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ORIGINAL ARTICLE

Effect of extraction parameters on curcumin yield from turmeric

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Abstract Effect of 4 independent variables – temperature (50–90°C), particle size (0.42–0.85 mm), mixing time (10–50 min) and solvent (ethanol) to meal ratio (10–50) on curcumin yield from turmeric (*Curcuma longa* L) was studied using central composite rotatable design. The experimental value of curcumin yield ranged between 4.49 and 12.89%. The second order model obtained for curcumin yield revealed a coefficient of determination (R^2) of 0.78 and standard error of 0.72. The linear, square and interaction terms were significant at $p < 0.05$ while lack of fit was non-significant at $p > 0.05$. Surface graphs were plotted to optimize the curcumin extraction. The maximum curcumin yield was obtained when temperature, particle size, mixing time and solvent to meal ratio were 60°C, 0.42 mm, 30 min and 50, respectively.

Keywords Turmeric · Extraction · Curcumin · Temperature · Particle size

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Introduction

The turmeric (*Curcuma longa* L) belongs to Zingiberaceae family and is distributed throughout tropical and subtropical region of the world. Turmeric is a perennial plant with pulpy, tuberous roots with a tough brown skin and a deep orange flesh. Turmeric has a mild fragrance slightly reminiscent of orange and ginger while its flavour is peppery, warm and bitter. The leading producers of turmeric are India, Indonesia, China, Philippines, Taiwan, and Jamaica. India is the world's largest producer of turmeric, contributing nearly 90% of the total production (Anon 2007).

Curcuminoids refer to a group of phenolics present in turmeric which are chemically related. The principal colouring components of curcumin are: diferuloylmethane, p-hydroxycinnamoyl feruloylmethane and p, p-dihydroxydicinamoyl methane (Srinivasan 1952). Curcumin is an orange-yellow crystalline powder obtained by solvent extraction of ground rhizomes of turmeric and purification of extract is done by crystallization. The curcumin is an oil-soluble pigment having a melting point of 174°C (Rao et al. 1970). It is stable at acidic pH but readily decomposes at pH above neutral. It is light sensitive especially in solutions but highly stable to heat.

Curcumin is very important due to its use in foods, cosmetics and medicines. It is added to foods such as fats, soups, confectionary, meat products and beverages, to impart colour. It is also used as an antioxidant to prevent the rancidity (Stankovic 2004). It is used as medicine to treat digestive disorders, purify blood, kill germs, protect liver and reduce cholesterol level. It is very effective as anti-inflammatory, anti-HIV, anti-cancer, anti-thrombosis and anti-Alzheimer (Khanna 1999). It has unique bio-protective properties which help in neutralizing the free radicals on the surface of skin, thereby retarding aging and damage due to UV radiations (Khanna 1999). Curcumin has different functional groups such as parahydroxy, keto and double bonds, which are responsible for its antioxidative, anti-inflammatory, anti-cancer and anti-mutagen activities.

The objective of this study was to determine the best combinations of temperature, particle size, mixing time and solvent (ethanol) to meal ratio in the extraction of curcumin from turmeric using response surface methodology.

Material and methods

Extraction of curcumin: Turmeric (*Curcuma longa* L) powder used for the extraction of curcumin was procured from local market. Turmeric powder was passed through different sieves (0.42, 0.50, 0.60, 0.71 and 0.85 mm) to get desired particle size. Turmeric powder (1 g) of each particle size and a measured amount of absolute alcohol were transferred to double-jacketed flasks (length 18.5 cm, diameter 6.5 cm), which was simultaneously stirred and heated using a water bath (Brookfield Inc., USA) at a selected temperature for a predetermined time. Absorbance of sample was taken at 425 nm using UV-spectrophotometer (1601, Shimadzu Co., Ltd., Tokyo, Japan) and amount of curcumin was calculated from standard curve (equation 1).

$$\text{Curcumin yield, \%} = \frac{\text{Curcumin extracted (g)} \times 100}{\text{Turmeric used (g)}} \quad (1)$$

Preparation of standard curve: A stock solution was prepared by dissolving 10 mg of curcumin in absolute alcohol to get concentration of 1 mg/ml. Different concentrations (0.001–0.005 mg/ml) were made by diluting the stock solution with absolute alcohol. The absorbance was read at 425 nm and plotted against concentration.

