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Microwave assisted drying and extraction technique; kinetic modelling, energy consumption and influence on antioxidant compounds of fenugreek leaves

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Abstract

Fenugreek leaves contains bioactive compounds, which are helpful in maintaining human health. These compounds are adversely affected in conventional drying methods. Therefore, this study was carried out to retain maximum amount of these components through microwave-assisted drying and extraction technique. The technique was optimized for simultaneous drying and extraction process for fenugreek leaves. The drying rate and drying time were influenced by the level of microwave power; time was reduced from 21 to 5 minutes, when microwave power was changed from 30 to 100 W. Unlikely, the drying rate increased from 1.79 to 4.56 ± 0.05 g/(100 g.min) with the change in power (30 to 100 W). Moreover, moisture ratio analysis determined that two term model was the best to demonstrate the fitness with experimental values. The energy consumption was lowest for 100W compared to other powers. Furthermore, comparison between microwave-based extraction and methanol-extraction indicated that the antioxidant activity was better preserved compared to conventional ones even at higher microwave power levels.

Keywords: fenugreek; microwave; bioactive compounds; drying time; energy consumption.

Practical Application: Its application includes drying as well as pharmaceutical industry.

1 Introduction

Fenugreek (*Trigonella foenum graecum L.*) is famous as seasonings and medicinal herb due to aromatic and functional compounds present in it. Its leaves are good source of minerals (iron, calcium, phosphorous), vitamins (vitamin C, riboflavin, niacin, thiamine, folic acid), carotene and flavonoids. These flavonoids are present as complex glycosides (C-glycosidic and O-glycosidic bonds) and among these major flavonoids are quercetin, vitexin, apigenin, etc (Nagulapalli Venkata et al., 2017). These compounds exhibit the potential for the treatment of various ailments e.g. diabetes (type I and type II), chronic cough, diarrhea, ulcer and rickets (Anoopkumar et al., 2020; Aylanc et al., 2020; El Sohaimy et al., 2015).

Fenugreek leaves are usually dried for further use by various methods i.e. sun drying (Shrivastava & Kumar, 2016), Solar drying (Navale et al., 2014; Shrivastava & Kumar, 2016; Singh et al., 2004), hot air drying (Bishnoi et al., 2016; Kalaskar et al., 2012), freeze drying (Kushwaha & Mustafa, 2012), convective drying (Jawake et al., 2017), fluidized bed drying (Jawake et al., 2018) and microwave based drying (Borda-Yepes et al., 2019; Hihat et al., 2017; Khan et al., 2016; Martins et al., 2019; Monton et al., 2019b).

These techniques have high efficiency in drying, but the major concerns are loss of water-soluble bioactive compounds (BACs) and higher processing time & cost (Ferreira et al., 2018; Rocha & Melo, 2011; Thamkaew et al., 2020). Additionally, conventional drying techniques significantly reduce the quality of the product in terms of colour and textural properties (Duc-Pham et al., 2019). Therefore, an emerging drying technique with the purpose of making process of dehydration more sustainable is needed. Microwave based technique (Ali et al., 2020; Khan et al., 2016; Monton et al., 2019a) has exhibited its potential for drying i.e. reduced drying time and cost. Thus, microwave method of drying is energy efficient and easy to use compared to other drying methods (Alvi et al., 2019; Gamboa-Santos & Campañone, 2019; Khan et al., 2016; Lv et al., 2019). However, loss of water-soluble bioactive compounds is still a major problem in microwave-based dehydration systems.

The superiority of microwave-based method urges to develop a novel method that may collect the vapours containing bioactive compounds as a superfluous improvement in addition to drying process. Therefore, present study focuses on the modification and optimization of microwave assisted drying and extraction (MADE) technique. In this regard, fenugreek leaves are used as model product for extraction and drying purposes. The extracted liquid obtained from microwave extraction method was compared

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with methanol-based method. While, the dried product was compared with fresh leaves of fenugreek to estimate the impact of microwaves on leaves.

