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Mathematical Modelling and Optimization of Quality Parameters for Extraction of Turmeric Oil Using Microwave Assisted Extraction Technology

Sachin Ghanghas, Vijay Kumar Singh,
Mukesh Kumar Garg, Parveen Dhanger

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ABSTRACT

In the present work, optimization of quality parameters was performed for maximum recovery of turmeric oil from the dried rhizome of turmeric (*Curcuma longa* L.) using microwave assisted extracted technology. Petroleum ether and Hexane solvents were used for extraction of essential oil at two different input parameters of microwave i.e., microwave power (200, 250, 300, 350 and 400 watt) and extraction time (10, 15, 20, 25 and 30 min). Resulted response were estimated by the ANOVA using central composite rotable design (CCRD) of response surface methodology (RSM) and the polynomial equation of the second order response surface were modelled using multiple regression analysis. The quality of extracted oil was evaluated based on quality parameters. Maximum yield of 4.97 % of oil was recovered with the chemical properties value of acid, iodine and saponification were 4.5%, 39.37% and 14.86 % respectively using Hexane solvent at microwave power of 300-watt and 20 minutes of extraction time. Determination coefficients (R^2) were equal to 90% or higher for individual responses stated that the developed models were well fitted to the experimental results.

Keywords Turmeric oil, Microwave power, Extraction time, Acid value, Iodine value, Saponification value.

INTRODUCTION

India is the major producer of turmeric (370.5 MT) which is about 80 % of the total production of the world with the covered area of 4.6 mha (Indiastat 2020). It got recognized all over the world because of its potentialities as a therapeutic herb due to its antioxidant, anti-migraine, inflammatory, antimicrobial, and anti-tumor properties (Hemmati *et al*. 2021). Turmeric oil has immense importance in industries and medical science and its biological activities are attributed to

Sachin Ghanghas[1]

Assistant Professor, School of Agriculture, Lovely Professional University, Phagwara, Punjab 144411, India

Vijay Kumar Singh[2]*

Associate Professor and Head, Department of Processing and Food Engineering, Mahamaya College of Agricultural Engineering and Technology, ANDUAT, Ayodhya, Uttar Pradesh 224229, India

Mukesh Kumar Garg[3]

Retd Professor and Head, Department of Processing and Food Engineering, COAE and T, CCS Haryana Agricultural University, Hisar, Haryana 125004, India

Parveen Dhanger[4]

Junior Research Fellow, Agriculture University, Jodhpur 342304, India

Email: vijurss@gmail.com
*Corresponding author

the phyto-constituents of oil. Its use in agri-food industries i.e., prevent post-harvest deterioration, use in fatty foods to inhibit chemical deteriorations, bio preservation of food stuffs and improve food shelf-life as well as in other industries such as pharmaceutical, cosmetic, perfumery, and nutritional purposes has recently risen tremendously (Ibáñez and Blázquez 2021, Sahoo *et al*. 2019).

Extraction process of the turmeric oil from its rhizome was obtained from several traditional extraction methods such as, soxhlet extraction, hydro distillation, steam distillation are used but these processes have some major drawbacks like unsaturated or ester compounds formation due to longer extraction times, low extraction yield and substantial thermal degradation of critical volatile compounds through thermal or hydrolytic effects (Priyanka and Khanam 2018). With the aim of avoiding these drawbacks and subsequently increasing the quality and quantity of the oil, microwave assisted extraction technology was used in the present study. Microwave-assisted extraction technology (MAET) in recent times got wider acceptance due to its lower extraction times and less solvent use (Pandey and Shrivastava 2018). Microwave radiation causing vibration of water and other polar molecules with an increase of temperature and evaporation of water that disrupts cells by internal pressure inside the oil gland cell wall and matrices which effectively leaches out the oil contained inside. In this way, the diffusion of target compounds is easier and faster, saving time and energy (Fiorini *et al.* 2020). It represents a more efficient technology for extraction of oils from the Zingibereaceace family, because it is able to reduce the extraction time to one fourth of the time required in hydro distillation, avoiding the formation of degradation products and obtaining the maximum yield (Ibáñez and Blázquez 2021). MAET method is a very safe, speedier, productive, efficient, and economically feasible technique for oil extraction (Sachin *et al.* 2020).

