. As independent variables, the study chose drying temperature (X_1) , drying air velocity (X_2) and drying time (X_2) .

The study used a central composite design (CCD) consisting of 20 experiments, formed by 6 pivot points and 6 central points, combined with a full 8 factorial design.

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_1^2 + \sum_{i=1}^{3} \sum_{i < j}^{3} \beta_{ii} X_j X_i$$
...(7)

The values of the parameters $(\beta_0, \beta_i, \beta_{ii}$ and $\beta_{ij})$ of the left-hand side in (7) were evaluated by the Design Expert Ver program. 11.0. The evaluation is made based on the agreement of the experimental results with the full quadratic polynomial model with three independent variables as in (7).

RESULTS AND DISCUSSION

Raw material

The nutrient composition (ash, moisture, crude fiber, carbohydrate and fat) can provide nutrients needed for the human body. The potential of *S. heptaphylla* for treating various diseases and promoting health has been widely recognized. Furthermore, the traditional use of this herb in relieving pain has been scientifically validated, confirming the diverse applications of *S. heptaphylla*. This paper provides a comprehensive discussion of the traditional uses, chemical components, botany, nutrients and pharmacology of *S. heptaphylla*.

To ascertain the difference in nutrition and medicinal quality of *S. heptaphylla* collected from Nghe An in Vietnam, the conventional nutritional methods were applied to determine moisture, crude fat, crude fiber and crude ash determined as in Table 1.

Fitting the response surface models

The CCD of RSM was employed to optimize the responses, which included TPCL, TFCL and MC, of dried *S. heptaphylla* leaves using the heat pump drying process. The CCD with three independent variables was used as drying temperature, drying air velocity and drying time. Preliminary experiments were conducted to determine the selected variables (Table 2) were evaluated within a specific input range.

In the current research, desirability functions were developed for minimum TPCL, TFCL and MC criteria. Furthermore, it is feasible to amplify the weighting by assigning a numeric range (from 1 to 5) to indicate the

significance of optimizing each response variable. However, for this study, the same importance was assigned to all responses (third value). Table 3 shows the experimental design and the corresponding three response variables.

Table 3, presents the drying conditions and the values of the three ratings provided. The minimum values of TPCL, TFCL and MC are 15.88%, 12.76% and 7.38%, respectively. From multiple linear regression analysis of 20 data entries, experiments with quadratic polynomial models of TPCL, TFCL and MC during heat pump drying from *S. heptaphylla* leaves were drawn (Table 4).

Table 1: The data determine raw materials quality.

Conventional nutrients	Vol.
Moisture	70÷75.2%
Crude fat	0.2÷0.3%
Crude fiber	14.5÷16.7%
Crude ash	2.8÷5.3%

Table 2: The CCD design employed coded levels for the independent variables.

Independent	Units	Coded		Co	oded variable lev	rels	
variables	Office	symbols	-α	-1	0	1	+α
Drying temperature	°C	X ₁	36.6	40	45	50	53.4
Drying air velocity	m/s	X_2	1.16	1.5	2.0	2.5	2.84
Drying time	Min	X_3	560	600	660	720	760

Table 3: The data obtained from the experiments for the three responses based on the CCD matrix.

Run	Coded an	d processed variab	le level	Response		
	X ₁	X ₂	X ₃ Drying time (min)	Y ₁	Y ₂	Y ₃
	Drying temperature (°C)	Drying air velocity (m/s)		TPCL (%)	TFCL (%)	MC (%)
1	45	2	660	18.69	16.12	8.34
2	50	1.5	600	15.88	16.31	8.23
3	40	2.5	720	23.15	19.7	9.73
4	53.4	2	660	30.96	30.46	7.38
5	45	2	760	20.9	21.82	8.07
6	45	2	660	19.77	16.88	8.18
7	40	2.5	600	27.79	18.63	11.68
8	50	2.5	600	23.3	18.59	7.98
9	45	2.84	660	23.44	18.67	8.02
10	45	2	660	20.15	16.29	8.21
11	45	2	660	18.22	15.82	8.47
12	45	2	660	19.37	16.62	8.82
13	50	2.5	720	29.67	27.76	7.88
14	40	1.5	720	27.77	25.56	10.68
15	45	1.16	660	19.68	20.53	10.7
16	45	2	660	17.81	17.31	8.73
17	36.6	2	660	37.06	29.7	11.86
18	45	2	560	18.42	12.76	9.79
19	40	1.5	600	30.54	20.7	12.3
20	50	1.5	720	25.72	27.68	8.97

The coefficient of determination (R^2) for the models were 0.9897 (Y_1), 0.9880 (Y_2) and 0.9654 (Y_3), this indicates that over 98.97%, 98.80% and 96.54% of the response variability was accounted for. This confirms that the established model is accurate and effective within the specified range limits. The Lack of Fit F-values for Y_1 , Y_2 and Y_3 were determined to be 0.4855, 3.10 and 3.04, respectively. These values indicate that the Missing Conformity is negligible compared to the pure error. This shows that the accuracy of the polynomial model is sufficient.