2 Materials and methods

2.1 Procurement of raw materials

Fresh fenugreek leaves were procured from farms of University of Agriculture, Faisalabad-Pakistan. The impurities & dust particles were removed from fenugreek leaves through washing with running water. Afterwards, leaves were placed on tissue paper for the removal of excess water from their surface. These leaves were used for further experiments. Additionally, chemicals were procured from well-known company Sigma Aldrich (Germany).

2.2 Drying and extraction process

Drying of fenugreek leaves was done through microwave assisted extraction and drying technique. In this regard, an experimental setup was built in lab facility which consist of a modified microwave oven HDG 236S (Homage, Korea) as shown in Figure 1. The samples of leaves (50 g) were placed in glass reactor for drying purpose to extract the bioactive compounds from these leaves. The oven was operated at different powers (30, 50, 80 and 100 W). The loss in moisture was investigated by measuring the weight of the samples at different time intervals (1, 2, 3, 4, ……22 minutes) with the help of weighing balance (Shimadzu, Japan). The water vapors containing bioactive compounds were liquified with the help of condenser (Figure 1) in glass bottles and preserved at 4˚C for further analysis. Additionally, methanol-based extraction was performed for the comparison point of view. For this purpose, sample (5 g) homogenized in methanol solution (80%) for 45 minutes at 8000 rpm. The supernatant layer was removed from the rest of material in tube and utilized for the TFC, TPC and DPPH analysis (Justine et al., 2019).

2.3 Drying rate

The values of drying rate for fenugreek leaves were calculated by using Equation 1 and expressed as g water/100 g.min.

$$Drying\ rate = \frac{V_t - V_{t+dt}}{dt} \quad (1)$$

where, t is drying time in minutes, V_t represents the moisture contents at time t, while V_{t+dt} are the moisture contents at t + dt interval (Alvi et al., 2019; Yilmaz & Alibas, 2017).

2.4 Moisture ratio

The moisture ratio of a product demonstrates the relative removal of water contents and calculated as below (Equation 2).

$$Moisture\ ratio\left(MR\right) = \frac{M_t - M_e}{M_o - M_e} \quad (2)$$

where, M_t, M_o, and M_e represents moisture levels in leaves at time interval of t, initial and equilibrium, respectively. This equation for moisture ratio was further modified to M_t/M_o by some researchers for ease in calculations as described in the literature (Al-Harahsheh et al., 2009; Doymaz, 2005; Karacabey, 2016). Besides, the most important aspect of drying process is mathematical modelling that may help to design drying equipment with optimum drying of desired product. The modelling was based on mathematical equations that can portray the system of drying (Sunil et al., 2014). Therefore, different models were selected from the literature and compared with experimental data to observe which model is best fit (Agbede et al., 2020).