Response Surface Methodology (RSM) is an effective statistical analysis tool for optimizing multi-variable and their interaction by via experimental design (Arora *et al*. 2021). Several researchers have functioned RSM in their work and used it for optimizing the different process parameters in extraction of essential oil such as extraction of cannabidiol-enriched hemp essential oil (Fiorini *et al.* 2020), rosemary essential oil (Yeddes *et al*. 2020). To date, no effort has been made to study the optimized process variables for the extraction of turmeric essential oil using MAET. Therefore, the present work was undertaken to see the effect of process variables viz., microwave power (MP) and extraction time (ET) for the recovery of maximum oil with best quality of turmeric oil using RSM.

MATERIALS AND METHODS

Plant material

The oil was extracted from dried turmeric (*Curcuma longa* L.) rhizomes which was procured from the local market in Hisar, Haryana (29°10’16.8”N 75°43’05.7”E). Rhizomes were finely grounded to 60-80 mesh powder using a hammer mill (Li *et al*. 2011). Hexane and Petroleum ether solvents were used for the extraction process. A fixed ratio (1:1:1) sample was prepared using turmeric powder, solvent, and distilled water respectively.

Modified microwave assisted extraction technology

For the extraction of turmeric oil, microwave oven (Samsung GW71B, frequency 2450MH) was modified from its original design Fig.1. A 3 cm circular hole at the top center of the chamber of microwave was fabricated. Glass wares include round face flat bottom flask, condenser, a two-piece receiver connectors and adapters were fitted on microwave for modification to facilitate the oil extraction process. Adjustment of the height and position of oil receiver and flask done using connector. The vapors make its way from the flask to the tubular condenser supported by the cooling water recirculation (Fig. 1).

Turmeric oil extraction

A sample was prepared using the turmeric powder, solvent (Hexane or Petroleum ether) and distilled water proportionally in 100g, 100ml and 100ml, respectively (Ching *et al*. 2014). Funnel was used for water separation whereas solvent got separated

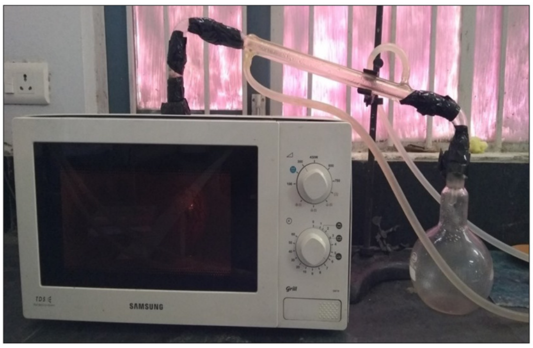

Fig. 1. Microwave assisted essential oil extraction unit.

via rotary evaporator instrument made by PERFIT, India. The research centrifuge (REMI R-23) was used for clarification of essential oil extracted from rotary evaporation. Suspended solid impurities were removed by sedimentation under the process of centrifugation at the speed of 4000 rpm for 20 min (Xiong *et al.* 2018).

Table 1. Process variables used in the central composite design for the two process parameters.

Process variables	Code	Variables codes				
		-2 (-α)	-1	0	1	+2 (+α)
Microwave power (Watts)	A	200	250	300	350	400
Extraction time (Minutes)	B	10	15	20	25	30

Experimental design

RSM was used to analysis the optimal condition for extraction process. For the extraction of turmeric oil, design expert application was used to carry out the experimental design and data analysis (Trial version 13, Stat-Ease, Inc. MN, USA). A five-level-two-factor central composite rotatable design (CCRD) was performed to analyze the combined significant effect of two process factors, microwave power and extraction time, denoted by the codes A and B, respectively (Table 1). The design comprised of 13 different combinations of four factorial points, four axial points, and five central points (replicates). To determine the optimal parameters of the microwave assisted extraction method for maximum oil recovery and the rich quality of oil, a two factor-five level CCRD was performed, followed by response analysis. The following parameters were chosen for optimization: Microwave power i.e., 200, 250, 300, 350, 400 W and

Table 2. Central composite rotatable design arrangement for variables A (MP), B (ET), and their response Y_1 (Turmeric oil (Hexane solvent)), Y_2 (Turmeric oil (Petroleum ether solvent)), Y_3 (Acid value), Y_4 (Iodine value), Y_5 (Saponification value).

