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Effect of hybrid drying technique on non-traditional Chicory (*Cichorium intybus* L.) herb: Phytochemical, antioxidant characteristics, and optimization of process conditions

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This research investigated the influence of microwave-assisted fluidized bed drying (MAFBD) on the antioxidant and phytochemical characteristics of Chicory. Microwave power, temperature, and air velocity were used as process variables varied between 180–540 W, 50–70 °C, and 15–20 m/s, respectively. The responses determined for deciding the optimal criteria were total phenolics content, ascorbic acid, DPPH radical scavenging activity, total chlorophyll, carotene content, total flavonoid content, tannin content, and saponin content of the dried chicory. Statistical analyses were done by using the response surface methodology, which showed that independent variables affected the responses to a varied extent. The design expert predicted 462.30 W microwave power, 70°C temperature, and 15 m/s air velocity as optimum conditions to obtain highest desirability for the dried chicory. Separate validation experiments were conducted, under optimum conditions, to verify the predictions and adequacy of the second-order polynomial models. Under these optimal conditions, the predicted amount of ascorbic acid content was 38.32 mg/100g DW, total phenolic content 216.42 mg/100g DW, total flavonoid content mg/100g DW, DPPH scavenging activity 36.10 μg/ml, total chlorophyll content 311.79 mg/100g, carotene content 7.30 mg/100g, tannin content 2.72 mg/100g, and saponin content 0.46 mg/100g. The investigated parameters had a significant effect on the quality of the dried chicory. Taking the aforesaid results into consideration, our study recommended MAFBD as a promising technique with minimum changes in antioxidant and phytochemical content of chicory.

KEYWORDS

total phenolics, antioxidant activity, chicory, phytochemical properties, optimization

Highlights

Chicory is a potential source of polyphenols, flavonoids, antioxidants, vitamins, β-carotene, and minerals. Among vegetables, chicory leaves have gained attention for their therapeutic, biological as well as nutraceutical properties like anti-inflammatory, anti-nematodal, anti-hyperuricemia, anti-diabetic, anti-proliferative, etc., However, it has limited shelf life as degradation takes place within a few days of postharvest, etc. So, in order to make it available during off-seasons, the drying process is adopted for high-quality and long-lasting food products. This study suggested MAFBD as a potential technique with better retention of bioactive components. The results of this work will provide valuable data for manufacturers of dryers at industrial scale. However, at pilot scale, the process still needs further research.

Introduction

Chicory (*Cichorium intybus* L.) is an under-utilized, non-traditional perennial herb belonging to the genus *Cichorium* of *Asteraceae* family and is cultivated across the globe (Perovic et al., 2021). It is native to temperate regions of the world and is also cultivated in regions such as Europe, North America, and Asia. Chicory leaves are eaten as a leafy vegetable (Zeb et al., 2019). For decades, it has been used as a traditional medicine as well as in food applications to enhance the suitability and sustainability of different food products. Chicory is nutritionally rich and is a potential source of polyphenols, flavonoids, antioxidants, vitamins, β carotene, and minerals (Petropoulos et al., 2017). Among vegetables, chicory leaves have gained attention for their therapeutic, biological as well as nutraceutical properties like anti-inflammatory, anti-nematodal, anti-hyperuricemia, anti-diabetic, anti-proliferative, etc. (Janda et al., 2021). It can be consumed as fresh (salad), dried, or cooked like spinach and is blanched prior to consumption in order to remove bitterness. Moreover, it has a limited shelf life as the degradation takes place within a few days of postharvest. So, in order to make it available during off-seasons, the drying process is adopted for high-quality and long-lasting food products (Sehrawat et al., 2018). Drying is a food preservative technique that reduces the moisture content and the bulk of a food material, which is an important criterion for shelf life and during transportation. Various drying techniques like fluidized bed drying, conventional drying, solar drying, microwave drying, etc., are used for different sectors of the food industry. Among all, the conventional hot air drying method produces serious effects on quality attributes as well as physicochemical properties of food products due to its less efficiency and more drying time (Lv et al., 2016). Thus, microwave drying in association with fluidized bed drying is suggested to reduce the above-mentioned limitation and can significantly increase the quality and shelf life of a product. The combination of both these techniques offers several benefits like uniform moisture, heat and mass transfer, low processing temperature, decreases drying time, easy to control microwave power, saving energy, and high efficiency (Zahoor and Khan, 2021). However, no research has been done on the microwave drying of Chicory as well as on the optimization of MAFBD of Chicory using response surface methodology.

Therefore, the current study was planned to study the influence of different parameters of MAFBD like air temperature (AT), microwave power (MWP), and air velocity (AV) on antioxidant and phytochemical characteristics of chicory and to optimize the drying conditions for process variables of acceptable chicory leaves.

Materials and methods

Chicory was purchased from the local market of Srinagar, Jammu and Kashmir. The leaves were separated from the stems and washed in order to eliminate the dust and dirt. These leaves were then stored at 4 °C prior to the drying experiment. The initial moisture content of fresh chicory leaves was measured by the hot air oven method at 105 °C for 24 h and a value of 94.5 % (w.b) was recorded (Ranganna, 1986).

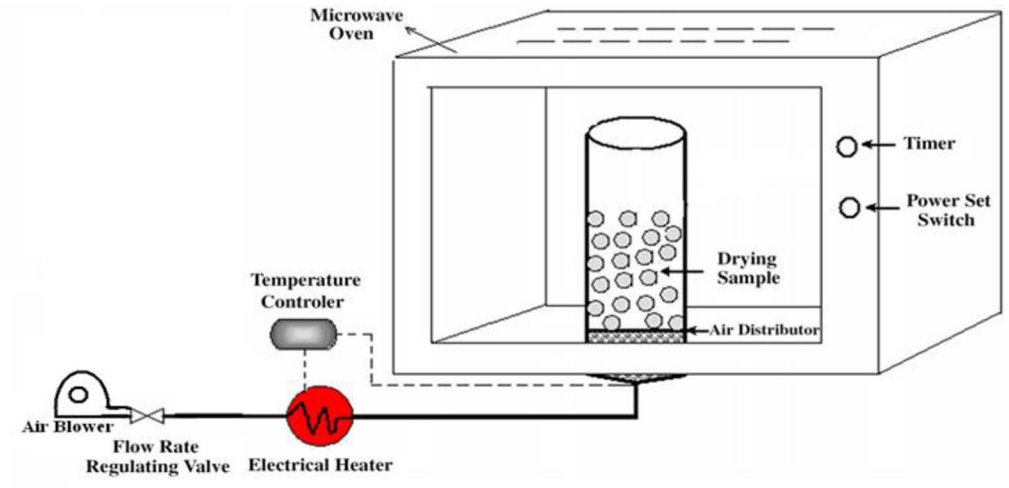
Chemicals

Ascorbic acid, produced by J.T. Baker (Holland), was used as a standard. Standard substance and samples were dissolved/extracted in the solution of 3% m-phosphoric acid (Riedel-de Haen, Germany). 1,1-Diphenyl-2-picryl-hydrazyl-hydrate (DPPH_) and Folin–Ciocalteu reagent were purchased from Sigma (Sigma-Aldrich GmbH, Steinheim, Germany). Gallic acid and quercetin were purchased from Sigma (Sigma, St. Luis, MO, USA). All other chemicals and reagents were of analytical reagent grade.

Microwave-assisted fluidized bed drying

Microwave-assisted fluidized bed drying prototype was designed and installed at the Department of Post-Harvest Engineering and Technology, AMU, Aligarh, India. The image and schematic diagram of the setup are shown in Figure 1A. Experimental studies were conducted by using a digital domestic microwave oven (LG Convection MWO, MC9286BQ, Gu, Seoul, Korea) with an input and output power of 1,950 W and 900 W, respectively and a magnetron that operates at 2,450 MHz frequency and converts electric energy into microwave power. The dimensions of the oven (L × W × D) were 376 × 574 × 505 mm, with a centrally mounted rotating glass and fan.