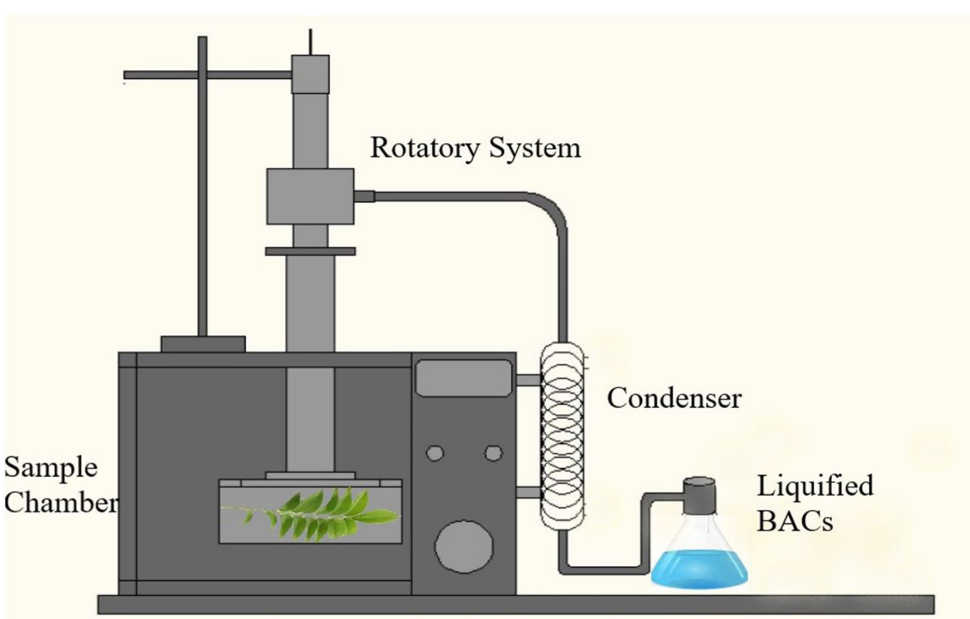

Figure 1. Experimental setup for Microwave based drying of fenugreek leaves.

Midilli Model $Moisture\ Ratio = \alpha * e^{-\kappa t^{n}} + \beta t$ (3)

Two term Model $Moisture\ Ratio = \alpha * e^{-\kappa_0 t} + \beta * e^{-\kappa_1 t}$ (4)

Page Model $Moisture\ Ratio = e^{-\kappa t^{n}}$ (5)

Two term exponential Model $Moisture\ Ratio = \alpha * e^{-\kappa t} + (1-\alpha) * e^{-\kappa \alpha t}$ (6)

Diffusion approximation Model $Moisture\ Ratio = \alpha * e^{-\kappa t} + (1-\alpha) * e^{-\kappa \beta t}$ (7)

Verma Model $Moisture\ Ratio = \alpha * e^{-\kappa t} + (1-\alpha) * e^{-gt}$ (8)

Moreover, diffusion coefficient was determined by using Fick's law (Mahjoorian et al., 2016). For microwave based drying process, Fick's law can be transcribed as below:

$$Moisture\ Ration = \frac{M_t - M_e}{M_o - M_e} = \frac{8}{\pi^2} \exp\left(\frac{-\pi^2 D}{4L^2}\right) \tag{9}$$

where, D is the diffusion coefficient (m^2/s) in the sample having thickness L in meters. Here, the value of D was obtained by slope of a graph which was plotted between the values of ln MR and time (minutes). The slop of this graph (α) is described as:

$$\alpha = \frac{\pi^2 D}{4L^2} \tag{10}$$

Furthermore, influence of various variables (time, power and moisture content) on the values of drying rate and moisture ratio were determined by applying box-Behnken design. The box-Behnken design was elaborated by quadratic equation by using JMP software (SAS Institute, USA).

2.5 Specific energy of MADE

Specific energy consumption of MADE process of fenugreek leaves at various powers can be determined by the following equation (Darvishi et al., 2013; Jahanbakhshi et al., 2020) and expressed in MJ/kg (Equation 11):

$$E_{Specific} = \frac{3.6 \times E_{Microwave}}{(M_o - M_t) \times M_s} \tag{11}$$

where, M_o and M_t are initial and final moisture contents of fenugreek leaves and M_s is the dry matter in kilograms. $E_{microwave}$ was calculated by Equation 12:

$$E_{microwave} = t \times p \tag{12}$$

where, t and p are the time in seconds and power in watts, respectively (Alvi et al., 2019; Khan et al., 2016).

2.6 Determination of DPPH activity

The antioxidant activities (DPPH activity) of the samples were determined by the method described in literature (Barkat et al., 2018; Curi et al., 2019). In this method, sample (0.5 mL) material in the form of extract obtained from Microwave and methanol extraction method added in methanol-based DPPH (4% w/v) solution; subsequently vigorous shaking of the mixture was carried out. Afterwards, the mixture was placed in darkness (overnight) and absorbance was measured at 515 nm using a spectrophotometer (IRMECO, Germany). The sample results of DPPH activity were reported in terms of percentage (%).

2.7 Total phenolic and flavonoid contents

Total phenolic compounds (TPC) were determined according to the method of Sun et al. (2005) with minor modifications (Aydar, 2020; Lu et al., 2011). In this method, Folin-Ciocalteu reagent was diluted with distilled water to prepare ten-time dilutions. Afterwards, a small portion of this solution (0.75 mL) was mixed with extracted sample (0.1 mL) and incubated for ten minutes. Subsequently, sodium carbonate solution (0.75 mL) was mixed with solution and placed in the dark conditions for forty-five minutes. Finally, absorbance reading of sample was measured at 765 nm with spectrophotometer (IRMECO, Germany). Besides, a calibration curve of absorbance was sketched against the different concentrations of gallic acid (0, 5, 10, 15, 20, 30 and 40 mg/L). The sample values were calculated and reported as gallic acid equivalent (mg/g).

