



Efficacy of Microwave Heating Parameters on Physical Properties of Extracted Oil from Turmeric (*Curcuma longa* L.)

**Sachin¹, Vijay Kumar Singh^{1*}, M. K. Garg¹, Amarjit Kalra¹, Sushant Bhardwaj¹,
Parveen¹, Rajender Kumar¹, Yadvika¹, Sunil Kumar¹, Nitin Kumar¹,
Arun Kumar Attkan¹, Anil Panghal¹ and Deepak Kumar²**

¹College of Agricultural Engineering and Technology, CCSHAU, Hisar - 125004, India.

²Department of Chemical Engineering, SUNY College of Environmental Science and Forestry,
Syracuse, NY 13210, USA.

Authors' contributions

This original research papers work was carried out in collaboration of all authors. Author Sachin designed and executed the experiments and wrote the first draft of the manuscript, Author VKS wrote the protocol and supervised to conduct the experiments. Authors MKG, AK, Yadvika and DK supervised the study design, methodology. Authors SK, NK and AKA finalized the statistical tool and analyzed the data, Authors RK and AP general work arrangement, Authors SB and Parveen supervised the literature searches and result reporting. All authors read and approved the final manuscript.

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ABSTRACT

In the study, essential oil extraction from turmeric was carried out using modified microwave distillation system and rotary evaporator unit. In present study, effect of input parameters i.e. microwave power (200, 250, 300, 350 and 400 W) and extraction time (10, 15, 20, 25 and 30 min.) on physical properties (oil yield, specific gravity, refractive index and colour) of essential oil were studied. Fixed ratio samples (1:1:1) i.e. turmeric powder, solvent and distilled water was used during the experiment conducted. Essential oil yield using hexane varied from 1.895% to 4.973% while from 0.180% to 1.226% using petroleum ether solvents. Quality parameters varied i.e. specific gravity from 0.910 to 0.923, refractive index from 1.478 to 1.506, colour values for oil ranges L* from

*Corresponding author: E-mail: vijurss@gmail.com;

70.60 to 98.60, a^* from -25.60 to 15.1 and b^* from 82.1 to 88.5. Optimum values of process parameters for maximum oil recovery (4.973%) and best quality of oil (specific gravity: 0.915; refractive index: 1.485) was found at microwave power 300 W and extraction time 20 min.

Keywords: Microwave power; extraction time; oil yield; specific gravity; refractive index.

1. INTRODUCTION

Turmeric (*Curcuma longa* L.) is a perennial herb of Zingiberaceae family. To extract oil from turmeric root, various conventional extraction methods are used such as hydro-distillation, soxhlet extraction, microwave-assisted extraction and steam distillation [1,2]. Essential oil as food flavours, food preservatives, pharmaceuticals and medicine has been used for many years [3]. The oil from turmeric contains many important constituents having varied properties like anti-microbial, anti-inflammatory, anti-dermatosis and insect repellent and also used in various digestive ailments [4,5]. The essential oil of turmeric has been reported about 84 chemical constituents. The essential oil of turmeric has turmerone { α (14), α -(13) and β (15)}, 1, 8-cineole (6), α -phellandrene (7), β -caryophyllene (16) and β -sesquiphellandrene (17) has major shares of essential oil [6,7]. Microwave assisted extraction (MAE) of fats and oils have recently gained popularity due to its reduced extraction time, energy and solvent consumption [8]. There are various methods of essential oil extraction from natural plants such as conventional hydro-distillation, steam distillation and solvent extraction. Major drawbacks of the above methods are degradation of some important volatile compounds due to long extraction times and unsaturated or esteric compounds degradation through thermal and hydrolytic effects [9]. Microwaves interact selectively with the free water molecules present, leads to localized heating. This energy is transmitted as waves which can enter in biomaterials and associated with polar particles into materials such as water to create heat and pressure. This pressure pushes and extends the cell wall and at long last annihilates it, which encourages the filtering out of phyto-constituents from the broken cells and in this way moving forward the extracted oil yield [10]. Nowadays microwave-assisted extraction (MAE) method has been found as an efficient and effective tool for the essential oil extraction [11,12]. It has various advantages i.e. quick energy transfer, effective heating, short time extraction, high yield extraction, organic solvent-free, high recovery of bio-active compounds and low operating costs [13]. MAE has already been utilized for essential