A



(I)

(II)

FIGURE 1
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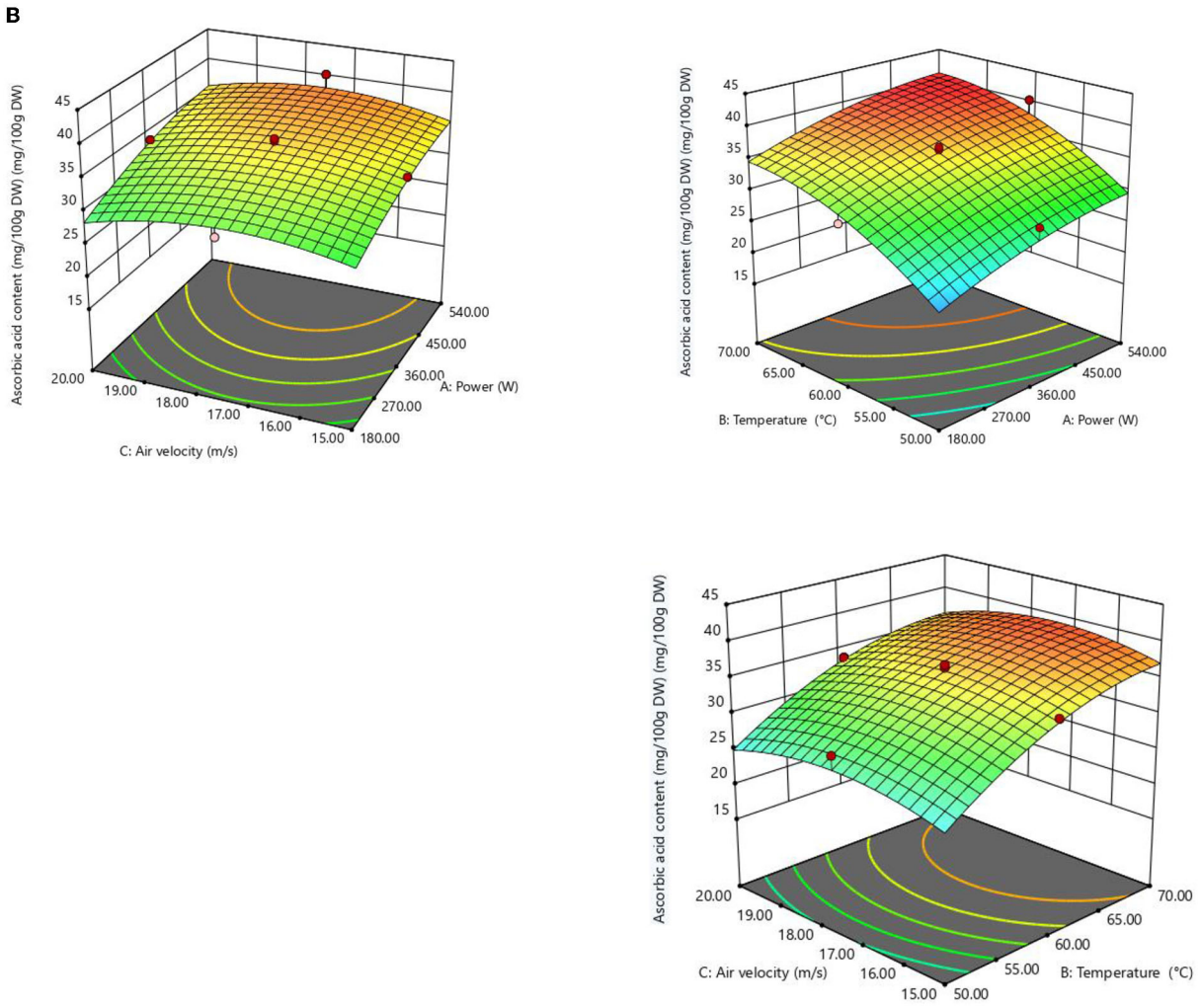

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Inside a microwave oven cavity, air was blown through a drying column by the motorized air blower for fluidization. Also, a heater was used to heat up the air flowing through the drying column. Both the air and temperature are being maintained and controlled by a digital display and magnetic relay type controller unit. The velocity of air was determined by Anemometer (Lutron, United States) ranged from 4–30 m/s, 1.4 108 km/hr, 8–58.3 knots. Moreover, microwave oven has an air vent cavity at the top from where the evaporated vapors can escape. Likewise, perforated bottom allows the flow of air for fluidization into the column. The experiment for the samples (size 250 g) was carried out until constant weight was achieved using microwave power (180–540 W), air temperature (50–70 °C), and inlet air velocity (15–20 m/s). The sample was weighed after each 5 min interval for determining the moisture content. Drying time was kept at 40–60 min depending on the inlet air temperature, air velocity, and microwave power combination. The mass measurement during the drying was determined by taking samples from the drying equipment at time intervals of 5 min and measuring their mass on an analytical balance. The samples were dried until the moisture content was reduced to around 6% (d.b).

Experimental design

The experimental design was based on response surface methodology (RSM) employing a central composite rotatable design (CCRD) to incorporate three independent variables, i.e., microwave power (180–540 W), air temperature (50–70 °C), and inlet air velocity (15–20 m/s). Minimum and maximum levels of independent factors were selected

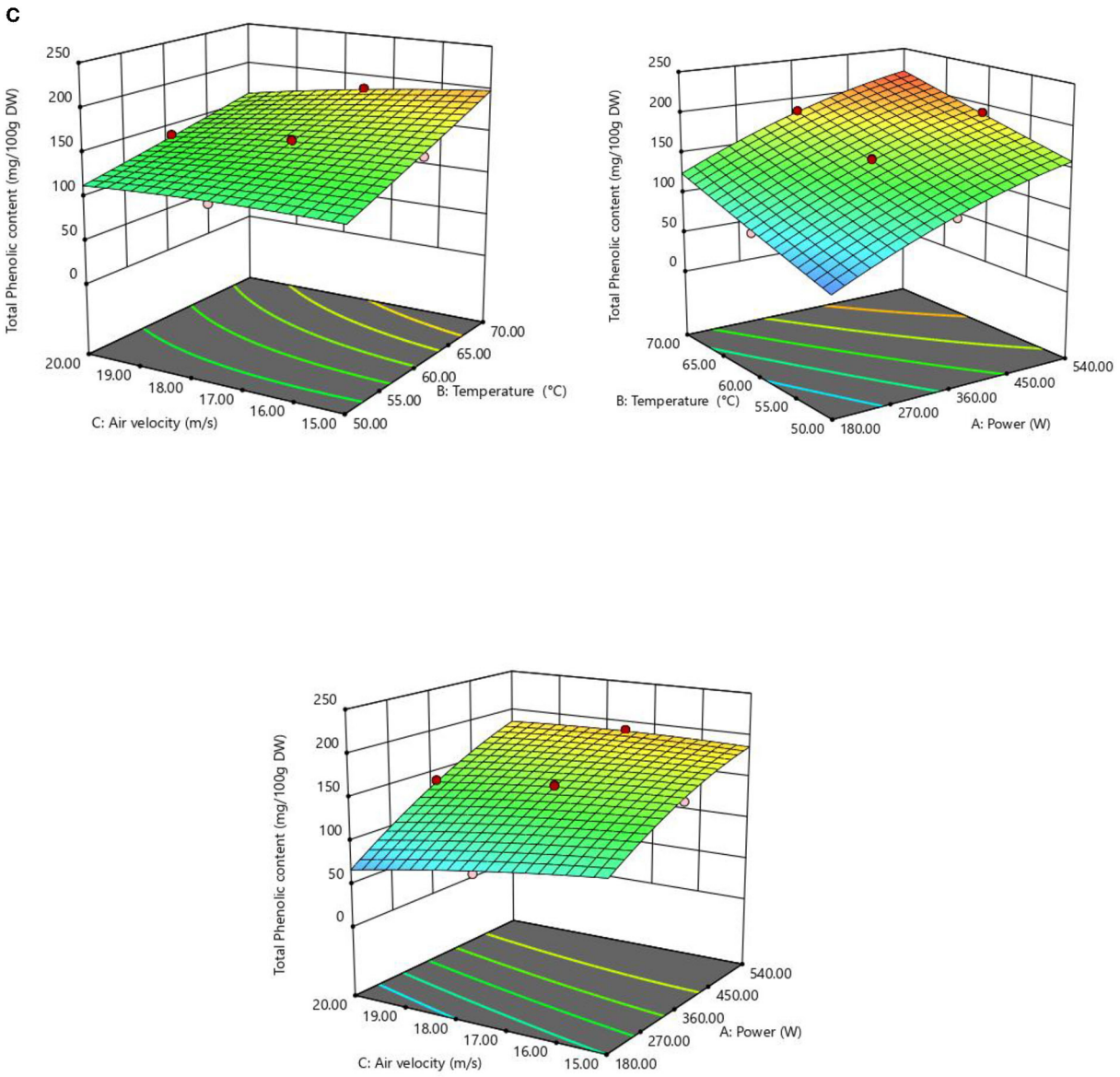

FIGURE 1
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based on the available literature and preliminary trials. The design overall yields five different levels for every variable and allows an improved assessment of their quadratic effects.

Quadratic models were tested to investigate the influence of independent variables on response variables using RSM software Design-Expert version 7 (Stat-Ease, Minneapolis, MN, USA). Data were fitted to a second-order polynomial model to achieve regression analysis and coefficients of the model with p-values ≤ 0.05 as shown in the equation below. The significance of regression ($p \leq 0.05$) and lack of fit (>0.05) were evaluated by analysis of variance (ANOVA) from Minitab software.

$$Y_K = \beta_{KO} + \sum_{i=1}^{3} \beta_{ki} X_i + \sum_{i=1}^{3} \beta_{kij} X_i^2 + \sum_{i \neq 1}^{3} \beta_{kij} X_i X_j \quad (1)$$

where β_{ko}, β_{ki}, and β_{kij} are the constants, linear, quadratic, and cross product regression coefficients, respectively and X_i's are the coded independent variables of microwave power, temperature, and inlet air velocity.