Run	A	B	Y_1		Y_2		Y_3		Y_4		Y_5	
	(Watts)	(Minutes)	(%)		(%)		(%)		(%)		(%)	
			A*	P*	A*	P*	A*	P*	A*	P*	A*	P*
1	350 (+1)	25 (+1)	4.71	4.76	0.99	1.17	5.40	5.37	40.89	40.68	14.51	14.50
2	350 (+1)	15 (-1)	4.60	4.66	1.11	1.19	5.30	5.24	40.72	40.32	14.53	14.53
3	250 (-1)	15 (-1)	3.61	3.60	0.30	0.47	3.80	3.80	38.96	38.77	16.2	16.25
4	250 (-1)	25 (+1)	3.86	3.85	0.60	0.87	3.90	3.94	39.20	39.2	15.01	15.05
5	200 (-α)	20 (0)	1.90	1.92	0.18	0.05	3.50	3.47	38.72	38.71	16.59	16.56
6	300 (0)	10 (-α)	3.98	3.97	0.71	0.67	4.40	4.42	39.35	39.54	15.95	15.94
7	400 (+α)	20 (0)	3.93	3.89	1.11	1.07	6.30	6.34	41.53	41.74	14.27	14.29
8	300 (0)	30 (+α)	4.32	4.31	1.19	1.05	4.70	4.69	40.32	40.33	14.71	14.71
9	300 (0)	20 (0)	4.97	4.92	1.23	1.14	4.50	4.44	39.37	39.41	14.86	14.79
10	300 (0)	20 (0)	4.95	4.92	1.21	1.14	4.30	4.44	39.31	39.41	14.83	14.79
11	300 (0)	20 (0)	4.88	4.92	1.19	1.14	4.50	4.44	39.35	39.41	14.77	14.79
12	300 (0)	20 (0)	4.92	4.92	1.22	1.14	4.40	4.44	39.28	39.41	14.81	14.79
13	300 (0)	20 (0)	4.91	4.92	1.23	1.14	4.50	4.44	39.32	39.41	14.71	14.79

Coded values for A, B enclosed within brackets, A*: Actual value, P*: Predicted value, MP: Microwave power, ET: Extraction time.

extraction time i.e., 10, 15, 20, 25, 30 min applied to samples (viz., Hexane and Petroleum ether solvent) coded as -2, - 1, 0, +1 and +2, and experimental combination were presented in the Table 2, respectively.

Quantitative and qualitative analysis

Oil yield (%)

The turmeric oil was calculated as per methods to AOAC (2000). The oil yield (%) was calculated by using the following formulae:

$$\text{Oil yield (\%)} = \frac{\text{Total oil in given volume of extract, g}}{\text{Total amount of sample taken for extraction, g}}$$

Acid value (%)

Acid value of all oil samples were calculated according to AOAC (2000) methods using the following formula.

$$\frac{\text{56.1 X Volume of standard potassium hydroxide ml X Normality of the potassium hydroxide solution N}}{\text{Weight of the sample g}}$$

Iodine value (%)

The iodine value was measured as per the procedure used by Sayyad and Farahmandfar (2017) using the following formula.

$$\text{Iodine value} = \frac{12.69 \text{ X } (B - S) \text{ X } N}{W}$$

Where,
B = Volume of standard sodium thiosulphate solution used in blank, ml
S = Volume of standard sodium thiosulphate solution used in sample, ml
N = Normality of the standard sodium thiosulphate solution, N
W = Weight of the sample, g

Saponification value (%)

Saponification value of the all oil samples were calculated according to AOAC (2000) methods using the following formula:

$$\text{Saponification value} = \frac{56.1 \text{ X } (B - S) \text{ X } N}{W}$$

Where,
B = Volume of hydrochloric acid required for the blank, ml
S = Volume of hydrochloric acid required for the sample, ml
N = Normality of the standard hydrochloric acid, N
W = Weight of the oil/fat taken for the test, g

Multiple regression equation

General predictive equation derived for the investigations from the multivariate regression analysis implemented on design expert, where left hand side is the predicted value of the dependent variable Y (i.e., oil yield via hexane (Y_1), petroleum ether (Y_2), acid value (Y_3), iodine value (Y_4), saponification value (Y_5)). At right end side, X_i and X_j represents, A and B which are the main effects of the independent process variables i.e., microwave power and extraction time, X_iX_j as AB represents the interaction effects, A^2 and B^2 are the quadratic effects of the respective process variables. The symbol, i and j are the factors level with k being the factors assessed. β_0 is the constant term; βi, β_{ij} and β_{ii} are coefficients of the linear effect, interaction and quadratic regression coefficients, respectively.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} \beta_{ij} X_i X_j \quad (1)$$

The significant terms in the model (Eq. 1) were found by analysis of variance for every response and the F value for judgement of significance at the probability level of (< 5%). The desirability function gives the optimal values of the process conditions.