oil extraction from different aromatic plants in food processing and pharmaceutical industries with great potential for future applications [14]. Microwave assisted extraction method is a safer, faster, highly efficient and cost-saving alternative [15]. Thus it can be inferred that it is a potential process for extraction of oil from turmeric.

In present study, essential oil yield from turmeric using microwave assisted extraction and its quality were analyzed. Moreover, the effects of extraction time and microwave power on the essential oil yield and physical parameters of turmeric under microwave assisted extraction were investigated using response surface methodology (RSM). The methodology consists of three steps including experimental design using well-established statistical experimental designs such as the central composite rotatable design (CCRD), regression analysis based response surface modelling and process optimization through the response surface models. RSM provides a quicker and more economical method for gathering research results in comparison with other methods (such as one factor-at-a time and full-factors experimentation) [16]. The main objective of research is to develop a process for microwave assisted extraction of essential oil and to analyze effect of process parameters on oil yield and quality parameters (i.e. specific gravity, refractive index, color). Microwave-assisted extraction has proven to impart significant effects on mass transfer and offers high throughput and extraction efficiency. A microwave power of 275 to 1,000 W and a temperature range of 30 to 60°C are noticed in the different studies. The review presents a comprehensive account of the modern extraction techniques, the parameters responsible for yield and quality, and their industrial applications. Besides, the review highlights the optimized parameters for oil extraction from different oil-bearing materials [17].

2. MATERIALS AND METHODS

2.1 Sample and Reagents

The dried turmeric rhizomes were procured and fine grounded to 60-80 mesh powder [18]

using a hammer mill. Hexane and petroleum ether solvents used for extraction of essential oil. Fixed ratio sample was prepared using turmeric powder, solvent (Hexane or Petroleum ether) and distilled water in the proportion of 100 g, 100 ml and 100 ml respectively [19].

2.2 Microwave Assisted Extraction Design

The microwave oven used in this study is Samsung GW718 with the operating frequency of 2450 MHz, which is suitable for oil extraction. A 3cm hole was made centering on the top of microwave chamber. Modifications were done to microwave system, glass wares (i.e. round face flat bottom flask, condenser, receiver round face flat bottom flask, two piece receiver adapters and connectors) were engaged to facilitate the oil extraction process. The round face flat bottom flask placed at center on pater dish plate inside the microwave distillation unit and connected to receiver adapter through connectors. Receiver adapter connects condenser unit, another one at other end to oil receiver flask as shown in Fig. 1.

2.3 Extraction of Essential Oil

Different combinations of operational parameters applied to fixed samples of turmeric to extract heterogeneous solution of essential oil, solvent, water and solid impurities. Water get separated using separating funnel, solvent get separated using rotary evaporator instrument (PERFIT

India) and impurities through Research centrifuge (REMI R-23) as shown in Fig. 2.

2.4 Experimental Design

Design of the experiments was conducted for optimizing the selected process parameters using central composite rotatable design (CCRD) method. A two factor-five level CCRD followed by response analysis was carried out to find the optimum conditions of the microwave assisted extraction process for maximum oil yield recovery and also the best quality of essential oil. The key parameters selected for optimization purpose were: microwave power and extraction time with five levels of microwave power i.e. 200, 250, 300, 350, 400 W and five levels of extraction time i.e. 10, 15, 20, 25, 30 min which were applied to fixed samples (viz., Hexane and Petroleum ether solvent) were selected for codes of - 1.414, - 1, 0, + 1 and + 1.414, and experimental combination with actual and coded values were given respectively (Table 1 and Table 2).