The optimal criteria were done by selecting a design goal such as maximize, minimize, target, within range, none (for

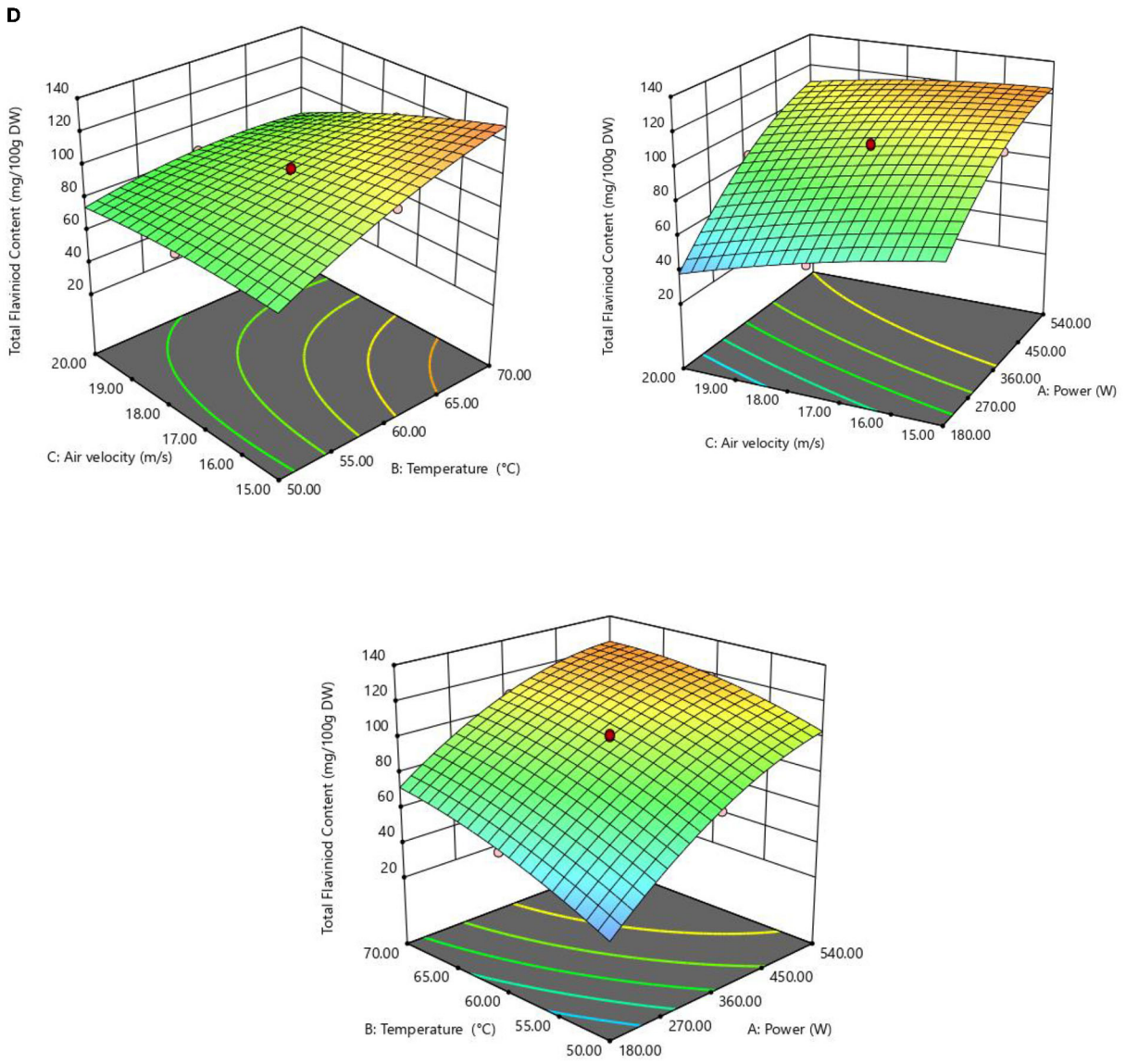

FIGURE 1
(Continued)

response only), and to an exact value (factor only) for each independent response variable. The goals were then combined into an overall desirability function. For the different importance values, the desirability function is given as,

$$D = (d_1^{r1} x d_2^{r2} x \ldots\ldots x d_n^{m})^{1} / \sum ri = \left(\sum_{j=1}^{n} d_j^{ri}\right)^{1/} \sum ri \quad (2)$$

where n is the total number of the responses and $d_i^{'}s$ are the values according to which goals for each response were set.

Quality parameters

Ascorbic acid activity

Ascorbic acid (AA) activity was estimated as per the procedure of Yousuf and Srivastava (2017) by using the following formula:

$$AA\left(\frac{mg}{100gDW}\right) = \frac{Titre \times Dye\ factor \times vol\ made\ up \times 100}{Aliquot\ taken\ for\ estimation \times sample\ weight} \quad (3)$$

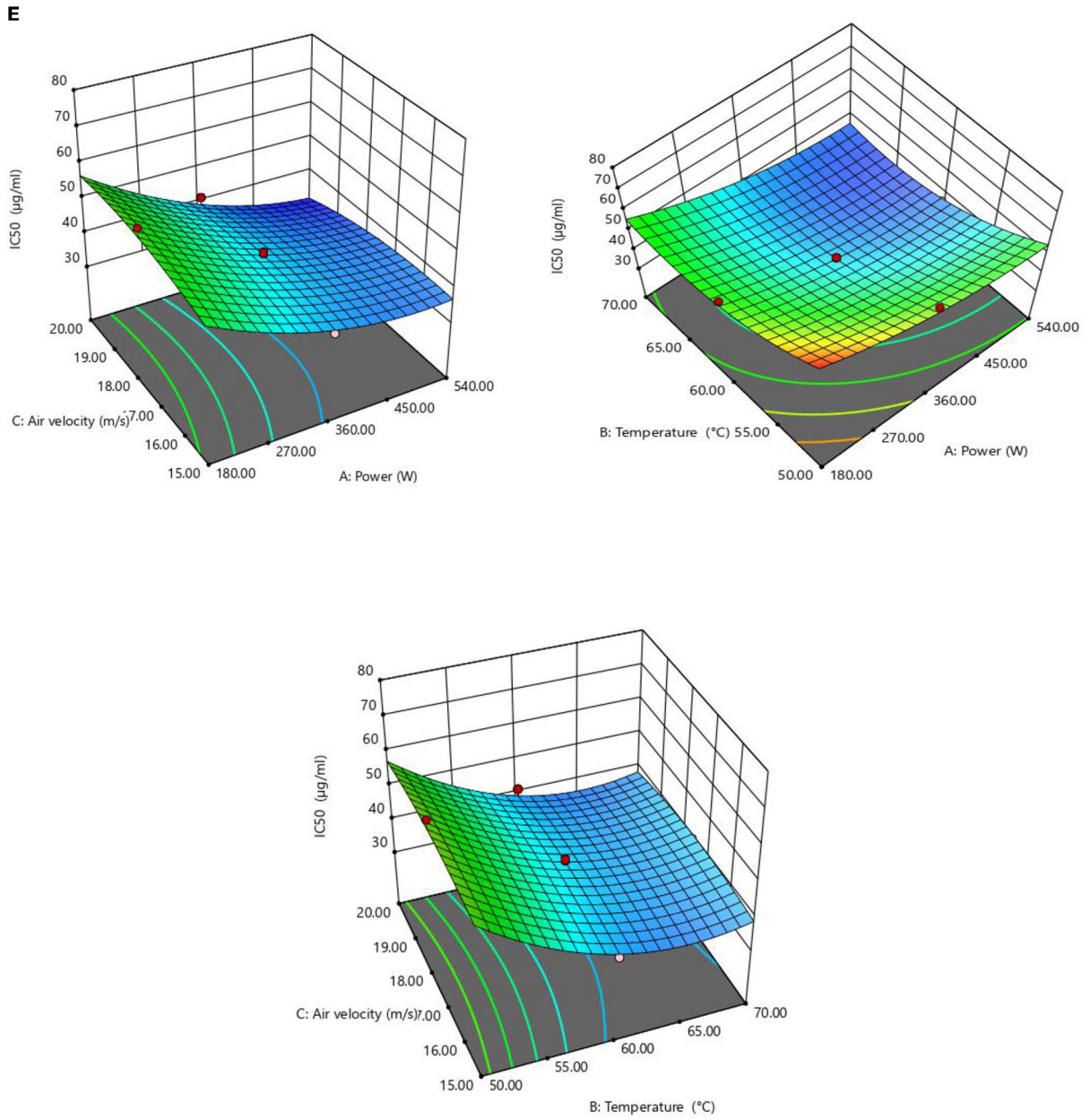

FIGURE 1
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Total phenolic content