RESULTS AND DISCUSSION

Optimization of microwave assisted extraction technology

The effects of MAE on essential oil yield were examined using RSM in CCRD. The model terms ($p<0.05$) were considered significant (Lopresto *et al*. 2014). It was observed that quadratic factors (A^2, $p<0.05$) with two linear terms of microwave power (A, $p<0.05$) and extraction time (B, $p<0.05$) had a very significant influence on oil yield, although quadratic factor (B^2) and interaction factor (AB) were not significant ($p > 0.05$). Research results show that the suggested model accurately represented the experimental results. The results of the coefficient of determinations (R^2) reflects the correctness of the regression model with higher values indicating a stronger association between experimental observed and predicted values.

Table 3. Regression equation for turmeric oil extraction using hexane and petroleum ether with respective R_2 values.

Regression equation	R^2
$Y_1 = 4.72693 + 0.800667A + 0.310833B - 0.1815AB - 0.456629A^2 - 0.205129B^2$	0.83
$Y_2 = -9.81286 + 0.0493011A + 0.261685B - 0.000416AB - 0.000060A^2 - 0.00294629B^2$	0.87

Effect of solvents on oil extraction

Recovery of turmeric oil using Hexane and Petroleum ether solvents ranged from 1.90 to 4.97 and 0.180 to 1.22 % respectively at different combinations. Petroleum ether assisted oil yield was far less as compared to Hexane solvent. It could be because of complicated synthesis of fatty acids and glucose breakdown components and presence of OH group which creates interference in extraction. Such complexes cause trouble in extraction (Araromi *et al.*