2.5 Physical Analysis of Essential Oil

2.5.1 Oil yield (%)

The extracted essential oil yield (g/100 g) was calculated for each experimental run as follows:

$$\text{Yield(\%)} = \frac{\text{Mass of extracted essential oil}}{\text{Initial dried mass of turmeric}} \times 100$$



Fig. 1. Microwave assisted essential oil extraction unit

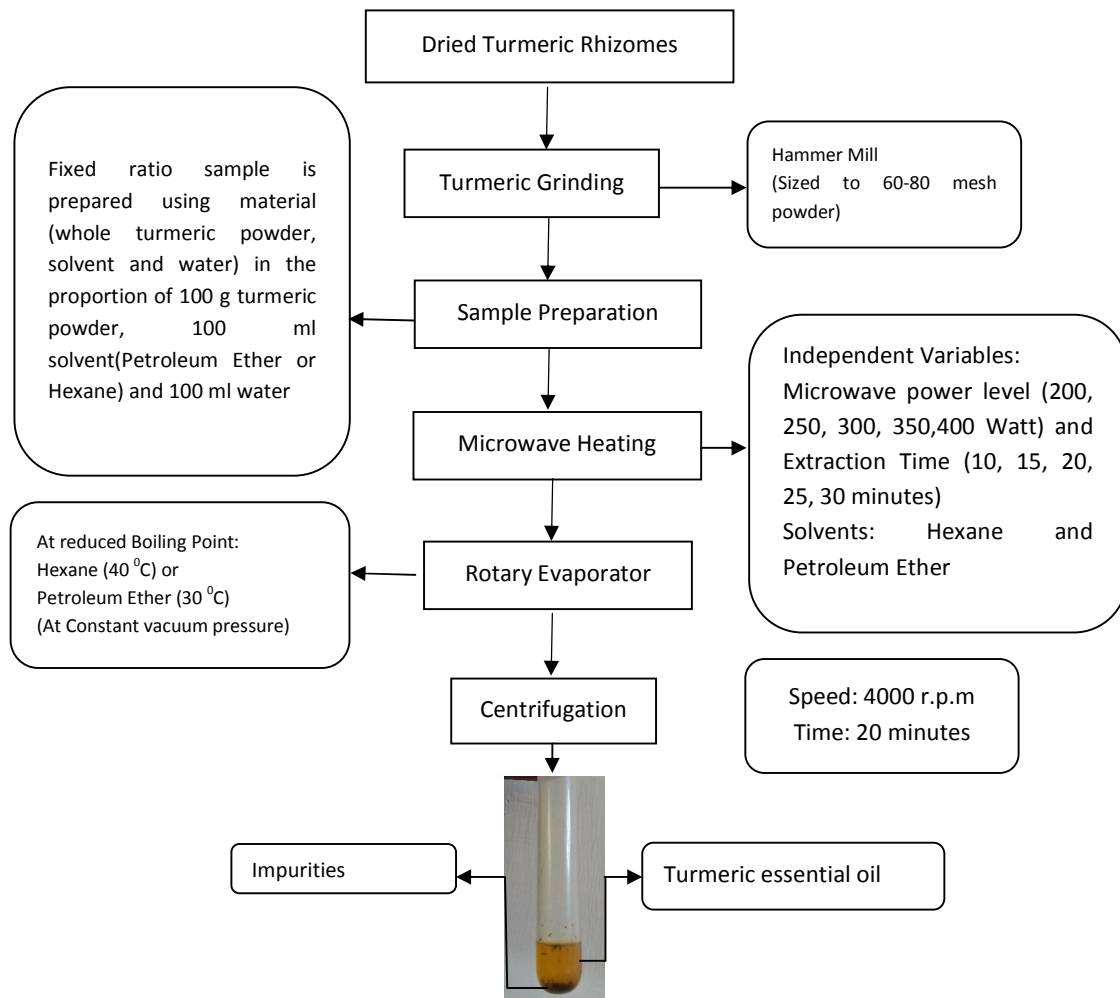


Fig. 2. Process flow for extraction of microwave assisted extraction of essential oil

2.5.2 Specific gravity

Specific gravity was determined according to rules of A.O.A.C, 2000 using pycnometer method.