The procedure of Zahoor and Khan (2021) was used to calculate the TPC in dried samples. Methanol as a solvent was used for the extraction process. Two grams of dried chicory leaves were homogenized in 20 mL of methanol. The homogenate was then kept undisturbed for 12 h. The obtained mixture was centrifuged at 10,000 g for 15 min. After centrifugation, 0.2 mL of aliquot is mixed with 1.5 mL of Folin–Ciocalteu reagent and 1.2 mL of 7.5 % of Na_2CO_3. The mixture was then kept aside for 2 h at 25 °C. Lastly, the absorbance was measured by a spectrophotometer at 765 nm. A calibration curve was made with gallic acid and the TPC was expressed as mg GAE/100g of dry sample. The calculation of total phenolic contents in a sample will be as follows:

$$T = C \times V/M.$$

T is the total phenolic content in mg/100g of the extracts as GAE, C is the concentration of gallic acid established from the

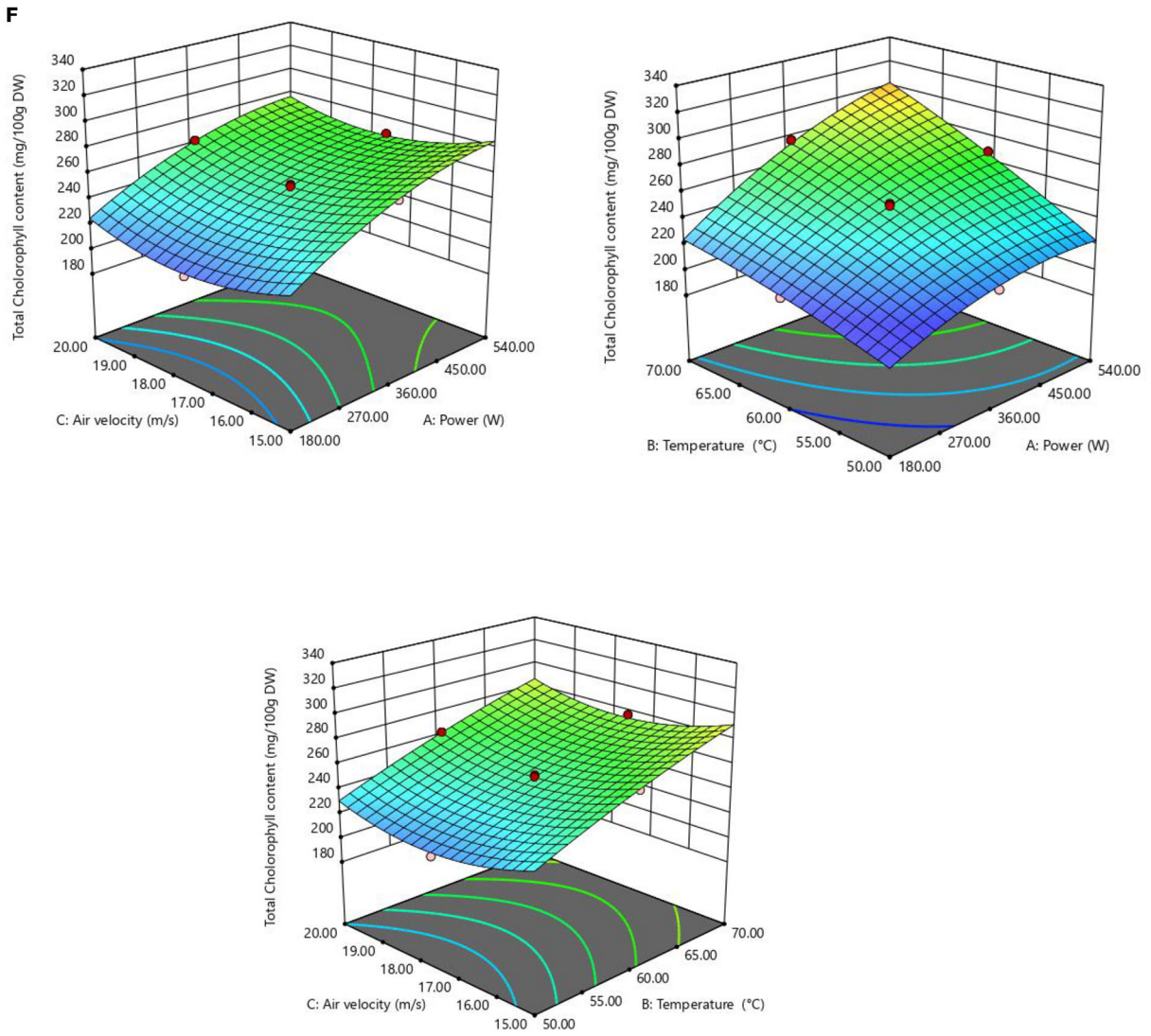

FIGURE 1
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calibration curve in mg·ml^{-1}, V is the volume of the extract solution in ml, and M is the weight of the extract in g.

Total flavonoid content

Total flavonoid content (TFC) was estimated by a procedure by Zou et al. (2004). About 0.1 mL of extract was taken in a 10 mL volumetric flask in which methanol and 4 mL of distilled water were added. Initially, 0.3 mL of $NaNO_3$ (5g/dL) was added to the flask and kept undisturbed for 5 min. Supplementation of 0.3 mL of $AlCl_3$ (10g/dL), followed by the addition of 2 mL of NaOH (1mol/L) to the mixture. The mixture was then immediately diluted to 2.4 mL with deionized water and absorbance was measured at 510 nm. Quercetin was used as a standard for calibration and TFC was expressed as mg QE/100DW.

DPPH radical scavenging activity

1,1-Diphenyl-2-picryl-hydrazyl-hydrate was estimated by the method of Lv et al. (2018) with slight modification. Methanol was used to prepare 0.1 mM of DPPH solution. Extraction of different concentrations was made, followed by the addition of 5 mL of DPPH solution and mixed properly. The mixture was left undisturbed for about 20 min in dark at room temperature. The absorbance of the sample was measured at 517 nm. DPPH radical scavenging activity was calculated by

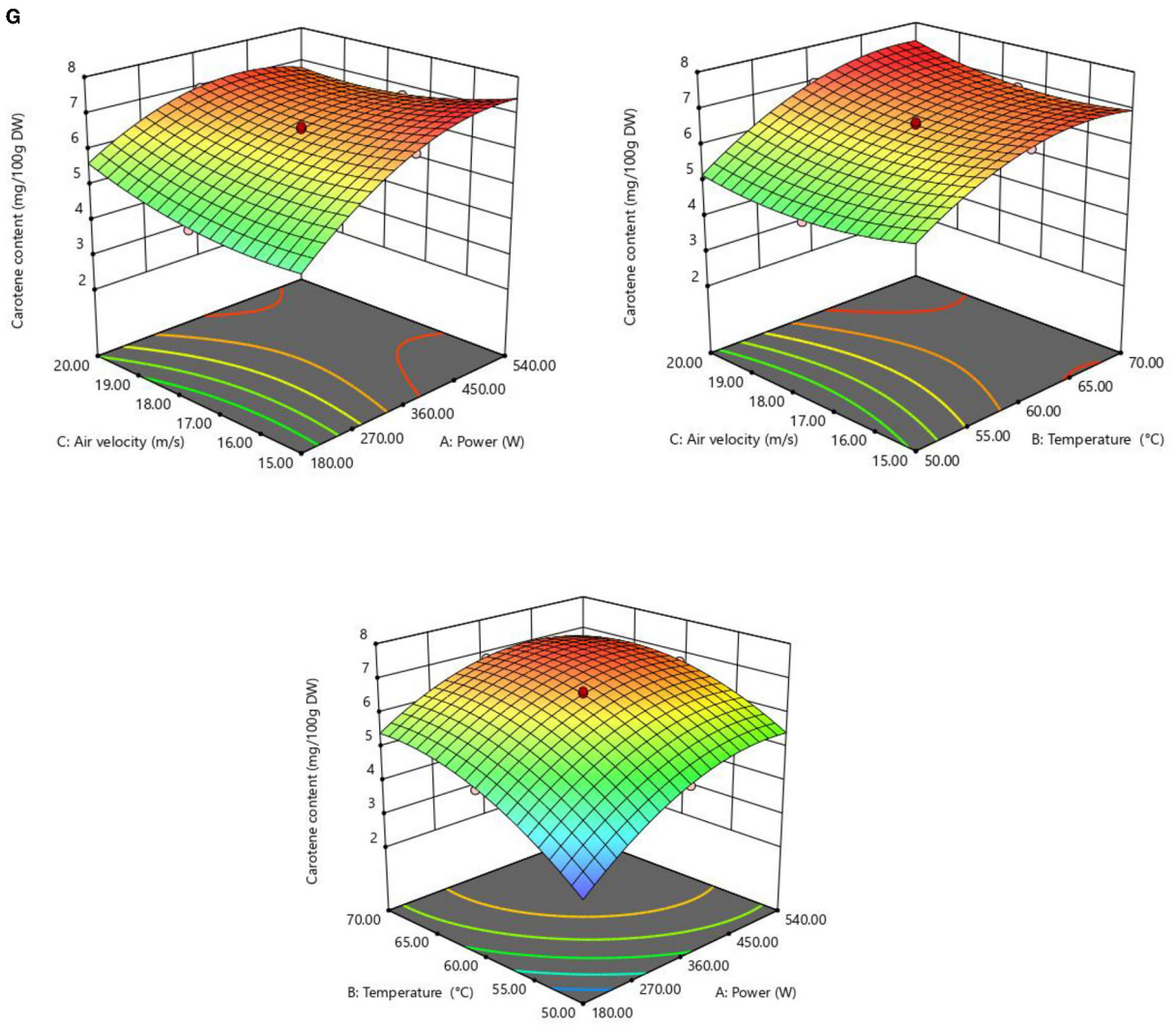

FIGURE 1
(Continued)

the following equation:

$$\%\ Inhibition = \frac{A_{control} - A_{sample} \times 100}{A_{sample}} \quad (4)$$

where $A_{control}$ and A_{sample} are the absorbance of control and sample, respectively.