Experimental design: The effects of four independent variables- temperature (X_1), particle size (X_2), time (X_3), solvent to meal ratio (X_4) at five levels on curcumin yield (response variable) were investigated using central composite rotatable design (Cochran and Cox 1957). Data pertaining to 4 independent and 1 response variable (curcumin yield) was analyzed to get multiple regression equation (equation 2).

$$Y = b_0 + \sum_{n=1}^{4} b_n X_n + \sum_{n=1}^{4} b_{nn} X_n^2 + \sum_{n<m}^{4} b_{nm} X_n X_m \quad (2)$$

where, b_0 is the value for the fixed response at the central point of the experiment; and b_n, b_{nn} and b_{nm} are the linear, quadratic and cross product coefficients respectively, X_n, X_m are process variables.

Optimization of curcumin yield: Regression model was used to compute the response variable (curcumin yield) by varying the values of 2 independent variables while keeping remaining 2 at zero coded level. The surface graphs were plotted for 2 selected independent and their response variables to find their levels with maximum curcumin yield.

Statistical analysis: Analysis of variance of coefficients of regression equation was carried out using Mintiab 11.12 (Minitab Inc USA) software. Experimental and predicted values of curcumin yield were analyzed for coefficient of determination (R^2) and standard error.

Results and discussion

Curcumin extraction: The experimental values of curcumin yield varied from 4.49–12.89% (Table 1). Mandal et al. (2007) performed the extraction optimization of curcumin extraction by microwave assisted process following orthogonal array design and reported the curcumin yield between 2.83–3.28%. The curcumin yield was higher in the present study as compared to previous one (Mandal et al. 2007). It might be due to the composition of turmeric (different sources), extraction condition (ethanol in present and acetone in previous study (Mandal et al. 2007)) and analytical technique (Spectrophotometric in present and HPTLC in previous study (Mandal et al. 2007)).

Extraction optimization: The data pertaining to the independent and response variables were analyzed to get a regression equation with linear, square and interaction coefficients (equation 3)

$$Y = 7.020 - 0.0696X_1 - 0.5763X_2 - 0.3961X_3 - 0.3691X_4 - 0.5708X_1^2 + 0.8796X_2^2 + 0.1369X_3^2 + 0.0975X_4^2 + 0.3845X_1X_2 - 0.2495X_1X_3 - 0.5607X_1X_4 + 0.2580X_2X_3 - 0.3258X_2X_4 - 0.7557X_3X_4 \quad (3)$$

The predicted values of curcumin yield were calculated using regression model and compared with the experimental values. The coefficient of determination (R^2) was 0.78 while standard error was 0.72, which indicated the adequacy of the applied model. The statistical analysis of data revealed that linear, quadratic and interaction coefficients were significant ($p < 0.05$). The lack of fit was non-significant ($p > 0.05$), which further validated the model.

Vijayakumar et al. (2006) studied synthesis of curcumin-bis-α-D-glucoside using central composite rotatable design involving five variables (enzyme concentration, curcumin concentration, incubation period, buffer concentration and pH) at 5 levels and reported R^2 value of 0.9 between experimental and predicted values.

Surface graphs for each combination of 2 independent variables were plotted against curcumin yield for optimizations (Fig. 1). Variation in temperature and particle size revealed that increase in temperature resulted in first increase and then decrease in curcumin yield while increase in particle size showed first decrease and then little increase in curcumin yield. Maximum curcumin yield was obtained at 60°C with 0.42 mm particle size while the other parameters such as mixing time and solvent to meal ratio were kept constant at 30 min and 30, respectively. This trend might be due to increase in solubility initially and afterwards thermal degradation with rise in temperature

Table 1 Central composite arrangement for independent variables X_1 (Temperature, °C), X_2 (Particle size, mm), X_3 (Mixing time, min) and X_4 (Solvent to meal ratio) and their response curcumin yield

Run	Variable levels (uncoded)				Curcumin yield, %	
	X_1	X_2	X_3	X_4	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

whereas as the particle size increased the surface area decreased resulting in decreasing trend but further increase in yield might be due to better leaching of pigment. The effect of temperature and time on curcumin yield revealed that with increase in temperature, curcumin yield first increased up to 60°C and then decreased however, mixing time showed decrease in yield. The maximum yield was obtained when the temperature was 60°C and time 10 min, the particle size and solvent to meal ratio were kept at zero level i.e. 0.60 mm and 30, respectively. It indicated that thermal degradation of the pigment takes place as the duration of mixing time increased and its magnitude was more at high temperature.