2.5.3 Refractive index

Refractive index was determined using Abbe's refractometer (Accumax, India) method used [20].

2.5.4 Color

Color of fresh turmeric essential oil was determined by Color meter (ColorTec-PCM by

Accuracy Microsensors, Inc. Pittsford, New York).

2.6 Statistical Analysis

Statistical analysis of the experimental database was performed using software Design Expert (version 11.0). The optimum condition for essential oil extraction from turmeric was determined utilizing analysis of variance (ANOVA) to find the effect of each parameter on the different responses. The significance of each term in the second order polynomial Type III – Partial model (quadratic and interactions) was tested by the associated ANOVA in the central composite rotatable design and also the accuracy of the regression model.

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

Process variables	Code	Variables codes				
		-1.414	-1	0	+1	+1.414
Microwave power(Watt)	A	200	250	300	350	400
Extraction Time(Minute)	B	10	15	20	25	30

Table 2. Response surface experimental design in terms of coded levels and actual levels

Run	Coded values		Actual values	
	A	B	Microwave power(Watt)	Extraction Time(Minute)
1	+1	+1	350	25
2	+1	-1	350	15
3	-1	-1	250	15
4	-1	+1	250	25
5	-1.414	0	200	20
6	0	-1.414	300	10
7	+1.414	0	400	20
8	0	+1.41	300	30
9	0	0	300	20
10	0	0	300	20
11	0	0	300	20
12	0	0	300	20
13	0	0	300	20

3. RESULTS AND DISCUSSION

3.1 Optimization of Extraction Process

Based on two parameters i.e. microwave power and extraction time, effects of microwave assisted extraction on essential oil yield was evaluated by central composite rotatable design (CCRD) using response surface methodology. Model terms ($P < 0.05$) were considered as significant, rest were non-significant (Lopresto et al. 2014). It was analysed that two linear terms of microwave power (A, $P < 0.05$) and extraction time (B, $P < 0.05$) and quadratic terms (A^2 , $P < 0.05$) had a significant effect on the essential oil yield while quadratic term (B^2) and interaction term (AB) were not significant ($P > 0.05$).

Non-significant results indicate that the proposed model not fit the experimental data well. The values of the coefficient of determinations (R^2), predicted R^2 (Pred. R^2) and adjusted R^2 (Adj. R^2) shows the response model accuracy and their higher values indicates the higher correlation between experimental data and predicted values. Term with a lower P value and a higher F-value has more effect on extracted oil yield. The higher F-value of linear terms measured than other quadratic and interaction terms proposed effectiveness of these factors on the yield of essential oil. Among all significant terms, the linear term of microwave power (A) and quadratic

term (A^2) showed the largest effect on the extracted oil yield. Interactions between the different variables were found as a significant with R^2 (Hexane and Petroleum ether) i.e. 0.8352 and 0.8714. Adequacy of fit of presented model produced 'F' values 7.09 and 9.49, which are clearly significant.

3.2 Effect of Solvents on Extraction of Oil Yield

The second order mathematical model obtained from regression analysis for oil yield of turmeric oil (viz. Hexane and Petroleum ether solvent) was developed as follows:

$$Y (\text{Hexane solvent}) = 4.72693 + 0.800667A + 0.310833B - 0.1815AB - 0.456629A^2 - 0.205129B^2 \quad (R^2=0.8352) \quad (1)$$

$$Y (\text{Pet. Ether}) = -9.81286 + 0.0493011A + 0.261685B - 0.000416AB - 0.000060A^2 - 0.00294629B^2 \quad (R^2=0.8714) \quad (2)$$

Equation 1 and 2 indicates importance of microwave power in extraction of oil yield while extraction time shows the minimal impact on essential oil yield. The linear effects of operational parameters are significant which indicates their positive effect on essential oil yield.