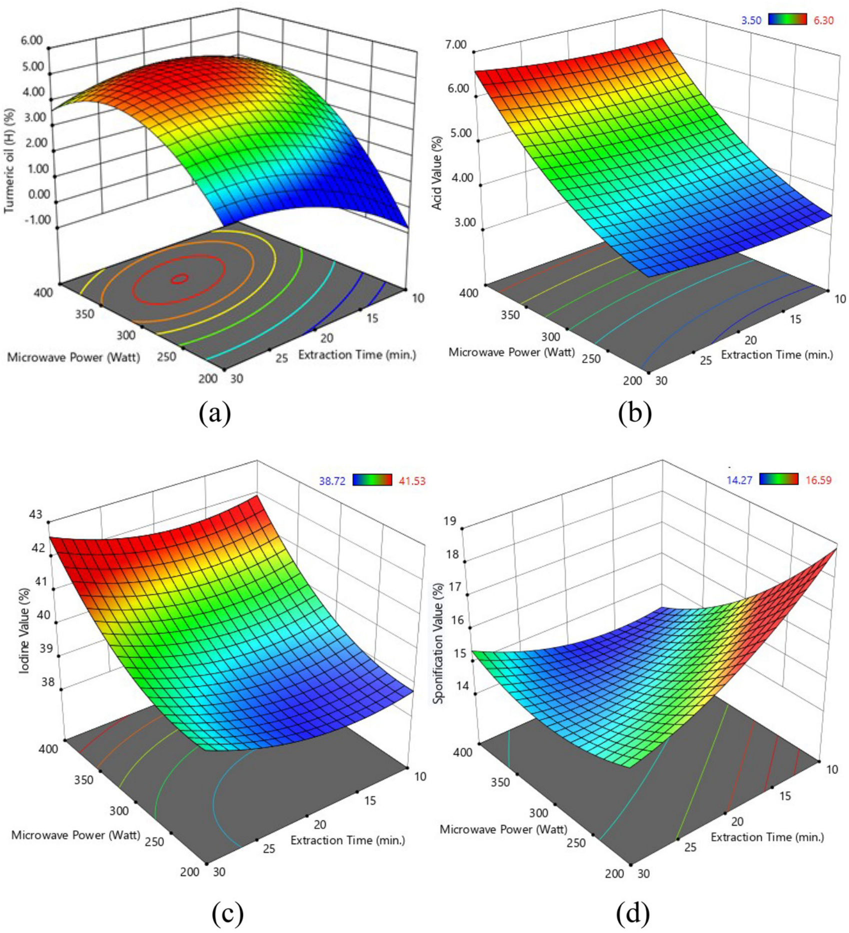

Fig. 2. 3D Response surface plots for turmeric oil via. Hexane solvent (a), acid value (b), iodine value (c) and saponification value (d) of turmeric oil as a function of process parameters.

2017, Wu *et al.* 2011). Hence, hexane as a solvent was considered for further quality assessment of turmeric oil. R^2 of quadratic models using hexane and petroleum ether as a solvent were 0.83 and 0.87 as shown in Table 3. Regression analysis showed that oil yield was significantly affected ($p < 0.05$) by microwave power and extraction time. Fig. 2 shows that with increase in process parameters, oil yield increases while microwave power has more significant effect than extraction time. Regression analysis equations for oil yield using hexane and petroleum ether solvent are presented in the Table 3, respectively.

Effect of process variables on acid value

Acid value of turmeric oil varied between 3.5 to 6.3%. The maximum value was found at the combination of 400 W for 20 minutes (Table 2). The degree of p-value indicated that both process parameters have significant effect but higher with microwave power ($p < 0.01$) compare to the extraction time ($p < 0.05$) both at linear and quadratic levels (Table 4). 3D response plot has shown a concave upward inclination as depicted by the positive quadratic coefficient of process parameters. Triglycerols found in oil are prone to microwave heating, particularly in the presence of water, which releases fatty acids from their ester linkage and increases free acidity. The acid value was tremendously affected by microwave power in comparison to extraction time. The R^2 value (0.99) indicate negligible 0.63 % non-explanatory variation while adequacy of fit F-value (221.74) signifies the model that was sufficiently near to fitting the model in the experimental results Table 4. The data was fitted using a quadratic polynomial model Eq. (2) as below:

$$Y_3 = 4.4448 + 0.7167A + 0.0667B - 7.3965 \times 10^{-18} AB + 0.1153A^2 + 0.0278B^2 \quad (2)$$

Intensive exposure of microwaves for small period of time brought up hydrolysis of fats and consequently hike in the acid value but with prolonged exposure, no significant effect observed rather degradation of oil has occurs (Kittiphoom and Sutasinee 2015, Sodeifian *et al.* 2019).

Table 4. Regression coefficients (β) of the fitted second order polynomial for dependent variables.

Factors	Turmeric oil (H) %	Turmeric oil (PE) %	Acid value %	Iodine value %	Saponification value %
		Quadratic model			
Intercept	4.915	1.1435	4.4448	39.4059	14.7890
A	0.4917**	0.2547**	0.7167**	0.7558**	-0.5675**
B	0.0867**	0.09517	0.0667*	0.1958*	-0.3075**
AB	-0.035	-0.1040	-7.40×10^{-18}	-0.0175	0.2925**
A^2	-0.5029**	-0.1465**	0.1153**	0.2047**	0.1581**
B^2	-0.1941**	-0.0707	0.0278	0.1322*	0.1331**
R^2	0.9984	0.8735	0.9937	0.9593	0.9969
Std Dev	0.0451	0.1728	0.0767	0.2257	0.0524
Lack of fit	Non-significant	Significant	Non-significant	Significant	Non-significant
R^2	0.9984	0.8714	0.9937	0.9593	0.9969
F value	871.14	9.49	221.74	33.03	445.04

**$p < 0.01$, *$p < 0.05$

A, B: Linear levels, AB: Interaction level, A^2, B^2: Quadratic levels.

