Research Article

Application of 2^k Full Factorial Design in Optimization of Solvent-Free Microwave Extraction of Ginger Essential Oil

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The solvent-free microwave extraction of essential oil from ginger was optimized using a 2^3 full factorial design in terms of oil yield to determine the optimum extraction conditions. Sixteen experiments were carried out with three varying parameters, extraction time, microwave power, and type of sample for two levels of each. A first order regression equation best fits the experimental data. The predicted values calculated by the regression model were in good agreement with the experimental values. The results showed that the extraction time is the most prominent factor followed by microwave power level and sample type for extraction process. An average of 0.25% of ginger oil can be extracted using current setup. The optimum conditions for the ginger oil extraction using SFME were the extraction time 30 minutes, microwave power level 640 watts, and sample type, crushed sample. Solvent-free microwave extraction proves a green and promising technique for essential oil extraction.

1. Introduction

The principal aim of green chemistry and engineering is to reduce chemical related impact on human health and to search alternative, environmentally friendly and energy efficient production methods. Green and clean extraction methods can offer more natural products, free from toxic solvents. The search for such green extraction methods is highly emphasized in essential oils industries since last decade because of consumer's preference towards natural products. Essential oils are volatile extract of the spices, medicinal and aromatic plants. The history of essential oil extraction and their use for various purposes is very old.

Zingiber officinale Roscoe, commonly known as ginger, is a member of Zingiberaceae family. Most Zingiberaceae family spices are fibrous rooted perennial herb which is cultivated in many tropical and subtropical areas, India, North East Asia, Australia, and Japan. The use of ginger as spice and medicine is very old and is mentioned in earliest Chinese and Sanskrit literature [1].

Ginger species possesses aromatic properties and has a commercial importance. There are two valuable extracts of

ginger, essential oil which varies as 0.8–4.2% and oleoresin in the range of about 7% depending on its origin habitat and agronomic treatment of culture [2]. Ginger oil possesses the natural aroma of crude ginger and is globally used in flavour, perfumer, and pharmaceutical and liqueur industry [3]. The therapeutic properties of ginger oil are antiseptic, antispasmodic, carminative, cephalic, expectorant, febrifuge, laxative, and stomachic [4, 5].

The general methods used for extraction of ginger oil are hydrodistillation, steam distillation, H-S distillation, SFE- CO_2 , solvent extraction, and microwave extraction as a recent technique; the oil yield, extraction time, and quality of oil extracted from each method differ significantly and dried rhizome was used in all the methods [6, 7]. Steam distillation is still the principal method of essential oil extraction in industry. All the conventional extraction methods possess the common characteristic of boiling the plant material with water or with organic solvents. Longer extraction time of these conventional methods may degrade the oil quality at the same time leaving the toxic solvent residue in essential oils.

Today, microwave technology for extraction of essential oils and natural extracts has got much attention from scientist

community. In 1992, Pare [8] was the first who demonstrated the use of microwave energy for the extraction of naturally produced compounds from plant tissues; extraction has advantages in terms of yield and selectivity, with better extraction time and essential oil composition free from residual solvents, contaminants, or artifacts, and is also environmentally friendly. Solvent-free microwave extraction (SFME) is a combination of microwave heating and distillation to extract essential oils from plant materials. SFME involves placing the plant material in a microwave reactor without adding any solvent and distillation is performed at atmospheric pressure. The internal heating of the in situ water within the plant material increases the internal pressure and makes the oil cells burst. This process thus frees essential oil which is evaporated by the in situ water of the plant material. SFME has been used for the extraction of essential oils from aromatic and medicinal plants [9–12].

In this study, solvent-free microwave extraction was adopted for oil extraction of green ginger rhizome. The extraction process was optimized in terms of essential oil yield using full factorial design, determining those variables that most influenced essential oil yield. A 2^3 full factorial plan was carried out to model the process. The simple first degree model was used which gave a representation of the response function according to the variables.

2. Materials and Methods

2.1. Raw Material. The ginger rhizome used in this research was purchased from local market nearby H.B.T.I., Kanpur, India. The rhizome was somewhat yellowish brown in color, branched, and laterally flattened. Pretreatment of ginger sample was done to ensure maximum yield of essential oil. Raw ginger was first washed with clean water and partially peeled to remove excess bark. Two types of sample were used: sliced and crushed. Slicing was done longitudinally to an average thickness of 0.20 cm and soaked in water for 18 hours to increase the moisture content for better yield [13].

2.2. Experimental Setup. In the present experimental work the process adopted was solvent-free microwave assisted extraction. Extraction was performed at atmospheric pressure. This was a domestic multimode microwave reactor with frequency of 2450 MHz with a maximum delivered output power of 800 W and input power of 1200 W, having the voltage supply of 230 volts and dimensions of the oven cavity are 206 mm (H) $\times$ 300 mm (W) $\times$ 302 mm (D), with total capacity of 18.5 liters.