For total flavonoid contents (TFC) determination, 1 mL of extract was mixed in 3 mL solution of methanol containing aluminum chloride and potassium acetate. This solution was further diluted by adding 5.6 mL of distilled water and placed at room temperature for half an hour. Afterwards, sample absorbance was determined with spectrophotometer (IRMECO, Germany) at 415 nm wavelength. The standard curve of quercetin was taken as reference and sample values were expressed in terms of quercetin equivalents (Ghafar et al., 2017).

2.8 Statistical analysis

All the experiments were performed in triplicates and their means along with standard errors were reported. The *JMP*[R] (SAS Institute, USA) was used to perform analysis of variance and Tukey's test that determine the level of significance and comparison between treatments. Moreover, fitness of the models was determined by the values of R^2, RMSE, SSE, RPD and Chi square (Guiné, 2018; Mota et al., 2010; Roberts et al., 2008; Equation 13-16).

$$Root\ Mean\ Square\ Error\,(RMSE) = \sqrt{\frac{1}{N}\sum_{i=1}^{n}\left(V_{\exp,i} - V_{\mathrm{mod}el,i}\right)^2} \tag{13}$$

$$Sum\ of\ Square\ Error\,(SSE) = \frac{1}{N}\sum_{i=1}^{n}\left(V_{\exp,i} - V_{\mathrm{mod}el,i}\right)^2 \tag{14}$$

$$Chi\ Square\,(CS) = \frac{1}{N - n_p}\sum_{i=1}^{n}\left(V_{\exp,i} - V_{\mathrm{mod}el,i}\right)^2 \tag{15}$$

$$\mathrm{Re}lative\ Percent\ Deviation\,(RPD) = \frac{100}{N}\sum_{i=1}^{n}\frac{\left|V_{\exp,i} - V_{\mathrm{mod}el,i}\right|}{V_{\exp,i}} \tag{16}$$

3 Results and discussions

3.1 Drying rate

Drying rates of fenugreek leaves were calculated with the help of Equation 1 and results have been shown in Figure 2. It was

