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EFFECT OF EXTRACTION PARAMETERS ON CURCUMIN YIELD FROM TURMERIC

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ABSTRACT

The impact of four independent variables—temperature (50–90°C), particle size (0.42–0.85 mm), mixing duration (10–50 minutes), and solvent-to-meal ratio (10:50)—on the extraction of curcumin from turmeric (Curcuma longa L) was analyzed using a central composite rotatable design. The experimental results showed that curcumin yield varied between 4.49% and 12.89%. The second-order model for curcumin yield had a determination coefficient (R^2) of 0.78 with a standard error of 0.72. The linear, square, and interaction terms were significant at $p < 0.05$, while the lack of fit was not significant at $p > 0.05$. Response surface graphs were utilized to optimize the curcumin extraction conditions. The highest curcumin yield was achieved at 60°C, with a particle size of 0.42 mm, a mixing time of 30 minutes, and a solvent- to-meal ratio of 50:1.

Keywords: Turmeric, Curcumin, Extraction, Temperature, Particle Size.

I. INTRODUCTION

Turmeric, or Curcuma longa L, belongs to the family Zingiberaceae. It is widely cultivated in tropical and subtropical regions of the world. It is a perennial with pulpy, tuberous roots covered with a thick, brown skin and orange-colored flesh. Its odor is mild, somewhat like orange with hints of ginger, whereas its flavor is peppery, warm, and bitter. Major producers of turmeric include India, Indonesia, China, the Philippines, Taiwan, and Jamaica, who account for nearly 90% of global production (Anon 2007)1

Curcuminoids, a category of phenolic compounds occurring in turmeric, account for its color. The three major curcuminoid compounds are diferuloylmethane, p- hydroxycinnamoyl feruloylmethane and p,p'-dihydroxydicinnamoyl methane (Srinivasan, 1952).(8) Curcumin is extracted from the rhizomes of turmeric with the help of solvents yielding a bright yellow crystalline impure compound. The purity of curcumin is enhanced through multiple recrystallizations. It is an oil soluble pigment having a melting point of 174°C by Rao et al. during the year 1970.(7) Though curcumin is stable in acid medium, it gets degenerated in alkaline medium and is light sensitive more so in solution, whereas it is stable in raised temperatures.

Curcumin is of great commercial demand due to its application value in food, cosmetic formulations, and medicine. It is applied in the food industries for giving color to various products that include fats, soups, confectioneries, meat products, and drinks. It protects from oxidation that prevents rancidity (Stankovic, 2004).(9) Medically, it is applied to control the ailments related to digestive problems; purify blood; reduce infections; defend the liver; and lessen cholesterol content. It is also very promising as an anti-inflammatory, anti-HIV, anti-cancer, anti-thrombotic, and anti-Alzheimer agent (Khanna, 1999).(5) Curcumin with its bio-protective properties can scavenge free radicals in the skin thereby reducing damage caused by aging and UV exposure (Khanna, 1999).(5) Functional groups comprising of hydroxyl, keto, and double bonds were found responsible for antioxidant, anti-inflammatory, anti-cancer, and anti-mutagenic activities2).

The study attempts to find optimal conditions for curcumin extraction from turmeric using factors like temperature, particle size, mixing time, and the ratio of solvent to meal employing the response surface methodology.

II. MATERIAL & METHODS

Sample Preparation Turmeric

Turmeric powder (Curcuma longa L.) bought for extraction was from a local supplier. The rhizomes were finely ground and the powder was sieved through sieves of varying sizes: 0.42 mm, 0.50 mm, 0.60 mm, 0.71 mm, and 0.85 mm in order to provide uniform particle size for experiments. Sieve analysis made sure that there was an equal particle size in which the influence of the curcumin yield would be determined by its particle size.

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Isolation Process of Curcumin

For each experiment, 1 g of powder turmeric was weighed and mixed with a known volume of absolute ethanol. This mixture was put into a double-jacketed flask of 18.5 cm length and 6.5 cm diameter to allow simultaneous heating and stirring. The water bath of the Brookfield Inc., USA was set to the desired temperature before each experiment. The extraction process was carried out for a specified time frame that varied between 10 and 50 minutes as per the experimental setup. Concentration of curcumin in the solution was determined by the UV-Visible spectrophotometer (Model 1601, Shimadzu Co. Ltd., Tokyo, Japan) at 425 nm.