The microwave oven was mechanically modified from its original condition to collect the water and oil vapors coming from the ginger sample once it was heated. Standard procedure was followed in modification of microwave oven to prevent the leakage of microwaves, published elsewhere [14]. The extraction vessel was a 1000 mL flat bottomed flask connected to a condensing system. The hole and the microwave containment choke were centered above the turntable and approximately in the center of the microwave chamber. Schematic diagram of the microwave extraction apparatus used for essential oil extraction is shown in Figure 1.

FIGURE 1: Microwave extraction apparatus.

2.3. Extraction of Essential Oil. The fresh ginger rhizomes were washed, cleaned, partially peeled, and thinly grated or crushed, as per requirement prior to extraction. A 100 g of sample was placed in reactor. The runs were taken at three different levels of time and microwave power and with two types of sample, crushed and sliced. Pale yellow colored oil, with a warm, spicy, lemon like odor, was obtained which was separated and dried over the minimum amount of anhydrous sodium sulfate to remove traces of moisture. The essential oil so obtained was stored at low temperature ($4 \pm 2^\circ\text{C}$) in dark; the percentage oil yield is expressed as follows:

$$\text{Oil yield (\%)} = \left(\frac{\text{mass of oil extracted}}{\text{mass of sample}} \right) \times 100. \quad (1)$$

2.4. Chemical Composition of Essential Oil. A standard gas liquid chromatography was used for the analysis of essential oil of ginger obtained from SFME. The analysis was carried out at Fragrance and Flavors Development Centre (FFDC), Kannauj, Uttar Pradesh, India. GLC analysis of the ginger oil was performed on split mode HP (Hewlett-Packard) (make HP 5890) having detector flame ionization with a carbowax column (30 m $\times$ 0.25 mm i.d., film thickness 0.325 μm). The oven temperature was programmed from 50°C (initial time 5 min) to 230°C with $50^\circ\text{C}/\text{min}$ and injector and detector have the temperatures 230°C and 240°C , respectively. The identification of compounds was done by comparing with the retention times of available standard. Area percentage in the chromatogram was used to know the percentage of each compound in the oil. α -zingiberene (30%), α -curcumene (9%), β -sesquiphellandrene (4%), and bisabolene (3.2%) were identified as major constituents in extracted ginger essential oil.

2.5. Mathematical Treatment. To investigate the efficiency of extraction process on ginger essential oil yield, a two-level, 2^3 full factorial design was constructed with two replications of experimental runs. Three variables were chosen, namely, extraction time (X_1), microwave power (X_2), and sample

TABLE 1: Coded and natural variables in 2^3 factorial design.

Levels	X_1 [min.]	X_2 [watt]	X_3
Basic level (0)	20	464	—
High level (+1)	30	640	Sliced
Low level (−1)	10	288	Crushed
Interval	10	176	—

TABLE 2: The 2^3 factorials design including the corresponding responses.

Run	Code variables			Oil yield y (%)
	x_1 (extraction time)	x_2 (microwave power)	x_3 (sample type)	
1	−1	−1	−1	0.10
2	1	−1	−1	0.26
3	−1	1	−1	0.14
4	1	1	−1	0.35
5	−1	−1	1	0.22
6	1	−1	1	0.20
7	−1	1	1	0.28
8	1	1	1	0.44
9	−1	−1	−1	0.12
10	1	−1	−1	0.25
11	−1	1	−1	0.14
12	1	1	−1	0.32
13	−1	−1	1	0.24
14	1	−1	1	0.17
15	−1	1	1	0.31
16	1	1	1	0.46

type (X_3). Each independent variable had 2 levels which were coded as −1 and +1. The coded values of independent variables were found from equation and given in Table 1:

$$x_i = \frac{x_i - x_0}{\Delta x}, \quad (2)$$

where x_0 is the base value at the center of experimental domain and x_i is original variable and Δx is the average value of the difference between highest and lowest values. The sixteen runs in design matrix of 2^3 full factorial designs are set up by randomization. A multiple regression, first degree model was used to express the response as a function of all three factors, which are centered and reduced variables:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3, \quad (3)$$

where β_0 , β_i , β_{ij} , and β_{ijk} represents the average effect, main effects, and two way and three way interactions effects, respectively. Design Expert version 8.0.7.1 (trial version) was applied for performing the experimental design and the data analysis [15].

3. Results and Discussion

3.1. Statistical Analysis. Table 2 shows the design matrix of experimental outcome, as carried out above specified levels

as shown in Table 1 and expressed as extraction yield (y) of essential oil extracted from ginger sample.