Response surface analysis of TPCL, TFCL and MC

Contour plots and response surfaces were generated for each fitted model, illustrating the combined effects of the two factors on the response variable. These graphs are created by varying two independent variables while keeping the other at its central value. The resulting visualizations can be seen in Fig 2 to 4.

Through the evaluation of these plots, the effects of the variables on the response variables were discussed. The contour plots and response surfaces provided a comprehensive understanding of how changes in the

independent variables influenced the corresponding response. This analysis facilitated the interpretation and discussion of the variables' impacts on the responses, enabling a deeper insight into the relationship between the factors and the observed outcomes.

Response surface analysis of total phenolic content loss (TPCL)

The impact of each pair of independent variables on the yield of TPCL was visualized in three plots in Fig 2, which represents the response surface analysis of TPCL.

The response surface plot of TPCL is shown in Fig 2 and Table 5, all three factors (drying time, drying temperature and air velocity) show a negative quadratic effect (p<0.0001). The quadratic effect of drying temperature on TPCL is evident, with the lowest TPCL observed at drying temperatures around 45-50°C. The value of TPCL decreases as the drying air speed or drying time decreases as shown in Fig 2c. TPCL is lowest when the drying air velocity is from 1.5 to 2 m/s and the drying time is from 600-630 minutes. For the stability of phenolic compounds, enzymatic reactions or free water content can affect the phenolics (Nicoli *et al.*,

Table 4: Second-order polynomial models for TPCL, TFCL and MC.

Response	Model equations
Y ₁ -TPCL	$Y_1 = 19.00 - 1.83X_1 + 0.76X_2 + 0.95X_3 + 2.34X_1X_2 + 2.95X_1X_3 - 0.67X_2X_3 + 5.31X_1^2 + 0.91X_2^2 + 0.24X_3^2$
Y ₂ -TFCL	$Y_2 = 16.53 + 0.51X_1 - 0.64X_2 + 3.05X_3 + 1.29X_1X_2 + 1.83X_1X_3 - 0.75X_2X_3 + 4.63X_1^2 + 0.92X_2^2 + 0.11X_3^2$
Y ₃ -MC	$Y_3 = 8.45 - 1.38X_1 - 0.54X_2 - 0.43X_3 + 0.03X_1X_2 + 0.53X_1X_3 - 0.15X_2X_3 + 0.48X_1^2 + 0.39X_2^2 + 0.24X_3^2$

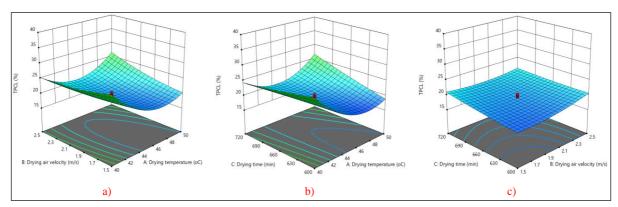


Fig 2: The response surface plots of TPCL.

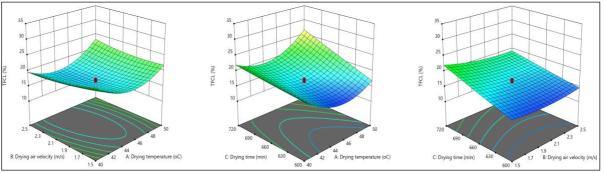


Fig 3: The response surface plots of TFCL

1999). Free water in *S. Heptaphylla* leaves should be removed and this study was done by heat treatment. The value of TPCL is minimized (15.88%) at the lowest air velocity (1.5 m/s), the shortest drying time (600 mins) and the highest temperature (50°C).

Response surface analysis of total flavonoid content loss (TFCL)

The response surface analysis of TFCL is depicted in Fig 3, using the same demonstration-style as that of the TPCL As shown in Fig 3 and Table 5, all three factors (drying temperature, air velocity and drying time) had a quadratic effect on TFCL (p < 0.0001). Drying air temperature greatly affects TFCL, TFLC is lowest when the drying temperature is about 44 to 48°C. The TFCL value decreased slightly when the drying air velocity increased, the TFLC was lowest when the drying air velocity was from 2 to 2.5 m/s. Drying time greatly affects TFCL, TFCL value decreases gradually and is lowest when drying time is 600-630 minutes.