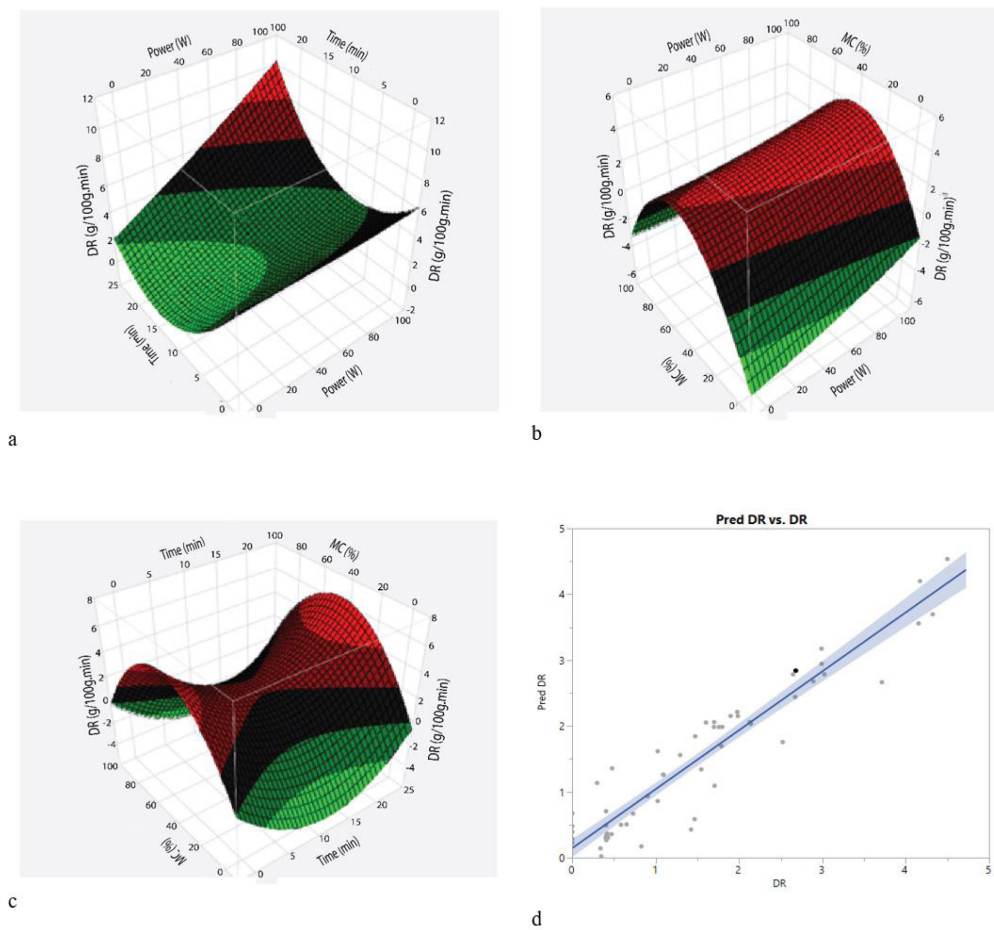

Figure 2. Response surface plots compare the drying rate of fenugreek leave influence by the interaction of time and power (a), power and moisture content (b), time and moisture content (c). While (d) represent the comparison of experimental and predicted value of drying rate.

evident that drying rate initially increased swiftly and reached up to 4.5 g/100 g.min within two minutes when heated at 100 W power (Figure 2a, b). At this point, maximum amount of moisture had been removed from the product surface. Afterward, value of drying rate decreased significantly indicating the reduction in moisture contents of the samples. Thus, drying rate of fenugreek leaves followed the parabolic trend in microwave based drying system (Figure 2b, c). Initially, the higher values of drying rate were due to higher moisture levels of leaves that absorbed more energy of microwaves; resulting in a prompt temperature rise. It stimulates the rapid evaporation process from the product surface (Figure 2b, c). After 50% moisture removal, less moisture level was present in sample for evaporation, compared to initial time; thereby reduced rate of drying (Soysal et al., 2006; Therdthai & Zhou, 2009; Wang et al., 2007). This lowering of the drying rate as a function of time was attributed to the reduced availability of free moisture as previously discussed in literature (Alvi et al., 2019; Khan et al., 2016, 2019). Besides, effect of microwave power on drying rate of leaves was observed. It was noted that the drying rate of leaves was increased from 1.79 to 4.56 ± 0.05 g/(100 g.min) when power was increased from 30 to 100 W. It indicated that increase in power had significantly increased the drying rate. Thereby, reducing the processing time; ultimately the processing cost.

Moreover, ANOVA was performed to assess the influence of independent variables on drying rate as linear, interaction and quadratic and residual coefficients. It was obvious that a significant effect of variables on drying rate (Table 1) was evident especially in terms of linear and quadratic effects. Besides, the authenticity of second order polynomial was evaluated through the values of coefficient of determination (R^2) and F-test. The resultant value for R^2 (0.98) is close to one and indicated the less variation in response model. Thus, this model may be used to explain current experimentation. The second order polynomial model equation obtained by response surface methodology analysis for drying rate is as follows (Equation 17):

$$DR = 3.167 + 0.638\,P - 1.160\,T - 0.065\,MC + 0.432\,P \times T - 0.429\,P \times MC + 0.076\,T \times MC + 0.082\,P^2 + 0.555\,T^2 - 4.371\,MC^2 \tag{17}$$

where, P, T and MC represent the power in watts, time in minutes and moisture content in percentage. These model values were compared with experimental ones and experimental drying rate was good fit with the predicted values (Figure 2d) as supported by the statistical values for predicted and actual drying rate (R^2=0.895, p<0.0001).