Table 3. Table for data of different operational and process parameters

Experimental runs	Microwave Power (Watt)	Extraction Time (Minutes)	Oil Yield (%), Hexane	Oil Yield (%), Petroleum ether	Specific Gravity	Refractive Index	Color		
							L*	a*	b*
1	350	25	4.577	0.989	0.916	1.478	88.5	14.1	82.4
2	350	15	4.501	1.105	0.919	1.48	88.1	15.1	82.9
3	250	15	1.960	0.301	0.913	1.492	71.2	13.5	84.3
4	250	25	2.762	0.601	0.912	1.489	97.4	-23.8	83.7
5	200	20	1.895	0.180	0.91	1.506	98.1	-24.4	88.5
6	300	10	3.501	0.710	0.914	1.498	98.6	-21.8	86.6
7	400	20	4.521	1.112	0.923	1.478	81.3	14.2	82.8
8	300	30	4.927	1.189	0.914	1.481	71.3	12.8	82.6
9	300	20	4.973	1.226	0.915	1.485	71.2	13.7	85.4
10	300	20	4.973	1.226	0.915	1.485	71.2	13.7	85.4
11	300	20	4.973	1.226	0.915	1.485	71.2	13.7	85.4
12	300	20	4.973	1.226	0.915	1.485	71.2	13.7	85.4
13	300	20	4.973	1.226	0.915	1.485	71.2	13.7	85.4

Table 4. The standard error, mean, coefficient of variation, coefficient of determination, adjusted and Pred R-Squared and adequate precision values for developed models

Particulars	Model 1	Model 2	Model 3	Model 4
SD	0.6261	0.1758	0.0008	0.0028
Mean	4.12	0.9475	0.9151	1.49
C.V. %	15.21	18.56	0.0847	0.1887
R ²	0.8352	0.8714	0.9652	0.9291
Adj. R ²	0.7175	0.7796	0.9403	0.8784
Pred. R ²	-0.2343	0.0722	0.6467	0.3944
Adeq precision	8.6898	9.6089	22.7770	14.0426
F-Value	7.09	9.49	38.81	18.33
P-value	0.0115	0.0051	< 0.0001	0.0007