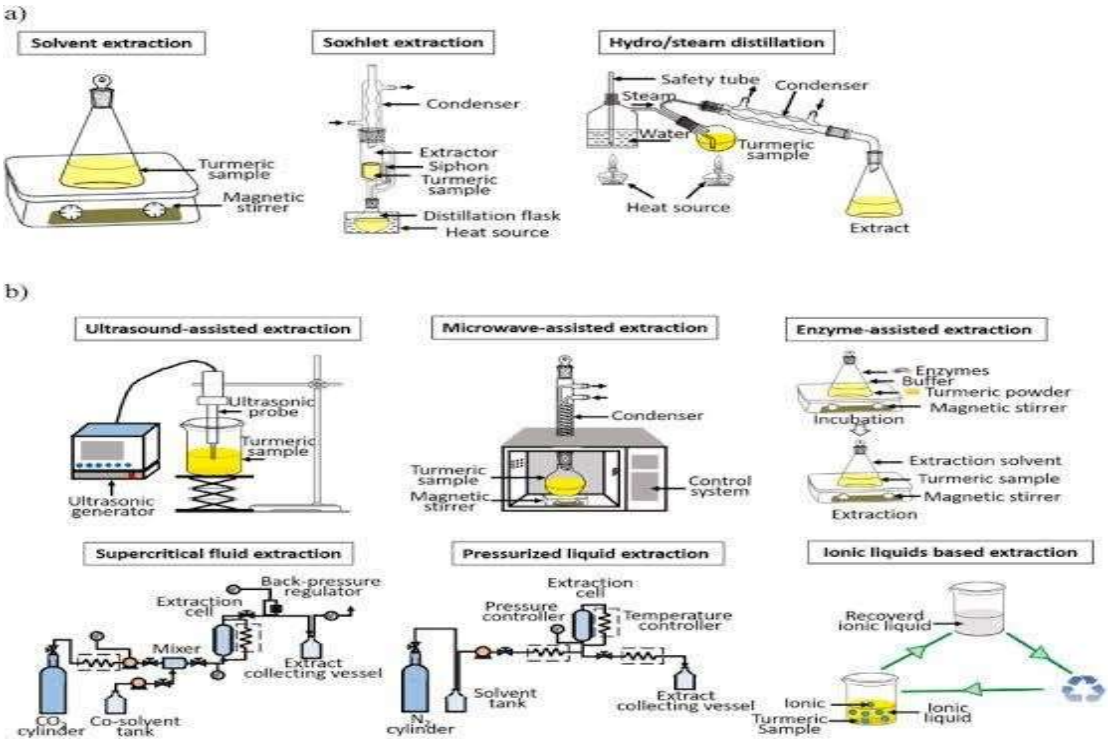

Fig 1:

Curcumin Yield Calculation

The yield of curcumin was calculated using the following formula: Curcumin yield (%) =

Curcumin extracted (g) × 100 / Turmeric used (g)

This calculation enabled a precise comparison of curcumin yield across different experimental conditions.

Preparation of Standard Curve for Curcumin

Curcumin, weighed as 10 mg was dissolved in absolute ethanol. With this, a stock solution containing a concentration of 1 mg/ml was prepared and further diluted to various levels of standard solutions for several concentrations between 0.001 mg/ml to 0.005 mg/ml. All solutions were measured for absorbance at 425 nm and a calibration curve that represented the concentration of curcumin related to the amount of absorbance. Such a calibration curve was used to calculate the curcumin concentrations in each experiment's extract.

Experimental Design and Response Surface Methodology

For understanding the effect of four independent variables, namely temperature (X_1), particle size (X_2), extraction time (X_3), and solvent-to-meal ratio (X_4) on curcumin yield, a central composite rotatable design was used. In this method, there are five different levels to study each variable to further investigate its effect on extraction. This statistical approach allows the generation of a response surface that can identify optimum extraction conditions. It has linear, quadratic, and interaction terms for the proper modeling of the relationships between the variables.