Table 1. Analysis of Variance for drying rate of fenugreek leaves.

	Source	DF	F Ratio	Prob > F	Remarks
	Model	9	53.7554		
Linear	Power	1	9.1391	0.0037	Significant
	Time	1	8.1236	0.0061	Significant
	MC	1	0.0200	0.8881	Non-Significant
Interaction	Power × Time	1	2.7962	0.1000	Non-Significant
	Power × MC	1	6.9177	0.0110	Significant
	Time × MC	1	0.0238	0.8779	Non-Significant
Quadratic	$Power^2$	1	0.3749	0.5428	Non-Significant
	$Time^2$	1	31.9122	<0.0001	Significant
	MC^2	1	130.3274	<0.0001	Significant
	Error	57			
	Total	66		<0.0001	Significant

DF indicates degree of freedom and F represents F-test

Table 2. Influence of microwave power on the effective diffusion and specific energy consumption during MADE processing of fenugreek leaves.

Power (W)	Effective Diffusivity (m^2/s)			Drying Time (min)	Specific Energy (MJ/kg)
	Average value (10^{-14})	First falling rate period (10^{-12})	Second falling rate period (10^{-12})		
30	0.74^{d}	0.67^{d}	0.15^{cd}	21 ± 1.5^{a}	2.39^{cd}
50	2.20^{c}	2.05^{c}	0.2^{cd}	13 ± 1.25^{b}	2.47^{a}
80	3.22^{b}	5.49^{b}	0.46^{b}	8 ± 0.4^{c}	2.33^{cd}
100	7.67^{a}	8.13^{a}	1.64^{a}	5 ± 0.5^{d}	1.86^{b}

3.2 Drying time and moisture ratio

During MADE processing, time and power levels were evaluated. It is evident from the results that maximum time (21 min) was required for drying at power 30 W of microwave (Table 2). By increasing the power, drying time was reduced significantly, and minimum time required for complete drying was 5 min at power 100 W. The difference in drying time may be attributed to variation in heating intensity among the different power levels (Coolong et al., 2008; Özdemir & Altan, 2011).

Moisture ratio (MR) of fenugreek was calculated according to Equation 2 and results are shown in Figure 3. The value of MR at different powers of microwave (30, 50, 80, & 100 W) were compared (Figure 3) and results indicated that MR rapidly decreased at 100 W. This rapid decrease in MR indicates the swift drying of leaves consequently reducing the processing time. Furthermore, the experimental moisture ratio is in good agreement with predicted moisture ratio (<0.0001) with good authenticity (R^2=0.997) of predicted values (Figure 3d).

Additionally, MR was predicted by various model equations (Equations 3-8) and compared with experimental values. According to statistical analysis, R^2 value was highest for Midilli model (0.9988) followed by two term model (0.9958), which indicates the good fit of the model (Table 3). However, higher values of RMSE (0.739) and RPD (20.25) values did not favour the model fitness with experimental values. Thus, two term model (Equation 4) was selected which had lower values of RMSE (0.018) and RPD (0.167) compared to Midilli. This model was further used to predict MR values and calculate the values of effective diffusion during the drying process of fenugreek leaves.

3.3 Diffusivity coefficient

The diffusive coefficient was calculated by using Equation 9 and 10. The results obtained had shown that diffusion process was controlled by the falling rate period. In this study, two phases of falling rates were observed that occurred in a constant slope. This slope determined the values of effective diffusion individually. The cut off value between these two falling rate periods was 0.3 of moisture ration. The effective diffusivities in the first falling rate period were influenced by the microwave powers and the range of values were 0.67 to 8.13 (10^{-12} m^2/s). Likewise, the values of effective diffusion in the second phase were influenced by power and values varied from 0.15 to 1.64 (10^{-12} m^2/s). The values of second period were about eight times smaller than first falling rate period (Table 2).

The average values of effective diffusion of fenugreek leaves during drying process at 30-100 W varied in the range of 0.73 to 7.67 (10^{-14} m^2/s) (Table 2). The diffusion coefficient increased with the increase of microwave power and results were in agreement with the research reported in literature (Alvi et al., 2019; Mahjoorian et al., 2016).