$A_{control}$ = Absorbance of 0.1 mM of DPPH solution prepared in methanol.

Total chlorophyll content

Total chlorophyll content (TCC) was estimated according to the procedure followed by Mehta et al. (2017). Briefly, to 20 mL of 80 g/dL of acetone, 1 g of sample was added. This step was done repeatedly till colorless residues were formed and then 80 g/dL of acetone was added in order to make up the total volume up to 100 mL. This step was again repeated and the absorbance of extraction was read by a spectrophotometer at 645 nm and 663 nm using the following equation:

$$CC\ (\mu g/mL) = 20.2(A_{645}) + 8.02(A_{663}) \quad (5)$$

where A_{645} and A_{663} are the absorbance of solution at 645 nm and 663 nm, respectively.

Carotene content

Carotene content (CC) was measured by the method given by Abbas et al. (2015). Dried samples were weighed and 10–15 mL of acetone, petroleum ether, and few crystal of anhydrous sodium sulfate were added to the crushed sample.

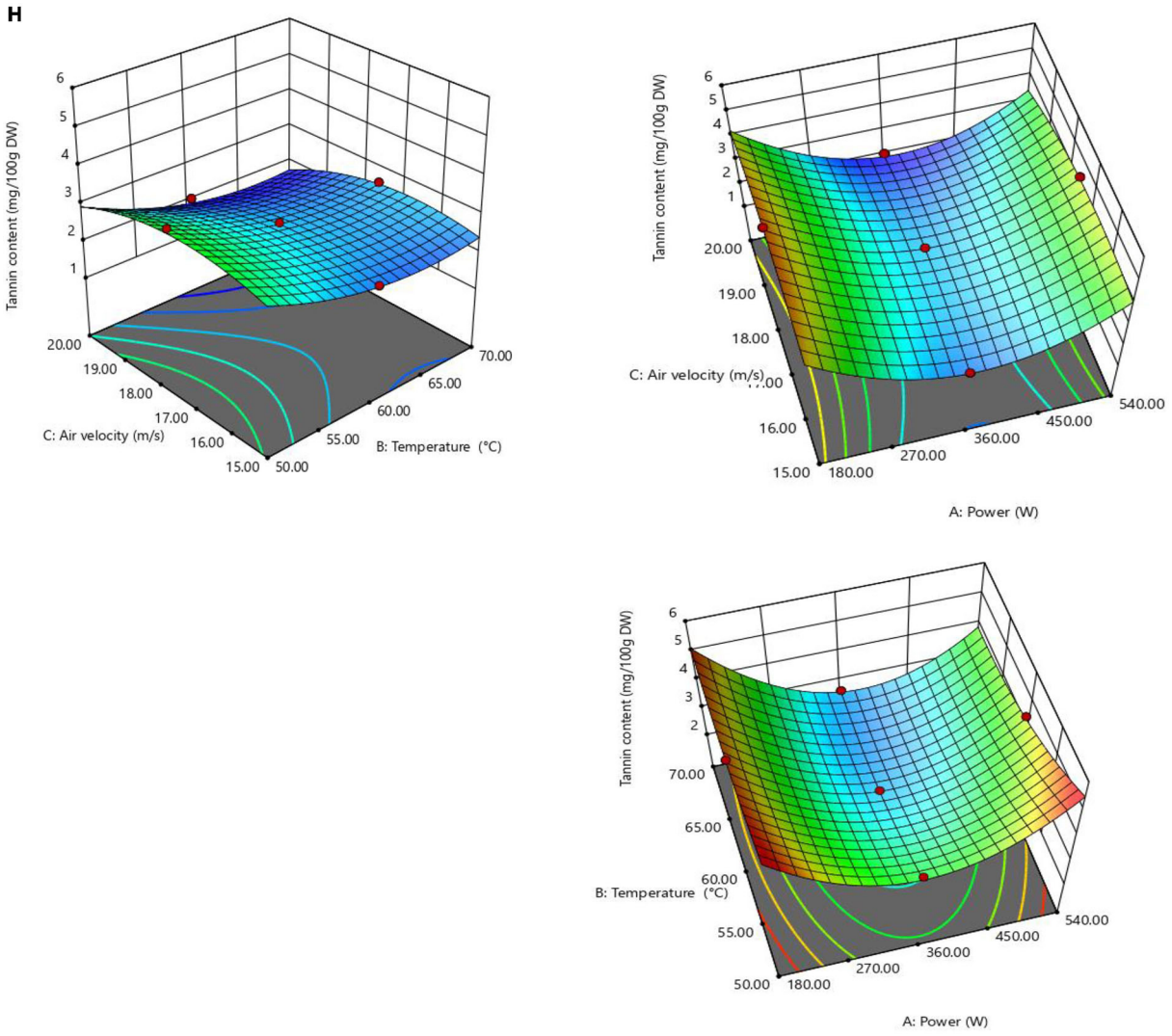

FIGURE 1
(Continued)

This procedure was again repeated and the supernatant obtained was mixed with 10–15 mL of petroleum ether, so as to get two separate layers. The bottom layer was removed and the volume of the upper layer was made up to 100 mL of petroleum ether. OD was measured at 450 nm by using the below equation:

$$\beta - \text{carotene(mg/100 g)} = \text{O.D.} \times 13.9 \times 10^4 \times 100/\text{weight of sample(g)} \times 560 \times 1000$$

Tannin content

Tannin content (TC) was estimated according to the method done by Chai et al. (2018) with little modifications. Five grams of sample were boiled in deionized water and then filtered. Two to three drops of 0.1 % ferric chloride solution were added to the filtrate. The presence of tannins in the sample is indicated by the color change blue-black or brownish green color.

Saponin content

Saponin content (SC) was measured according to the procedure followed by Abbas et al. (2015). In a beaker, 10 g of ground sample were mixed with 100 cm^3 of 20% aqueous ethanol and stirred with a magnetic stirrer for 12 h at 55°C. The solution was filtered through Whatman No. l filter paper and the residue was extracted again with 200 cm^3 of 20% aqueous ethanol. Under vacuum, the extracts were mixed and reduced to about 40 cm^3. The extract and 20 cm^3 diethyl ether were combined in a 250 cm^3 separating funnel and vigorously shaken. The aqueous layer was removed. The purification process was