Optimization of Curcumin Yield

Based on the experimental data, regression models were designed for the optimization of the yield of curcumin. Curcumin yield response variable was estimated as the function of two independent variables that varied

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between a few sets of other independent variables fixed at their zero-coded values. Surface response plots are plotted to get graphical impressions of how variable interactions influenced curcumin yields.

Optimization of the results through Design Expert 7.0 gave further analysis that identified ideal conditions to achieve the maximal curcumin content along with maximized levels of soluble solids. Use of the desirability function method resulted in finding 30 potential solutions that meet all the desired criteria. The optimized condition representing the highest desirability is shown in Table 3. In that kind of condition, it has obtained percent of soluble solids determined on a dry weight basis of 2.9% and that curcumin content obtained per dry weight basis was found to be 1.1%. These will provide yields of 12.6% for the solids and 39.4% for curcumin, respectively.

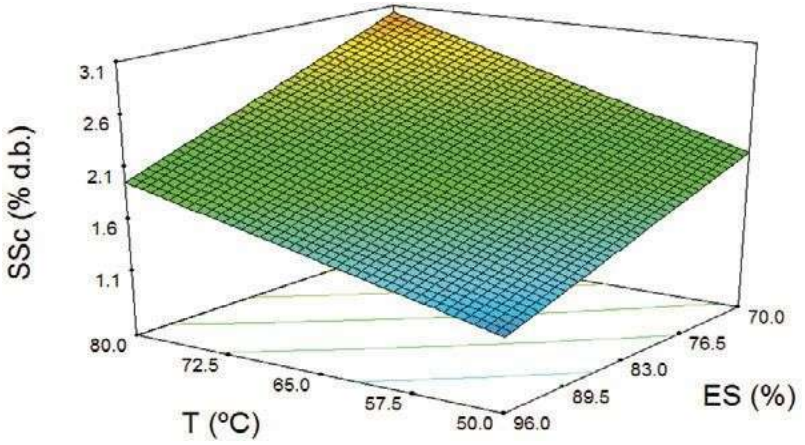

Fig 2:

Figure surface response plot of soluble solid content as a function of extraction temperature and ethanolic strength in the solvent mixture.

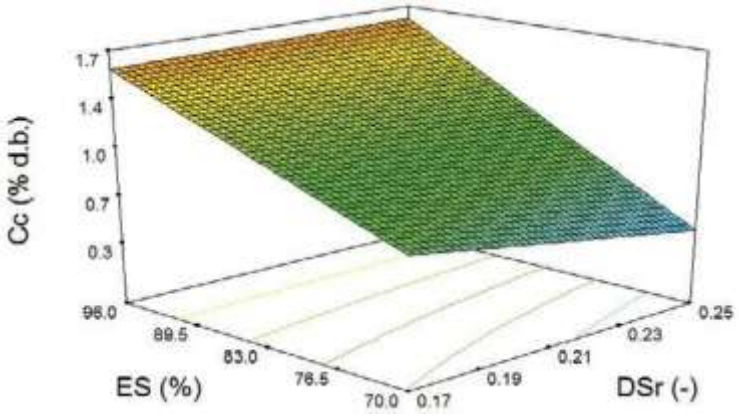

Fig 3:

Figure surface response plot of curcumin content as a function of weight of drug to weight of solvent ratio and ethanolic strength in the solvent mixture.

Statistical Analysis

ANOVA was used to check the significance of each variable and their interaction. For this purpose, Minitab 11.12 (Minitab Inc., USA) was applied. Adequacy of the regression model was checked by examining the coefficient of determination, R^2, and standard error. Predictive accuracy of the model was checked by comparing experimental and predicted values of curcumin yield at different conditions. P-Value was used to indicate the level of significance which was set at $p < 0.05$ with every term in the model being significant.