3.4 Specific energy consumption

The energy consumption of microwave assisted drying and extraction method was calculated and shown in Table 2. It is evident from the results that 80, 50 and 30 W consumed more energy (2.33, 2.47 and 2.39 MJ/kg, respectively) compared to 100 W (1.8672 MJ/kg). This may be attributed to lowest processing time (5 minutes) at 100 W processing compared to

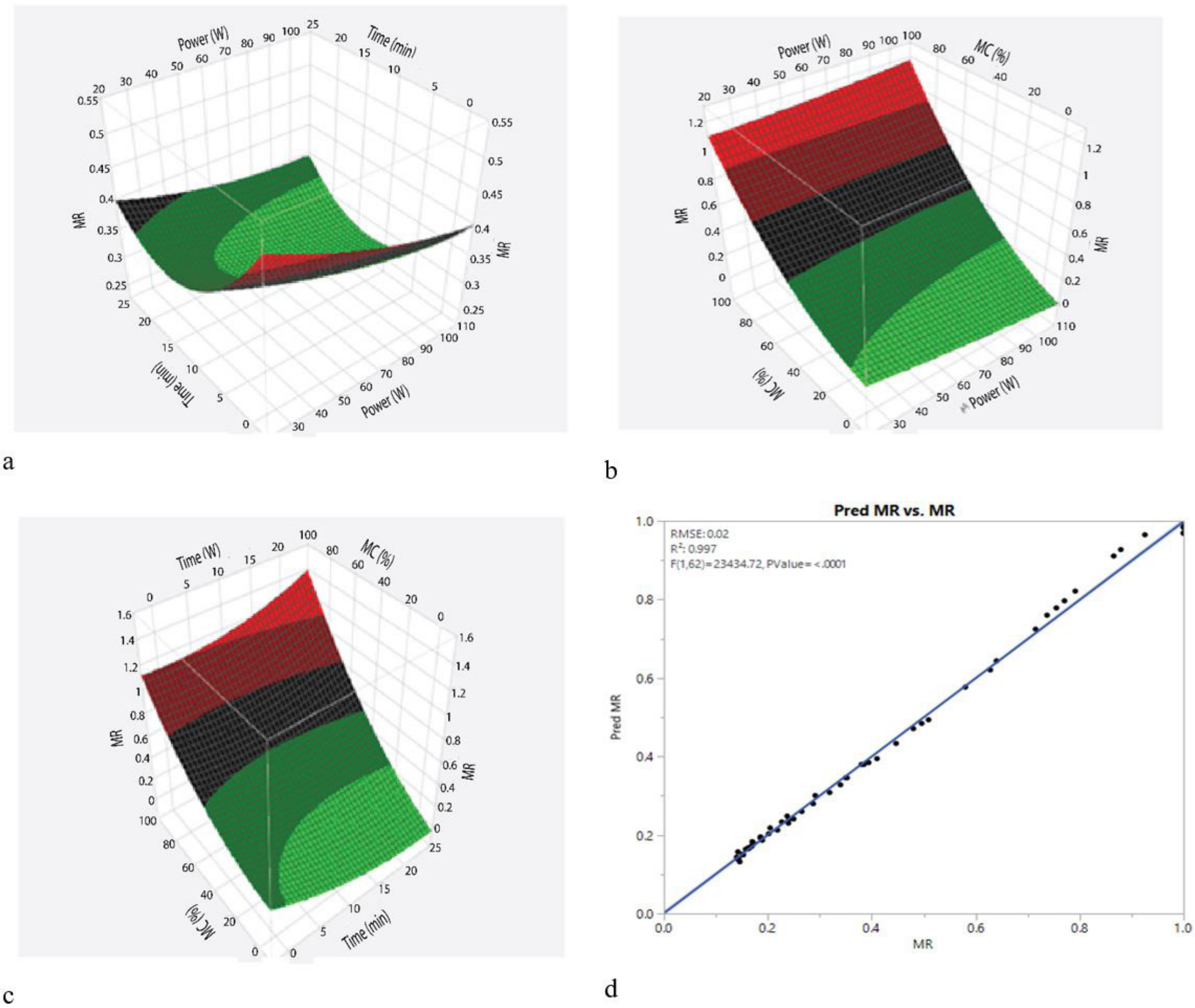

Figure 3. Response surface plots, moisture ratio as a function of time and power (a), moisture ratio as a function of power and moisture content (b), moisture ratio as function of time and moisture content (c), comparison of experimental and predicted moisture ratio (d).