Response surface analysis of moisture content (MC)

Fig 4 shows the response surface analysis of MC, where three graphs show the effect of each pair of independent variables on MC. All three factors (drying time, air velocity and drying temperature) had a quadratic effect on MC (p<0.0001). Theoretically, the drying temperature is the main process variable for MC. The effect of air velocity on drying results can vary depending on the characteristics of the product. Normally, using high temperature drying air, prolonged drying time and high air velocity can reduce the final MC. However, when drying biological products with extremely high heat transfer rates, barrel hardening can occur (Corzo O. et al., 2008). In this study, the lowest MC value determined by the proposed model was 7.38% at medium air velocity (2.0 m/s), drying time (600 min) and high temperature. (53.4°C) in the operating range of the system.

Optimization and model verification

The optimal conditions for drying *S. heptaphylla* leave by heat pump were determined to achieve the minimum TPCL, TFCL and MC criteria. Table 6 shows the results for the common case with equality of the weighting coefficients in the final objective function.

Using the desired function approach, the simultaneous optimization results indicate that the optimal drying conditions to obtain the lowest TPCL, TFCL and MC were determined to be 48°C, 1.9 m/s over a period of 600 minutes

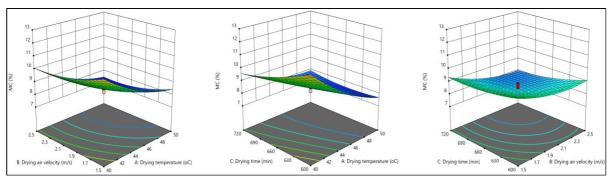


Fig 4: The response surface plots of MC.

Table 5: The analysis of variance (ANOVA) was performed to evaluate the model.

Source	Y ₁ - PC		Y ₂ - FC		Y ₃ - MC		
Course	F-value	p-value	F-value	p-value	F-value	p-value	
Model	106.36	< 0.0001 ^s	91.62	< 0.0001 ^s	30.99	< 0.0001 ^s	
X ₁ (Temperature)	73.47	< 0.0001 ^s	6.02	0.0340 ^s	180.22	< 0.0001 ^s	
X ₂ (Air velocity)	12.59	0.0053 ^s	9.23	0.0125 ^s	27.86	0.0004 ^s	
X ₃ (Time)	19.87	0.0012 ^s	212.16	< 0.0001 ^s	17.17	0.0020 ^s	
X_1X_2	70.82	< 0.0001 ^s	22.05	0.0008 ^s	0.0457	$0.8350^{\rm NS}$	
X_1X_3	112.51	< 0.0001 ^s	44.44	< 0.0001 ^s	15.32	0.0029 ^s	
X_2X_3	5.75	0.0374 ^s	7.47	0.0211 ^s	1.18	0.3022^{NS}	
X ₁ ²	656.29	< 0.0001 ^s	514.15	< 0.0001 ^s	22.93	0.0007 ^s	
X ₂ ²	19.31	0.0013 ^s	20.44	0.0011 ^s	14.98	0.0031 ^s	
X ₃ ²	1.33	0.2749 ^{NS}	0.2699	0.6147 ^{NS}	5.54	0.0404 ^s	
Lack of Fit	0.4855	0.7767 ^{NS}	3.10	0.1202 ^{NS}	3.04	0.1239^{NS}	
R ²	0.9897		0.9880		0.9654		
C.V%	3.36		3	3.8		4.13	

S: Significant; NS: Nonsignificant.

Table 6: Comparison between the predicted and experimental values for the drying heat pump process of S. heptaphylla leaves.

In	dependent varia	bles	Dependent variables	Optimu	ım value	% Difference	
X ₁ (°C)	X ₂ (m/s)	X ₃ (min)	(Response)	Experimental	Predicted	(CV)	
			Y ₁	17.22±0.20	16.67	3.3	
48	1.9	600	Υ ₂	14.83±0.15	14.41	2.9	
			Y ₃	8.47±0.12	8.23	2.9	

(10 hours). The test results provide TPCL, TFCL and MC measurements during heat pump drying. of leaves of *S. heptaphylla* were 17.22±0.20%, 14.83±0.15% and 8.47±0.12%, respectively. The experimental values are in close agreement with the predicted values (TPCL= 16.67%; TFCL= 14.41%; MC= 8.23%) obtained from the corresponding regression models, with the system The number of variation (CV) ranges from 2.9% to 3.3%.

CONCLUSION

By using the RSM's CCD, together with the usual graphical and desirable functional methods, the study successfully determined the optimal region in the experimental region. The answers were predicted using quadratic polynomial models. To achieve the lowest loss in total phenolic content (TPCL), total flavonoid content (TFCL) and moisture content (MC) during heat pump drying of leaves of *S. Heptaphylla*, the optimal condition is the temperature of 48°C, the drying time is about 600 minutes and the drying air velocity is 1.9 m/s. Under these conditions, TPCL, TFCL and MC were found to be 17.22±0.20%, 14.83±0.15% and 8.47±0.12%, respectively.

Conflict of interest

The authors certify that they have NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or nonfinancial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the subject matter or materials discussed in this manuscript.

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