III. RESULTS & DISCUSSIONS

The values of curcumin isolated by extraction from turmeric showed a range of 4.49 to 12.89 percent as reflected by Table 1, generated from various permutations of factors involved in extraction. There is a strong indication from these results that particle size, temperature, solvent-to-meal ratio, and time for mixing are

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statistically meaningful factors influencing curcumin extraction efficiency. In contrast, yields in the range of 2.83% and 3.28% were reported by Mandal et al. (2007),(6) for microwave-assisted extraction, with the yields in the current work being significantly higher. Contributing factors may be: the type of solvent applied (ethanol vs. acetone), the nature of the extraction process itself, and the characteristics of the turmeric samples.

Temperature and Particle Size Effects

From the graphs of surface response, it has been demonstrated that temperature has some significant effects on curcumin yield. The effect increases the yield with an increase in the temperature and later peaks at 60°C to reduce further. This was made possible because high temperatures also add to increased solubility of curcumin in ethanol, thereby facilitating improved extraction efficiency. However, beyond a certain point, the elevated temperatures may cause thermal degradation of curcumin, which would result in a lower yield. This observation is in agreement with the results of Sun et al. (2002),(10) who reported that curcumin is sensitive to heat at temperatures above 60°C.

Particulate size also determined the yield of curcumin. A yield was realized for smaller sizes, such as 0.42 mm, since greater surface area will allow faster penetration of solvents to leach pigments effectively. The curcumin yields decrease with larger particle size.

Size increased most probably because the surface area left for extraction was lowered in this process. Parallel conclusions were reached by Vijayakumar et al., (2006),(11), who noted that particle size drastically affected curcumin glucosides yield.

Effect of Mixing Time and Solvent-to-Meal Ratio

Mixing time had a less pronounced but still significant effect on curcumin yield. The optimum mixing time was determined to be 30 minutes. Mixing times less than this resulted in incomplete extraction, while longer mixing times did not improve yield and may have even contributed to thermal degradation at higher temperatures. It coincides with the study done by Cuong and Anh in the year 2007, whose findings indicated curcumin degradation due to overexposure to high temperature during the extraction process.

The curcumin yield was also affected by the solvent-to-meal ratio. A ratio of 50:1 (solvent-to-meal) produced the highest yield. This may be because higher volumes of solvent result in greater concentration gradients, and therefore, extraction is more efficient. Lower solvent-to-meal ratios produced lower efficiency since the solvent becomes saturated with curcumin and extraction is less efficient. Stankovic (2004)(9) reports that increasing the volume of the solvent relative to meal improved curcumin extraction although this effect leveled off with further increases in the solvent-to-meal ratio.

Model Validation and Statistical Analysis

The regression model for curcumin yield fits the experimental data very well. The coefficient of determination is 0.78 and the standard error is 0.72. All the linear, quadratic, and interaction terms are significant at $p < 0.05$, which again validates the model's precision in predicting yield for all combinations of parameters. The lack of fit was not significant ($p > 0.05$), which meant that the model described well the relationship between the independent variables and curcumin yield. Similar model validation results were also reported by Vijayakumar et al. in 2006 (11) where the R^2 value for curcumin-bis-α-D-glucoside synthesis by response surface methodology was at 0.9.

The experimental results agreed well with the computed values, within minimal deviations. For example, optimum conditions included: temperature 60°C, particle size 0.42 mm, mixing time 30 minutes, and a 50:1 solvent-to-meal ratio. In such conditions, the experimentally determined curcumin yield was 15.12%, while the calculated value was 14.03%. This extent of precision is a sign of the model's reliability in generating curcumin extraction results based on varying conditions.

Several factors can significantly depend on the efficiency of the curcumin extraction process such as extraction method, solvent composition, time, temperature, ratio of the solvent-to-drug, and pressure. For this reason, the optimization technique used that considered all these parameters. A summary of RSM analysis is provided as Table 1 a summary of the main and interaction effects are shown while significance levels are in per cent; the complete ANOVA tables for the dependent variables are not available

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Table 1:

Run	Variable Levels (uncoded)					Curcumin Yield %
Sr. No.	X1	X2	X3	X3	Exp	Predicted
1	–1 (60)	–1(0.50)	–1(20)	–1(20)	8.75	9.23
2	1 (80)	–1(0.50)	–1(20)	–1(20)	11.02	9.94
3	–1 (60)	1(0.71)	–1(20)	–1(20)	7.78	7.45
4	1(80)	1(0.71)	–1(20)	–1(20)	10.76	9.70
5	–1(60)	–1(0.50)	1 (40)	–1(20)	6.54	6.91
6	1(80)	–1(0.50)	1(40)	–1(20)	5.89	6.63
7	–1(60)	1(0.71)	1(40)	–1(20)	6.55	6.16
8	1(80)	1(0.71)	1(40)	–1(20)	6.80	7.41
9	–1(60)	–1(0.50)	–1(20)	1 (40)	9.06	8.76
10	1(80)	–1(0.50)	–1(20)	1 (40)	6.45	7.22
11	–1(50)	1(0.71)	–1(20)	1 (40)	6.02	5.67
12	1(80)	1(0.71)	–1(20)	1 (40)	5.74	5.67
13	–1(60)	–1(0.50)	1(40)	1 (40)	8.01	9.46
14	1(80)	–1(0.50)	1(40)	1 (40)	6.29	6.93
15	–1(60)	1(0.71)	1(40)	1 (40)	6.02	7.40
16	1(80)	1(0.71)	1(40)	1 (40)	6.51	6.41
17	–2 (50)	0(0.60)	0(30)	0 (30)	5.68	4.87
18	2 (90)	0(0.60)	0(30)	0 (30)	4.49	4.59
19	0 (70)	–2(0.42)	0(30)	0 (30)	12.89	11.69
20	0 (70)	2(0.85)	0(30)	0 (30)	8.89	9.38
21	0 (70)	0 (0.60)	–2(10)	0 (30)	7.05	8.36
22	0 (70)	0 (0.60)	2(50)	0 (30)	8.78	6.77
23	0 (70)	0 (0.60)	0(30)	–2(10)	7.48	8.14
24	0 (70)	0 (0.60)	0(30)	2(50)	8.04	6.67
25	0 (70)	0 (0.60)	0(30)	0 (30)	7.02	7.02
26	0 (70)	0 (0.60)	0(30)	0 (30)	7.01	7.02
27	0 (70)	0 (0.60)	0(30)	0 (30)	7.01	7.02
28	0 (70)	0 (0.60)	0(30)	0 (30)	7.00	7.02
29	0 (70)	0 (0.60)	0(30)	0 (30)	7.01	7.02
30	0 (70)	0 (0.60)	0(30)	0 (30)	7.02	7.02
31	0 (70)	0 (0.60)	0(30)	0 (30)	7.02	7.02

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Table 2: Predicted optimum condition for dynamic maceration extraction of curcumin from turmeric.

Factors	Low	High	Optimum
Et (h)	12	24	12
As (rpm)	30	70	30
DSr (-)	1/6	1/4	1/6
T (ºC)	50	80	80
ES (%)	70	96	70

Et: extraction time; AS: agitation speed; DSr: mass of drug to mass of solvent ratio; T: Extraction temperature; ES: ethanolic strength of the solvent mixture.