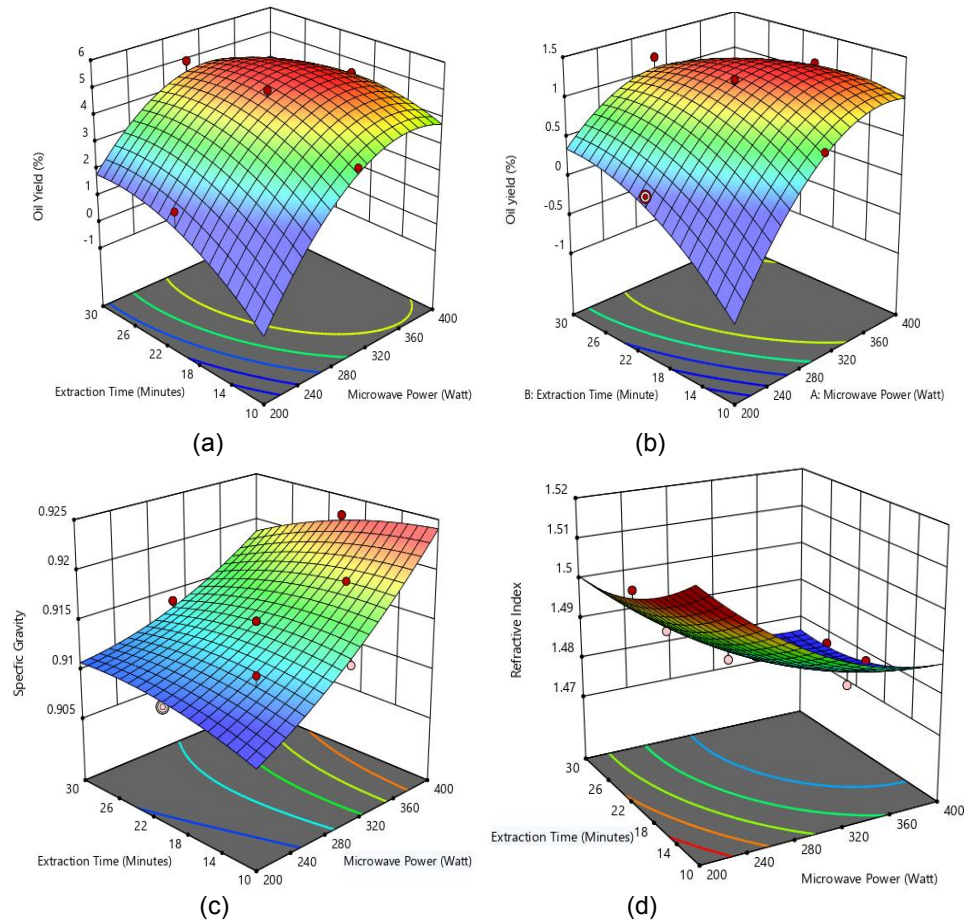


Fig. 3. Effect of microwave power and extraction time on oil yield using hexane solvent (a), oil yield using petroleum ether solvent (b), Specific gravity of oil (c) and Refractive index of oil (d)

Table 3, shows that the oil yield increased from 1.895 to 4.973% and 0.180 to 1.226% with increase operational parameters. Similar observations were found by the various researchers i.e. [21] for turmeric rhizomes, [22] reported the turmeric cultivars contained 3.0 to 5.0% volatile oil and [23] found 0.8 to 4.4 per cent. Non-polar solvents have both sides charged and are able to penetrate into the matrix of turmeric (lack an O-H end) and get trapped with oil and heated up by surrounding water which gave sufficient temperature to reach boiling point of solvent and get vaporized [24]. Essential oil yield using petroleum ether was very less than the yield obtained using hexane as solvent. It might be due to complex formation between fatty acids and carbohydrate breakdown components. Such complexes will certainly cause trouble in plant oil extraction with petroleum ether, similarly reported by [25,26].

3.3 Effect of Independent Parameters on Oil Yield

It was observed that time required for extraction of oil yield significantly ($P < 0.05$) decreased from 30 to 10 min with increase in power from 200 W to 400 W applied. It might be due more assimilation of microwave energy into water inside the plant matrix with increased microwave power and extraction time which ruptures the cells and bring cellular material (essential oil) to get bound with surrounding solvent and took less time to reach boiling point of solvent. Optimum condition for microwave assisted extraction was 300 W for 20 min brought the highest 4.973% oil yield. Interestingly, the increased extraction time higher than 70 min for 200 W and 40 min for 400 W, the oil samples were burned.

Response surface plot of essential oil yield as a function of operational parameters i.e. microwave

power and extraction time (Figs. 3a and 3b) shows essential oil yield significantly affected by increasing until 300 W for 20 min. Variation in the oil yield was due to rise of the microwave power which leads to vibration of water molecules in shorter time period. While further increase in process parameters, hexane get vaporized before partially penetrating cell matrix of turmeric, thus reduce in yield. Thus microwave power presents both positive and negative effects on essential oil yield while with increasing extraction time the essential oil yield less affected. The results were found similar with the observations of the [27] for *Annona squamosa*, [28] for *Abelmoschus esculentus*, [29] for grape seeds and similar effect was found by [30] for mango seed kernel oil. There may be another reason that with the quick variation of temperature due to high microwave power causes partial thermal degradation of essential oil and affects its oil yield [31,32]