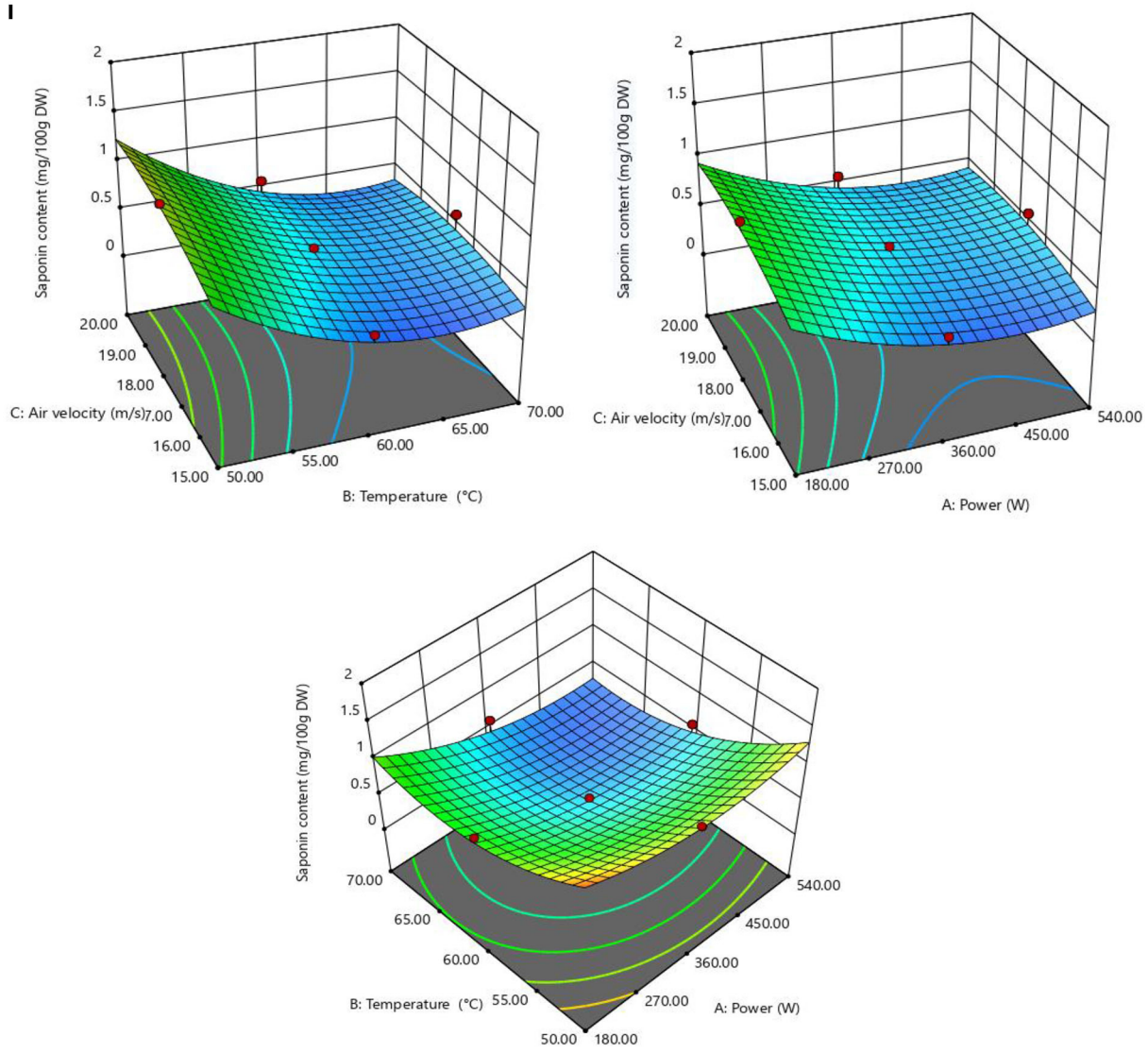

FIGURE 1
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repeated until a colorless aqueous extract was obtained. The pH of the remaining aqueous solution was adjusted to 4.5 by adding 4 g of NaCl and shaking it with 60 and 30 cm^3 portions of n-butanol. The butanolic extract was washed two times with 10 cm^3 of 5% aqueous sodium chloride, and then dried in a fume cupboard to yield saponin, which was weighed and expressed as mg/100g.

Results and discussion

The current study aimed to assess the influence and interaction of drying variables on the responses. The independent variables were also assessed to obtain the optimum level of variables. The optimum variables were then validated and analyzed for the investigated responses. The results of the ANOVA are shown in Table 1.

Ascorbic acid content

Vitamin C is relatively unstable to heat, oxygen, and light, so, its retention can be used as a quality indicator of drying conditions. Because vitamin C is essential for human nutrition and is used as a quality indicator in food processing (Wang et al., 2020). Santos and Silva (2008) investigated vitamin C retention in fruits and vegetables during drying processes. They concluded that temperature and time are the most important

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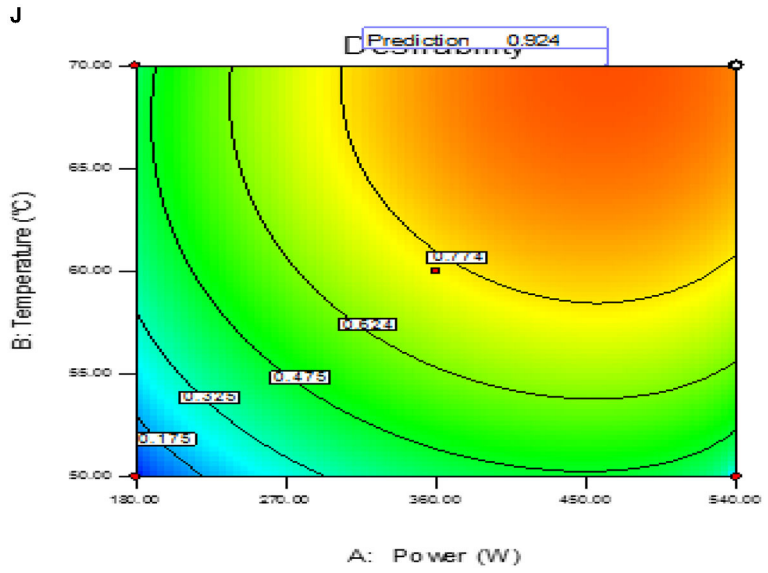

FIGURE 1
(A) Experimental apparatus (Microwave assisted fluidized bed drying system) (I) schematic diagram (II) in a working condition. **(B)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on ascorbic acid content. **(C)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on total phenolic content. **(D)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on total flavonoid content. **(E)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on DPPH radical scavenging activity. **(F)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on total chlorophyll content. **(G)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) on carotene content. **(H)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) tannin content. **(I)** Response surface plots showing combined effect of microwave power, temperature and inlet air velocity) saponin content. **(J)** Desirability graph at air velocity of 15m/s.

environmental variables influencing vitamin C degradation. The concentration of oxygen in the drying atmosphere also has an effect on the final content of the dried product. However, because different fruits and vegetables have different chemical and physical properties, more research is needed to provide a better understanding of the oxidative phenomena of vitamin C during the drying processes. Drying conditions caused a significant change in the AA content. In dried chicory, the ascorbic acid content varied from 19.78 to 40.23 mg/100 g DW depending on the drying conditions. The lowest content of ascorbic acid was found at 180 W, 50 °C, and 20 m/s, and the highest ascorbic acid was obtained at 540 W, 60 °C, and 17.50 m/s (Table 2), suggesting that lower temperature (50 °C) and power (180 W) caused higher degradation of ascorbic acid in dried pepper than higher temperature (70 °C) and power (540 W). Unlike what would be expected during microwave drying, the ascorbic acid content increased rather than decreased, despite the fact that ascorbic acid is heat liable. The reason could be that the lower temperature used during drying required more drying time, which accelerated ascorbic acid oxidation (Horuz et al., 2017). A higher temperature and microwave power substantially reduced the drying time needed to reduce final moisture. This means that in order to achieve the same final moisture content, higher drying temperature gives faster drying process. It is apparent from Table 2 that temperature is the most paramount factor affecting the ascorbic acid content. The fitted model for AA is presented in Equation (6).

$$Y = 35.98 + 3.39X_1 + 5.85X_2 - 2.73{X_2}^2 - 2.47{X_3}^2 \quad (6)$$

where X_1 is the microwave power; X_2 is the air temperature; X_3 is the inlet air velocity.

Table 3 shows a significant effect of linear terms of temperature and microwave power on the AA content of the dried sample. However, the interaction between them did not show any significant effect on the AA of dried chicory. Temperature and drying time play an important role in AA degradation. From Figure 1B, the AA content enhanced initially with increment in temperature and air velocity upto 60 °C and 17.5 m/s, respectively and then declines with further increment. The reason could be the deterioration of ascorbic acid due to its sensitivity at both higher temperatures and lower temperatures (longer drying operations. Our results are in agreement with those previously reported by Zahoor and Khan (2019) who reported the impact of microwave convective drying on bitter gourd. They reported that an increase in the ascorbic acid content at higher power and temperature could be ascribed to the inactivation of AA oxidase. High-temperature short time

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TABLE 1 Analysis of Variance (ANOVA) of the fitted second order polynomial model for various responses and second order polynomials model equations for investigated responses.

Source	Sum of squares	DF	Mean square	*F*-value	*p*-value
Ascorbic acid content					
Model	615.70	9	68.41	36.05	<0.0001
Residual	18.98	10	1.90	18.98	
Lack of fit	15.27	5	3.05	4.13	0.0729
Pure error	3.70	5	0.74	3.70	
Cor total	634.68	19			
$R^{2,a} = 0.9701$; $CV^b = 4.21\%$					
Total phenolic content					
Model	37,553.00	9	4172.56	584.12	<0.0001
Residual	71.43	10	7.14		
Lack of fit	59.36	5	11.87	4.92	0.0527
Pure error	12.08	5	2.42		
Cor total	37,624.44	19			
$R^2 = 0.9981$; CV = 1.86%					
Total flavonoid content					
Model	15,835.03	9	1,759.45	646.20	<0.0001
Residual	27.23	10	2.72		
Lack of fit	22.63	5	4.53	4.93	0.0525
Pure error	4.59	5	0.92		
Cor total	15,862.25	19			
$R^2 = 0.9983$; CV = 1.82%					
DPPH scavenging activity					
Model	2,613.43	9	290.38	66.17	< 0.0001
Residual	43.88	10	4.39		
Lack of fit	36.60	5	7.32	5.03	0.0504
Pure error	7.28	5	1.46		
Cor total	2,657.31	19			
$R^2 = 0.9835$; CV = 4.44%					
Total chloloropyll content					
Model	17,421.25	9	1,935.69	509.97	< 0.0001
Residual	37.96	10	3.80		
Lack of fit	28.75	5	5.75	3.12	0.1185
Pure error	9.21	5	1.84		
Cor total	17,459.21	19			
$R^2 = 0.9978$; CV = 0.78%					
Carotene content					
Model	26.31	9	2.92	924.18	< 0.0001
Residual	0.032	10	3.163E-003		
Lack of fit	0.019	5	3.870E-003	1.58	0.3151
Pure error	0.012	5	2.457E-003		
Cor total	26.34	19			
$R^2 = 0.9988$; CV = 0.93%					
Tannin content					
Model	24.52	9	2.72	267.97	< 0.0001
Residual	0.10	10	0.010		
Lack of fit	0.082	5	0.016	4.20	0.0705

(Continued)

TABLE 1 (Continued)

Source	Sum of squares	DF	Mean square	*F*-value	*p*-value
Pure error	0.020	5	3.907E-003		
Cor total	24.62	19			
$R^2 = 0.9959$; CV = 2.83%					
Saponin content					
Model	3.23	9	0.36	29.07	< 0.0001
Residual	0.12	10	0.012		
Lack of fit	0.099	5	0.020	3.93	0.0797
Pure error	0.025	5	5.017E-003		
Cor total	3.36	19			
$R^2 = 0.9632$; CV = 15.94%					

[a]Coefficient of determination. [b]Coefficient of variance (%).

heating results in the inactivation of enzymes, leading to the disruption of matrix and release of more AA content that are being transferred to the other chicory parts.