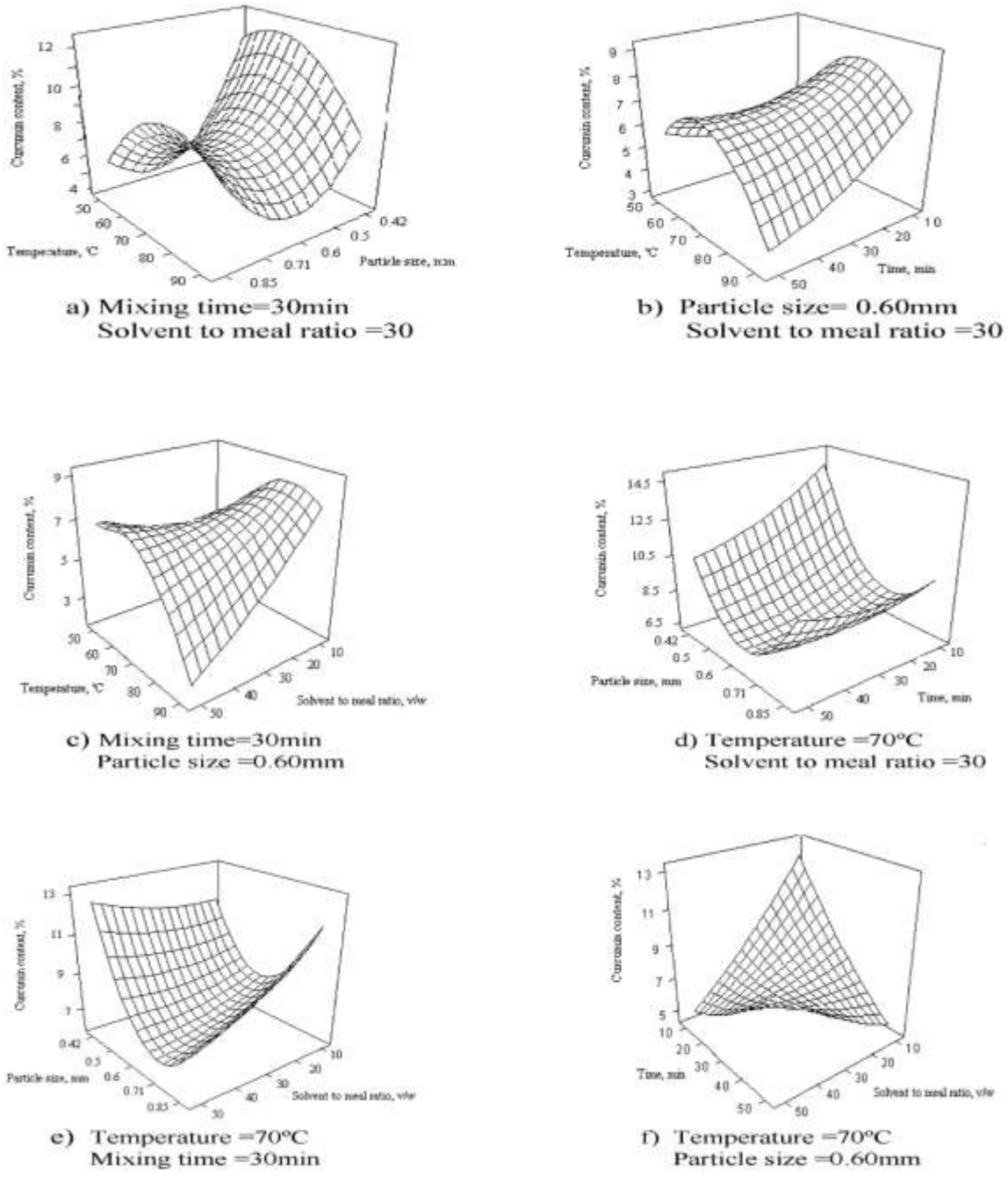

Fig 4: Effect of mixing time, solvent to meal ratio, temperature and particle size on curcumin extracted from turmeric

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Comparison with other studies

This study, with a relatively higher yield than in the earlier studies of Mandal et al. (2007); Sun et al. (2002) (6), demonstrates that optimization of extraction parameters is important. Ethanol probably contributed to the higher yield because this solvent has been proven very effective in extracting bioactive compounds from plant materials (Cuong & Anh, 2007)(3). The central composite rotatable design also allowed for better exploration of the independent variables' effects, thus optimizing the extraction process better.

Furthermore, the destruction of curcumin through higher temperatures and longer extraction time indicates that the parameters controlling its extraction need to be set with much care in order to prevent its degradation industrially. Such results, therefore, may have deep implications for industries using this compound in pharmaceuticals and cosmetics and food products when maintaining the integrity of this compound is of utmost priority.

IV. CONCLUSION

Curcumin extraction yield from turmeric was affected by factors such as temperature, particle size, mixing time, and solvent to meal ratio. Optimization of these factors based on the central composite rotatable design allowed curcumin yields to be between 4.49% and 12.89%. The regression model used was appropriate for curcumin yield prediction, since it had a high accuracy in the R^2 value at 0.78 and a standard error of 0.72. Optimal conditions for curcumin extraction were 60°C, 0.42 mm particle size, 30 minutes mixing time, and a 50:1 solvent-to-meal ratio.

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