Table 3. A statistical analysis of various thin layer drying models used in this study to predict the MR.

MODEL NAME	MODEL CONSTANT	R2	CHI ((^{2})	SSE	RMSE	RPD
MIDILLI	n = 1.244	0.9988	0.573	1.991	0.739	20.25
	κ = 0.066					
	α = 0.997					
	β = 0.005					
TWO TERM	α = 1.037	0.9958	3.242×10^{-4}	3.413×10^{-4}	0.018	0.167
	κ_0 = 0.102					
	β = $1.133e^{-4}$					
	κ_1 = -0.265					
PAGE	n = 1.061	0.9947	0.29	4.335×10^{-4}	0.525	194.95
	κ = 0.084					
TWO TERM EXPONENTIAL	α = 1.476	0.9941	4.621×10^{-4}	4.865×10^{-4}	0.021	1.792
	κ = 0.114					
DIFFUSION APPROXIMATION	α = 38.873	0.9935	5.039×10^{-4}	5.304×10^{-4}	0.022	0.877
	κ = 0.079					
	β = 0.995					
Verma	α = 0.504	0.9934	5.142×10^{-4}	5.412×10^{-4}	0.022	0.425
	κ = 0.096					
	g = 0.096					

Table 4. A comparison of TPC, TFC and DPPH activity of MADE and conventional extraction methods.

EXTRACTION METHOD	DPPH	TPC	TFC
MADE	8.04 ± 1.49[a] %	16.87 ± 0.37[a] (mg GAE/mL)	21.07 ± 0.44[a] (mg QE/mL)
CONVENTIONAL	3.04 ± 1.80[b] %	14.01 ± 0.08[b] (mg GAE/g)	18.45 ± 0.49[b] (mg QE/g)

DPPH, TPC, and TFC represents 2-2-Di-Phenyle-2-Picryl Hydrazyl activity, Total Phenolic Contents and Total Flavonoid Contents, respectively.

others (21 minutes @ 30 W); longer the processing time, higher the energy consumption. Thus, heating the food products at 100 W is sustainable in terms of time, cost and energy utilization.

3.5 DPPH, total phenolic and flavonoid contents

DPPH, TPC and TFC contents of MADE extract were compared with methanol-based extraction method (Table 4). The results indicated that liquid sample, obtained through MADE processing, exhibited better values of TFC, TPC and DPPH activity compared to methanol-based extraction method. These findings were comparable to the literature values (Cheng et al., 2013; Khan et al., 2016). Moreover, higher values of TPC, TFC and DPPH activity in comparison with methanol-based extraction method exhibited that the proposed technique (MADE) did not affect the activity of bioactive compounds of extract and this claim was supported by the findings of (Ferreira et al., 2018) who reported that microwave drying does not affect the bioactive compounds (BACs) values of the product.

4 Conclusion

In this study, MADE technique was evaluated for drying of fenugreek leave as well as the extraction of BACs from these leaves. The drying time of MADE process was significantly reduced (75%) at highest processing power (100 W) and it consumed less energy compared to other microwave powers. Likewise, the drying rate values were found to be very high (4.56 ± 0.05 g/100 g.min) at 100 W that reduced the moisture content rapidly and ease in swift drying. Similarly, the MR values decreased quickly at 100 W compared to other powers and lowest energy (1.8672 MJ/kg) was consumed. Thus, 100 W for 5 min considered to be the optimum for drying of fenugreek leaves. Moreover, BACs extracted through MADE technique exhibited better TPC, TFC, and DPPH values compared to methanol-based extraction process. Based on these findings, it can be concluded that microwave based drying and extraction is an efficient and sustainable process compared to both traditional methods of drying and extraction.

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