Variation in temperature and solvent to meal ratio revealed that there was decrease in curcumin yield with increase in temperature but solvent to meal ratio had marginal effect (Fig. 3). The decrease in yield with temperature could be due to heat degradation but the rate of degradation was less at lower solvent to meal ratio or when the concentration of pigment in alcohol was higher. Maximum curcumin yield was obtained when solvent to meal ratio was 50 and temperature was 60°C while other parameters such as particle size and mixing time were kept constant at 0.60 mm and 30 min, (zero level) respectively. The effect of particle size and mixing time on the curcumin yield revealed that maximum curcumin yield was obtained at 0.42 mm particle size and

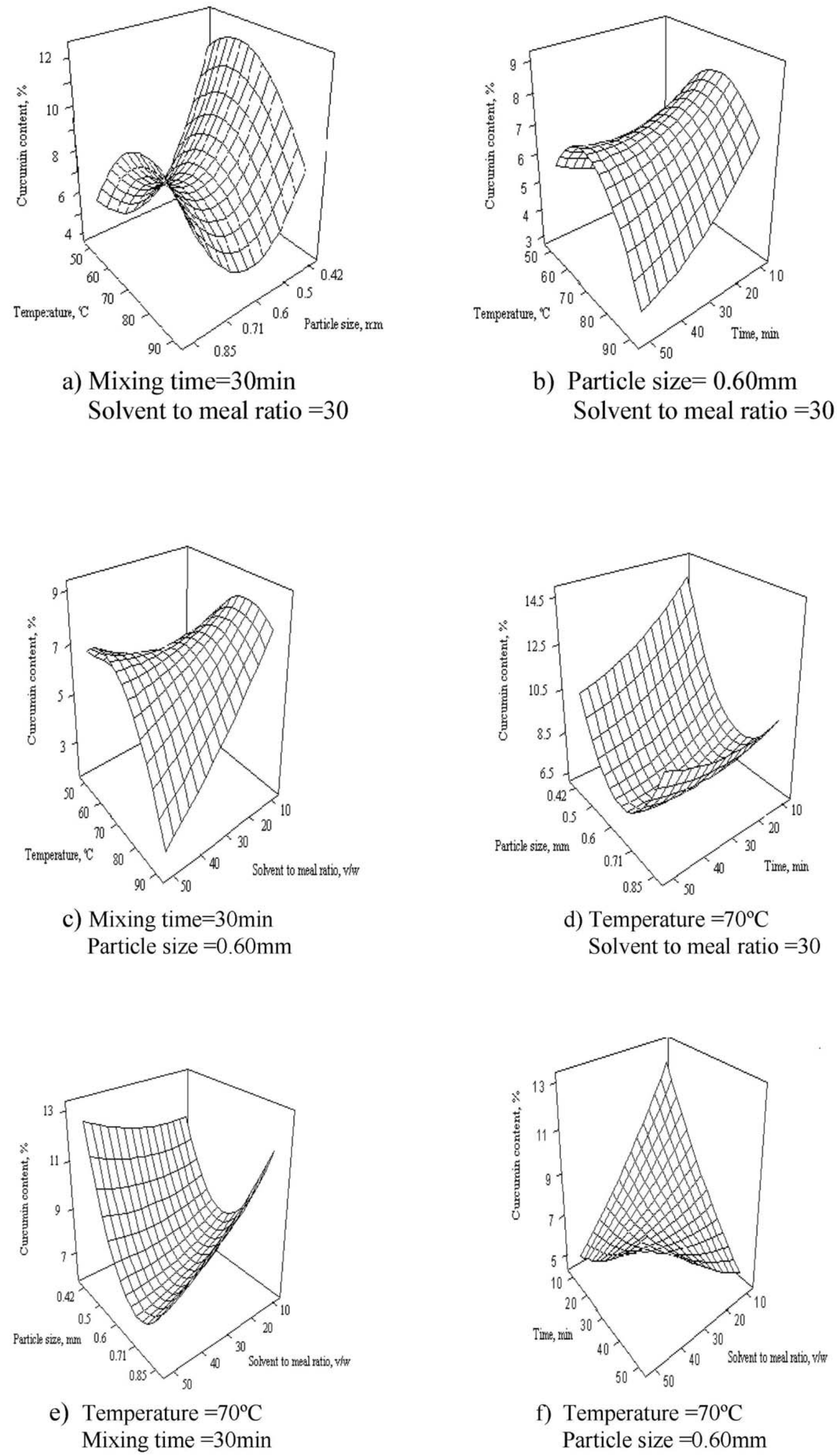

Fig. 1 Effect of mixing time, solvent to meal ratio, temperature and particle size on curcumin extracted from turmeric

10 min mixing time while the other parameter such as temperature and solvent to meal ratio were kept at i.e. 70°C and 30, (zero level), respectively. The yield was higher at lower particle size and short mixing time due to high surface area and less thermal degradation, respectively.

Variation in particle size and solvent to meal ratio revealed that with increase in particle size there was decrease in curcumin yield whereas there was slight change in curcumin yield with increase in solvent to meal ratio. The extraction was higher at lower particle size due to high surface area and at higher solvent to meal ratio due to greater concentration gradient. Maximum curcumin yield was obtained at 0.42 mm particle size and solvent to meal ratio was 50 while temperature and mixing time were kept at 70°C and 30 min, (zero level) respectively. The effect of time and solvent to meal ratio for the curcumin yield showed that with increase in the mixing time and solvent to meal ratio the curcumin yield increased. It could be due to better extraction at greater concentration difference at higher solvent meal ratio and prolonged extraction at longer mixing time. Maximum curcumin yield was obtained when solvent to meal ratio was 50 and 50 min mixing time while the particle size and temperature were kept at 0.60 mm and 70°C, respectively.