Total phenolic content

Chicory contains a good amount of total phenolics. However, drying may affect the total phenolic content of chicory. Depending on different drying conditions, the TPC of the sample varied from 43.23 to 225.05 mg/100g DW. The highest total phenolics were found at 540 W, 70 °C, and 15 m/s of MP, AT, and AV, respectively (Table 2). The greater retention of polyphenols at higher power and temperature could be due to the destruction of oxidative enzymes during faster and shorter heating. The fitted model for TPC is presented in Equation (7).

$$Y = 150.27 + 49.16X_1 + 32.01X_2 - 10.51X_3 - 1.38X_1X_2 + 9.88X_1X_3 - 7.85X_2X_3 - 10.61{X_2}^1 - 3.06{X_3}^2 \quad (7)$$

Results from Table 3 depicted that all linear and quadratic terms of temperature showed a significant effect on total phenolic content. Moreover, MWP–AV and AT–AV interactions also showed a significant effect. A positive effect of microwave power and temperature on TPC is shown in Figure 1C, indicating a better retention of total phenolics with an increase in AT and MWP Higher temperature and microwave power heat the product quickly, resulting in the protection of phenolic compounds. Hamrouni-Sellami et al. (2013) studied the effect of different drying methods on the TPC of sage (*Salvia officinalis L.)*. They explained that highest contents of phenolics are due to the microwaves causing the production of elevated temperature and vapor pressure within the plant tissue, which results in cell wall disruption causing more phenolics to be liberated. Moreover, it has been shown in the literature that different drying techniques affect the TPC of studied material and that the effect of different drying methods on the TPC is compound-dependent, which means that the consistency of phenolic compounds varies under different drying operations (Šumić et al., 2013). However, an increase in air velocity at a given microwave power and temperature extended the drying time and hence less retention of TPC.

Total flavonoid content

Flavonoids are essential phytochemicals that are accountable for the protective result of vegetables. Different drying conditions result in a varied range of TFC (37.45–138.86 mg/100g DW) of dried chicory. From Table 2, TFC is reduced at lower power and temperature, while total flavonoid was better retained at 540 W, 70 °C, and 15 m/s. This might be attributed to the disruption of the cell wall of chicory tissue during drying, which leads to the increased extraction of compounds from the sample (Wang et al., 2020). The fitted model for TFC is presented in Equation (8).

$$Y = 100.35 + 30.66X_1 + 14.91X_2 - 10.51X_3 - 4.43X_1X_2 - 10.18X_2X_3 - 11.36{X_2}^1 - 5.50{X_2}^2 - 2.85{X_3}^2 \quad (8)$$

From the statistical analysis, all linear, quadratic, and interaction terms of drying conditions showed a significant effect on TFC. From Figure 1D, microwave power and temperature showed a positive effect on TFC. The findings of this research are in parallel with the findings of Rababah et al. (2015) who investigated the impact of the drying method on the content of flavonoids of common Mediterranean herbs. They also reported an escalation in flavonoids is due to the increasing temperature and power decreased the enzyme activity of flavonoids to degrading enzymes such as polyphenol oxidase. Moreover, an increase in microwave power caused a reduction in drying time and better retention of flavonoids. This decrease in drying time

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TABLE 2 Experimental results of investigated responses obtained under different drying conditions.

Run	Independent variables			Responses							
	Power (W)	Temperature (°C)	Velocity (m/s)	Ascorbic acid content (mg/100g)	TPC (mg GAE/100g)	TFC (mg QE/100g)	DPPH (μg/ml)	Chlorophyll Content (mg/100g)	CC (mg/100g)	Tannin Content (mg/100g)	Saponin Content (mg/100g)
1	180.00	50.00	15.00	20.33	69.03	37.45	68.55	208	3	5.12	1.2
2	540.00	50.00	15.00	25.87	150	98.93	56.23	243	6.3	5.2	1.1
3	180.00	70.00	15.00	33.33	151.3	98.3	52.63	240	5.18	4.9	0.9
4	540.00	70.00	15.00	38.66	225.05	138.86	36.53	320.85	7.23	3.8	0.14
5	180.00	50.00	20.00	19.78	43.23	24.27	74.64	206.44	3.56	5	1.6
6	540.00	50.00	20.00	26.63	162	103.2	48.26	234	5.3	4.7	1.3
7	180.00	70.00	20.00	32.33	92.4	41.19	54.23	236.96	6.4	4.3	0.9
8	540.00	70.00	20.00	38.23	207.35	105.6	32.12	312	7	3	0.15
9	180.00	60.00	17.50	29.92	87.2	57.04	59	209.22	4.78	5	1
10	540.00	60.00	17.50	40.23	190.37	118.26	34.93	267.93	6.72	4.3	0.57
11	360.00	50.00	17.50	29.33	116	79.02	62.32	215.33	4.9	3.7	1.2
12	360.00	70.00	17.50	37.93	184.24	108	39.23	277.12	6.81	2.6	0.59
13	360.00	60.00	15.00	34.23	153.68	107.99	38.26	266.59	6.8	2.4	0.35
14	360.00	60.00	20.00	33.56	139	84.33	40.94	262.35	6.93	2.01	0.56
15	360.00	60.00	17.50	35	150	102	40.36	252	6.6	2.5	0.45

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TABLE 3 Estimated regression coefficients of the second order polynomial model for responses (in coded units).

Regression coefficient	Ascorbic acid content	Total phenolic content	Total flavonoid content	DPPH radical scavenging activity	Total chlorophyll content	Carotene content	Tannin content	Saponin content
β_0	35.98	150.27	100.35	41.29	249.81	6.56	2.61	0.46
Linear								
β_1	3.39[a]	49.16[a]	30.66[a]	−10.10[a]	27.72[a]	0.96[a]	−0.33[a]	−0.23[a]
β_2	5.85[a]	32.01[a]	14.91[a]	−9.53[a]	28.02[a]	0.96[a]	−0.51[a]	−0.37[a]
β_3	−0.19	−10.51[a]	−12.29[a]	−0.20	−2.67[a]	0.068[a]	−0.24[a]	0.082[a]
Cross product								
β_{12}	−0.14	−1.38	−4.43[a]	0.061	11.67[a]	−0.30[a]	−0.27[a]	−0.14[a]
β_{13}	0.24	9.88[a]	5.16[a]	−2.51[a]	−1.66[a]	−0.38[a]	−0.072	−0.024
β_{23}	−0.20	−7.85[a]	−10.18[a]	−0.12	−0.17	0.18[a]	−0.097[a]	−0.074
Quadratic								
β_{11}	−1.29	−10.61[a]	−11.36[a]	5.10[a]	−11.08[a]	−0.77[a]	1.95[a]	0.23[a]
β_{22}	−2.73[a]	0.72	−5.50[a]	8.91[a]	−3.43[a]	−0.66[a]	0.45[a]	0.34[a]
β_{33}	−2.47[a]	−3.06	−2.85[a]	−2.26	14.82[a]	0.35[a]	−0.49[a]	−0.099

[a]Significant at 0.05 level.

and retention in flavonoids is because of the generation of larger vapor pressure difference between the surface and the interior of the product upon the penetration of microwave energy. The microwave power absorption accelerates the drying process by increasing the internal pressure and concentration gradient and hence better retention of flavonoids (Zahoor and Khan, 2021).

DPPH radical scavenging activity

The antioxidant activity of chicory was ascertained by the widely used DPPH radical scavenging activity. DPPH activity of the sample varied from 32.12 to 74.64 (μg/ml) depending on the different drying conditions. Maximum antioxidant activity, i.e., lowest IC_{50} value was observed at 540 W, 70°C, and 20 m/s and minimum antioxidant activity, i.e., highest IC_{50} was found at 180 W, 50 °C, and 20 m/s (Table 2). The fitted model for AA is presented in Equation (9).

$$Y = 41.29 - 10.10X_1 - 9.53X_2 - 2.51X_1X_3 + 5.1{X_2}^1 + 8.91{X_2}^2 \quad (9)$$

According to the results from Table 3, the interaction between power and temperature and temperature and velocity did not show any significant effect. However, a significant effect was observed on all the linear and quadratic terms of MWP and AT on the DPPH radical scavenging activity (Figure 1E), depicting that an increase in power and temperature showed increased antioxidant activity (AOA). The production of melanoidins improves the antioxidant effect at elevated temperatures. Moreover, an increase in AOA could be due to the greater antioxidant power of polyphenols at intermediate stages of oxidation or the production of newer compounds with greater antioxidant activity (Zahoor and Khan, 2019). Furthermore, as stated by Samoticha et al. (2016), the outputs of enzymatic and non-enzymatic browning reactions during the drying process can cause antioxidative properties.