Effect of process variables on iodine value

Iodine value indicates the unsaturation of oils and the stability of oil in food and industrial applications (Sayyad and Farahmandfar 2017). Iodine value of turmeric oil ranges from 38.72 to 41.53 %. The maximum value of iodine was found with the combination of 400 W for 20 minutes Table 2. Results were found in the agreement of Sachin (2018). The coefficient value for process parameters exhibited the significant effect on output with more impact of microwave power (β=0.7558, $p < 0.01$) comparing to the extraction time (β=0.1958, $p < 0.05$) both at linear and quadratic levels. Response surface plot revealed a concave upward inclination as depicted by the positive coefficients of process parameters at quadratic levels. The coefficient of determination R^2 value showed 95.93 % variability in the data, and model with F-value 33.03 signifies the fit in the research data (Table 4). The quadratic Eq. (3) for fitting data is as follows:

$$Y_4 = 39.4059 + 0.7558A + 0.1958B - 0.0175AB + 0.2047A^2 + 0.1322B^2 \quad (3)$$

Effect of process variables on saponification value

Saponification value (SV) for oil specifies the average chain length of total fatty acids existing in the oil (Wu *et al*. 2011). Saponification value varied between 14.27 to 16.59 %. Results were found in concurrence with Sachin (2018). Maximum value of saponification was obtained with same extraction time of 20 minutes but at 400 W and 200 W (Table 2). The coefficient value for process parameters exhibited the negative effect on the saponification value with little supplementary effect of microwave power (β= -0.5675, $p < 0.01$) comparing to the extraction time (β= -0.3075, $p < 0.01$) at linear and positively affecting at quadratic levels. Results revealed that SV decreased at higher value of microwave power for longer periods and similar results were found by Uoonlue and Muangrat (2019), Mohammadpour *et al*. (2019), Muangrat and Pongsirikul (2019).

3D response plot visualizes a concave downward curve which was the result of positive quadratic coefficient. The coefficient of determination R^2 valued showed 99.69 % variability in the data with F-value (445.04) signifies the model fit in the research data (Table 3). The experimental data were fitted using a quadratic model with Eq. (4) as follows:

$$Y_5 = 14.789 - 0.5675A - 0.3075B + 0.2925AB + 0.1581A^2 + 0.1331B^2 \quad (4)$$

Table 5. Numeric optimization criteria for process conditions along with predicted responses.

Name	Goal	Lower Limit	Upper Limit	Predicted Solution	Actual response value	Desirability
Process parameters						
A: MP	minimize	200	400	305.24	300	0.948
B: ET	minimize	10	30	21.26	20	
Dependent variables						
Y_1: TO yield	maximize	1.9	4.97	4.83	4.97^{NSD}	
Y_2: AV	is in range	3.5	6.3	4.54	4.5^{NSD}	
Y_3: IV	is in range	38.72	41.53	39.55	39.37^{NSD}	
Y_4: SV	is in range	14.27	16.59	14.67	14.86^{NSD}	

MP: Microwave power, ET: Extraction time, TO: Turmeric oil, AV: Acid value, IV: Iodine value, SV: Saponification value, NSD: Non-significant difference.

Model validation and numerical optimization

The process parameters were optimized numerically using statistical software Design Expert 13 software (Trial version) and were assigned to minimum goal during optimization. Also, the goals were allotted to each response parameters. The turmeric oil yield was set to maximize; acid value, iodine value and saponification value were set to be in the range. The numerical optimization suggests that the 305.24 W and 21.26 minutes resulted in an optimized output with desirability of 0.948 (Table 5). A high desirability score demonstrated that the process parameters were suitable for presenting desirable outcomes in terms of responses. Experimentation was performed out at the projected optimal conditions in order to validate the optimal parameters. The experiment findings were comparable to those reported via RSM and were confirmed by the t-test. The validated experimental values for oil yield, acid value, iodine value and saponification value (4.97, 4.5, 39.37 and 14.86 %) were found be not significantly different from predicted values. Thus, suitability of the models to predict various responses was ascertained.

CONCLUSION

The central composite rotatable design of RSM is evidenced throughout research work for being effective and accurate in the optimization of various process variables as well as critical responses for the quantitative and qualitative analysis of turmeric oil. Model approach and 3D surfaces for predicting the interaction effect demonstrated that all the models were able to accurately predict the response under consideration. The specified process parameters had a significant effect on the responses with high desirability function (0.948) which could be obtained by optimum combination of 300 W and 20 minutes for extraction of turmeric oil. The obtained response model would be beneficial to find optimum parameters for any other factor combination, and the process can be intensified for large scale production of turmeric oil of stable quality. Microwave-assisted extraction gives high points of interest to use this technique over the other conventional methods for oil extraction, i.e. less time consumption, low energy demand, and quality product.

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