It was observed that the curcumin yield first increased and then decreased with increase in temperature. It might be due to increase in solubility with increase in temperature but beyond 60°C thermal degradation reduced the yield. Particle size revealed a typical pattern of first decrease and then increase with increase in size. The decrease in yield might be due to decrease in surface area with increasing size but further increase in yield might be due to better leaching of pigment. The mixing time did not affect the yield at fixed solvent (ethanol) to meal ratio which indicated that extraction is nearly complete at 10 min mixing time. Considering all the extraction conditions it can be concluded that maximum curcumin yield may be obtained when temperature, particle size, mixing time and solvent (ethanol) to meal ratio are 60°C, 0.42 mm, 30 min and 50, respectively.

Goyal and Korla (1993) examined the curcumin quality of 4 turmeric rhizomes during storage. Curcumin content continued to decline upto 10 months, but the change, thereafter, was minor. The curcumin content was found to be maximum in the sample of without storage and was in the range of 2.5 to 4.8%. Sun et al. (2002) recovered more than 80% of curcumin from Chinese herbal medicine by solid-phase extraction cartridge based on tributyl phosphate resin. Mandal et al. (2007) performed the extraction optimization of curcumin by microwave assisted *in vitro* plant bursting by orthogonal array designed and reported optimal results with microwave power (20%), irradiation time (4 min) and particle size (0.50 mm). Cuong and Anh (2007) performed the investigations on the raw curcumin extraction from turmeric based on the alkalinisation method and found 44.58% efficiency when extracted in 42.59% alkali concentration at 90°C for 50 min.

Confirmatory studies: The experiment was run at optimum conditions of temperature (60°C), particle size (0.42 mm), mixing time (30 min) and solvent (ethanol) to meal ratio (50) and got curcumin yield of 15.12%. Under similar conditions calculated amount of curcumin yield using regression model was 14.03%. This study confirmed that conditions were optimal for the curcumin from turmeric powder.

Conclusion

Variables such as temperature, particle size, mixing time and solvent (ethanol) to meal ratio affected the curcumin yield from turmeric. Combination of the parameters as per central composite rotatable design revealed curcumin yield of 4.49–12.89%. The regression model was adequate in predicting the curcumin yield with good accuracy as the coefficient of determination (R^2) and standard error were 0.78 and 0.72, respectively. Optimal conditions for curcumin extraction were 60°C temperature, 0.42 mm particle size, 30 min mixing time and solvent (ethanol) to meal ratio of 50.

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