Total chlorophyll content

The total chlorophyll content (TCC) of chicory was significantly influenced by drying conditions. TCC varied from 208 to 312 mg/100g in dried samples. The highest and lowest chlorophyll content was found at 540 W, 70°C, and 20 m/s and 180 W, 50 °C, and 15 m/s, respectively (Table 1). The fitted model for AA is presented in Equation (10).

$$Y = 249.81 + 27.72X_1 + 28.02X_2 - 2.67X_3 + 11.67X_1X_2 - 1.66X_1X_3 - 11.08{X_2}^1 - 3.43{X_2}^2 + 14.82{X_3}^2 \quad (10)$$

Table 3 shows a significant effect of all linear and quadratic terms of AV, AT, and MWP on the total chlorophyll content of

dried chicory. Also, the interaction between power–temperature and power–velocity showed a significant influence on TCC. From Figure 1F, the effect of power and temperature can be visualized, in which higher power and temperature incremented the chlorophyll content. This could be attributed to the quick rate of drying at higher power and temperature responsible for the better retention of the chlorophyll content. Moreover, faster and shorter drying caused inactivation of enzymes related to color, resulting in the retention of chlorophyll content of the sample (Mehta et al., 2017). In addition to this, there is a competition between peroxidase enzyme and chlorophyllase and it is extensively illustrated that chlorophyll is unstable chemically and is therefore can be degraded easily upon heating (Cao et al., 2020). However, an increase in air velocity at a given microwave power and temperature showed a negative effect on TCC. This may be due to the extended drying time at higher air velocity and the flow of air at higher velocity would actually cool the samples and reduce the temperature at the surface of the product and thus resulting in a decrease in moisture diffusivity (Zahoor and Khan, 2021).

Carotene content

The drying conditions vary the CC of dried chicory ranged from 3 to 7.23 mg/100g. The maximum and minimum carotene content was found at 540 W, 70 °C, and 15 m/s and at 180 W, 50 °C, and 15 m/s, respectively (Table 2). The fitted model for AA is presented in Equation (11).

$$Y = 6.56 + 0.96X_1 + 0.96X_2 - 0.068X_3 - 0.30X_1X_2 - 0.38X_1X_3 + 0.189X_2X_3 - 0.77{X_2}^1 - 0.66{X_2}^2 + 0.35{X_3}^2 \quad (11)$$

According to the results from Table 3, all linear terms of power, temperature, and velocity showed a significant effect. Moreover, all quadratic terms and interaction terms also showed a significant effect on carotene content. From Figure 1G, it could be seen that microwave power and temperature showed a positive effect on carotene content. The greater retention of carotenes maybe due to the exposure of sample for less time to higher power and temperature and thus reduced carotene degradation. Moreover, during longer drying process, thermal energy generated by collisions and high vapor pressure inside plant tissue can lead to the degradation of carotenes (Song et al., 2020).

Tannin content

Tannins are phenolic compounds that are water soluble and occur mainly occur in fruits, barks, and leaves plants. Tannins interfere with the digestion of protein by forming tannin protein complexes (Samtiya et al., 2020). The TC of dried chicory was noticed from 2.01 to 5.2 mg/100g depending on drying conditions. The higher TC was reported at 540 W, 50°C, and 15 m/s and lower at 360 W, 60°C, and 17.5 m/s (Table 2). The fitted model for AA is presented in Equation (12).

$$Y = 2.61 - 0.33X_1 - 0.51X_2 - 0.24X_3 - 0.27X_1X_2 - 0.097X_2X_3 + 1.95X_1 + 0.45{X_2}^2 - 0.49{X_3}^2 \quad (12)$$

According to the statistical indicators, all linear terms of microwave power, temperature and velocity, and quadratic terms showed a significant effect on the tannin content of chicory. Moreover, a significant effect was reported in power–temperature and temperature–velocity interactions. These effects could be seen in Figure 1H, in which power and temperature showed a reverse trend (negative effect) on the tannin content. It means that the tannin content reaches to a minimum with the increment in temperature and power and then starts to increase with the further increase in these variables. Suhag et al. (2021) suggested that water-soluble and heat-labile nature of tannin are mainly responsible for the substantial reduction of tannin at higher temperature and power. Moreover, interactions of tannin with other compounds to form insoluble complexes are the main reason for their reduction in higher power and temperature.

Saponin content

Saponins are secondary, non-volatile metabolites mainly found in plants. The adverse effect of saponins on humans is impairment in growth and decrease in the bioavailability of iron (Fleck et al., 2019). The saponin content of dried chicory varied from 0.14 to 1.6 mg/100g. The highest content of saponin was found at 180 W, 50°C, and 20 m/s and lowest content at 540 W, 70 °C, and 15 m/s (Table 2). The fitted model for AA is presented in Equation (13).

$$Y = 0.46 - 0.23X_1 - 0.37X_2 + 0.082X_3 - 0.14X_1X_2 + 0.23{X_2}^1 - 0.34{X_2}^2 \quad (13)$$

Statistical analysis showed that MWP, AT, and AV had a significant effect on all linear terms. Also, quadratic terms of power and temperature and interaction of power–temperature showed a significant effect. These effects could be seen in Figure 1I, in which power and temperature showed a negative effect on the saponin content, meaning that, the saponin content decreases with an escalation in temperature and microwave power. The reason may be due to the changes in the structure at higher temperature and power, which caused a reduction of saponin content (Vanga et al., 2020).

TABLE 4 Simultaneously optimized microwave assisted fluidized bed drying conditions with target and predicted values of investigated responses.

Variables	Goal	Experimental range		Importance	Values		Variation level (%)
		Lower	Upper		Predicted	Actual	
Power (W)	In range	180	540	3			
Temperature (°C)	In range	50	70	3			
Air velocity (m/s)	In range	15	20	3			
Response							
Ascorbic acid content	Maximized	19.78	40.23	3	38.32	37.72	1.56
Total phenolic content	Maximized	43.23	225.05	3	216.42	217.22	0.36
Total flavonoid content	Maximized	24.27	138.86	3	137.69	136.85	0.61
DPPH scavenging activity	Minimized	32.12	74.64	3	36.10	35.11	2.7
Total chlorophyll content	Maximized	206.44	320.85	3	311.79	312.55	0.24
Carotene content	Maximized	3	7.23	3	7.30	7.38	1.09
Tannin content	Minimized	2.01	5.2	3	2.72	2.67	1.86
Saponin content	Minimized	0.14	1.6	3	0.46	0.47	2.17

Optimized Conditions: 462.30 W; 70.00 °C, 15.00 m/s, $D^a = 0.924$. [a]Desirability function.

Optimization

For a successful execution of different tasks and the development of high-quality food products, optimization is an important parameter. In the mathematical optimization problem, the value of the objective function is either minimized or maximized over the set of viable alternatives. Optimization in the drying process is conducted to get higher efficiency under faster drying conditions. Nevertheless, during the optimization of the drying process, the product quality is characterized by various responses. RSM was used to do the simultaneous optimization of various responses and its desirability function (D).

Numerical optimization of drying conditions was done to obtain the best combination of microwave power, temperature, and air velocity. The optimization was done to maximize AA, TPC, TFC, TFC, CC, and minimize IC_{50}, TC, and SC, while the other responses were kept in range. By applying the desirability function method, optimal conditions of 462.11 W, 70°C, and 15 m/s were attained for eight responses with a desirability value of 0.924 (Figure 1J). The predicted values of responses shown in Table 4 were almost similar to the actual values recorded after following the optimal conditions of power, temperature, and air velocity with a variation of less than 2.7.

Conclusion

In this study, chicory was dried using MAFBD, and RSM was adopted to optimize the process conditions. Taking into account, the maximum amount of total phenolics, total flavonoids, ascorbic acid, total chlorophyll content, carotene content, as well as minimum IC_{50} value, tannin content, and saponin content, the optimum drying conditions of 462.11 W, 70 °C, and 15 m/s were established for MAFBD. The results suggested that using MAFBD is a potential technique with better retention of bioactive components. The graphical and statistical analysis depicted that drying conditions such as power, temperature, and velocity had a significant influence on the responses. The results of this work will provide valuable data for the manufacturers of dryers at industrial scale. However, at the pilot scale, the process still needs further research.

Data availability statement

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author/s.

Author contributions

IZ and FA: conceptualization. IZ and AA-G: methodology. TG and AA: validation. IZ: writing—original draft preparation. NA and SW: writing—review and editing. TG, FA, and SW: visualization. AA-G and AA: supervision. All authors contributed to the article and approved the submitted version.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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