It is observed from response table that an interaction model best represents the microwave extraction process in terms of coded variables:

$$Y = 0.25 + 0.056X_1 + 0.055X_2 + 0.040X_3 + 0.031X_1X_2 + 0.028X_2X_3 - 0.029X_1X_3 + 0.019X_1X_2X_3. \quad (4)$$

This regression equation shows that the optimum yield should be located in the experimental domain or very close to it, as the values of β 's are not very high. An average yield of essential oil we can extract from the current experimental setup is $\beta_0 = 0.25\%$ for the current levels of the factors. To assess the goodness of fit of the empirical model and to check the adequacy of model that has been generated by the factorial experiment, analysis of variance (ANOVA) was conducted at 95% confidence level ($P = 0.05$) given in Table 3. Results showed adjusted- R^2 (0.9783), an adequately high degree of correlation between experimental and predicted values. Coefficient of determination R^2 (0.9885) was desirably high (close to 1), adequate precision (AP) 30.411, standard deviation (SD) 0.016, and coefficient of variation (CV) 6.32%. Based on the ANOVA result, it is clear that the model is

TABLE 3: Analysis of variance.

Source	Sum of squares	Degree of freedom (df)	Mean square	F value	P value prob. > F	Remark
Model	0.17	7	0.024	97.83	<0.0001	Significant
A-extraction time	0.051	1	0.051	202.50	<0.0001	
B-power	0.048	1	0.048	193.60	<0.0001	
C-sample	0.026	1	0.026	102.40	<0.0001	
AB	0.016	1	0.016	62.50	<0.0001	
AC	0.013	1	0.013	52.90	<0.0001	
BC	0.012	1	0.012	48.40	0.0001	
ABC	5.625E - 003	1	5.625E - 003	22.50	0.0015	
Pure error	2.000E - 003	8	2.500E - 004			
Cor total	0.17	15

FIGURE 2: Normal probability plot of the residuals.

good simulation of extraction experiment. The predicted R^2 value is also in agreement with the adjusted- R^2 value.

Figure 2 shows the normal probability plot of the residuals for percentage of oil yield. The normal distribution of error can be seen as the value of residuals fit on a straight line to a major extent. The ANOVA results for degradation produced the Pareto chart of main and interaction effects shown in Figure 3, bar lengths being proportional to the absolute value of the estimated effects, which not only help compare the relative importance of effects but also decide the ranking of factors. The interpretation of this chart indicates that the influence of the extraction time is important in the response, followed by power level and both the factors have similar effect on extraction process. Sample type is less effective in comparison to other two factors; uneven crushing of socked ginger sample was favored and extraction using sliced sample resulted in lower yield. The combined effect of duration of extraction and type of sample was not favorable,

FIGURE 3: Pareto chart for significance of parameters.

as was the combined effect of duration of extraction and heating power level. However, we note the very low values for these interactions, which can therefore be ignored in practice, especially for small-scale production units.

3.2. Process Optimization. In order to optimize the conditions of extraction experiment, a desirability function (d) for the simultaneous optimization of multiple responses was used. This function can be described as follows:

$$d = \left(\prod_{i=1}^N m_i^{r_i} \right)^{1/\sum r_i}, \quad (5)$$

where d varies in range of $0 \leq d \leq 1$, and N , r_i , and m represent the number of responses, importance of a particular response, and partial desirability function for specific responses, respectively [16]. Design expert software has inbuilt optimization tool which uses (5). This study aimed to extract the highest amount of essential oil from the sample.

Numerical optimization of extraction process was done using Design Expert 8.0.7.1 and Table 4 shows the settings provided to Design Expert and the final result obtained in

TABLE 4: Setting goal for each factor and response for formulation of optimization and selected optimized optimum conditions.

Factors/response	Goal	Selected optimum conditions	
		Coded	Actual
Extraction time (min.)	In range	1	30 min
Microwave power (w)	In range	1	640 watts
Sample type	In range	1	Crushed sample
Oil yield	Maximum	0.45%	0.45%

numerical optimization. There were 17 solutions found but solution with the highest yield was selected as final result.

The optimum condition in SFME is whenever extraction was performed at high level of all three factors. In other words, the best combination of condition to extract essential oil from green ginger rhizome was extraction time 30 min., microwave power level at 640 watts, and crushed sample by using SFME. This optimum oil yield which was 0.45% carries a desirability value of 0.972.

4. Conclusions

Solvent-free microwave extraction was used for the extraction of useful essential oil from green ginger rhizome. It can be concluded from this study that duration of time is the most dominant factor followed by microwave power and type of sample. For green ginger, solvent-free microwave extraction proved a promising technique with high quality of essential oil. An average of 0.25% of ginger oil can be extracted using current setup.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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