3.4 Effect on Specific Gravity

$$\text{Specific gravity} = 0.914966 + 0.003A - 0.000333333B - 0.0005AB + 0.000372845A^2 - 0.000252155B^2 \quad (R^2 = 0.9652)$$

Response surface plot of specific gravity of essential oil as a function of operational parameters microwave power and extraction time (Fig. 3c) shows the specific gravity impressively affected until 400 W for 30 min. Specific gravity values increases from 0.910 to 0.923 with an increase in operational parameters. It may be due to incorporation of oxygen in unsaturated fatty acids present inside the essential oil which increases the proportion of saturated fatty acids (oxidized triglycerides) which contributes to increase in specific gravity. The interaction effects of microwave power and extraction time on the specific gravity are presented in Fig. 3c. Individual effect of process parameters (i.e. 200 to 400 W; 10 to 30 min. might be due to threshold energy requirement for fatty acids to get oxidized at given microwave power and required short extraction time and less effect for further increase in extraction time. Similar range was reported by [33].

3.5 Effect on Refractive Index

$$\text{Refractive index} = 1.48414 - 0.00658333A - 0.00325B + 0.00025AB + 0.00169612A^2 + 0.00107112B^2 \quad (R^2 = 0.9291)$$

Response surface graph of refractive index as a function of operational parameters microwave

power and extraction time (Fig. 3d) shows the refractive index significantly influenced until 400 W for 30 min. Refractive index decreases from 1.506 to 1.478 with increase in microwave power and extraction time. It may be due to difference in the flavour compounds concentration, traces of solvent and emulsification of water with oil which get extracted during cell rupture present in the essential oil. Instant decrease in refractive index was observed from 200 to 300 W at same extraction time 20 min. It might be due to increase in traces of water present in the oil with increment in microwave power but once essential oil get trapped by solvent during cell rupture in short extraction time, further increment in extraction time gave less effect. The similar findings were reported by [34,35,36].

3.6 Effect on Color Value

The color values varies for L*, a*, b* scale from 70.60 to 98.60, -25.60 to 15.1 and 82.1 to 88.5 for different experimental runs. The analysis of color components L*, a* and b* in the samples demonstrated a significant variation in results. L* coordinate component ranges from 0 black to 100 white where turmeric oil samples vary between 70.60 to 98.60 indicate lightness in extracted oil, chromaticity coordinate a*, the color component ranges from -60 green to 60 red where turmeric oil samples shows values ranging from -25.60 to 15.1, indicating light greenish to light red, chromaticity coordinate b* ranged from (-60) blue to (90) yellow where turmeric oil samples showing variation from 82.1 to 88.5 and a tendency to bright yellow on all samples. Color components L*, a* and b* in the samples were compared with commercially available oil in the market, its color values 75, 14.7, 85 were found within the range of turmeric oil found in the experiment, online portal (e-paint.co.uk) was used for color determination from L, a, b values and was found as pale yellowish red, which is associated with previous literatures cited as [37] for fresh, dried and cured turmeric. There was no effect of experimental combinations on color value of extracted oil. Similarly observed by [38] for frying of oils and fats.

4. CONCLUSION

Essential oil extraction process from turmeric (*Curcuma longa* L.) under microwave assisted extraction system was optimized using response surface methodology technique and it was concluded that the essential oil yield and quality of the essential oil was affected by operational

parameters i.e. microwave power and extraction time. Optimum conditions for extracted essential oil yield were found at 300 W for 20 min with hexane solvent. Maximum essential oil yield under these conditions was measured as 4.973% and essential oil quality parameters were obtained as: Specific gravity: 0.915; Refractive index: 1.485. Microwave assisted extraction provided number of advantages over conventional distillation methods i.e. quick extraction times, low energy consumption and cleaner production.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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