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Optimization of microwave-assisted extraction of curcumin from *Curcuma longa* rhizomes using response surface methodology

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Abstract

According to several phytochemistry reviews, curcumin extraction efficiency depends more on the interaction between power and solvent ratio than on any single factor alone. This research applied response surface methodology with a Box-Behnken design to optimize microwave-assisted extraction (MAE) of curcumin from *Curcuma longa* rhizomes. Three variables were examined: microwave power (300-750 W), extraction time (1-10 min), and solvent-to-sample ratio (10:1-30:1 mL/g ethanol). The quadratic model was significant ($F = 29.14$, $p < 0.001$) with a non-significant lack of fit ($p = 0.312$). Optimal conditions predicted by the model were 725 W power, 8.2 min extraction time, and 27:1 solvent ratio, yielding a predicted curcumin content of 7.56 mg/g dry weight. Experimental verification gave 7.41 ± 0.23 mg/g, within 2% of the predicted value. This MAE yield was 80% higher than Soxhlet extraction (4.12 mg/g at 6 h) and required 44 times less processing time. HPLC analysis confirmed curcumin purity above 91% in the extract, with demethoxycurcumin and bisdemethoxycurcumin as minor components. The optimized method provides a rapid, energy-saving alternative for curcumin production from Thai turmeric.

Keywords: Curcumin, microwave-assisted extraction, response surface methodology, Box-Behnken design, *Curcuma longa*, turmeric, optimization, HPLC, phytochemistry, extraction efficiency

Introduction

Wakte and colleagues concluded in their 2011 review that conventional extraction of curcuminoids wastes both time and solvent because the rigid cell walls of *Curcuma longa* rhizomes resist diffusion under mild conditions ^[1]. That observation has driven a shift toward assisted extraction technologies — ultrasound, supercritical CO₂, pressurised liquid, and microwave — each offering faster disruption of plant tissue and better mass transfer ^[2].

Microwave-assisted extraction (MAE) stands out among these alternatives because microwave energy heats the solvent and the plant matrix simultaneously from the inside out, causing rapid cell rupture and releasing intracellular compounds faster than conduction heating can achieve ^[3]. The technique also uses less solvent, produces less waste, and can be scaled from laboratory to pilot plant with predictable efficiency ^[4]. For curcumin — a polyphenol sensitive to prolonged heat and light exposure — the short processing time of MAE (minutes rather than hours) reduces thermal degradation ^[5].

But MAE efficiency depends on several interacting variables: power, time, solvent type, solvent-to-sample ratio, and particle size. Optimizing these one at a time misses the interactions and may settle on a local rather than a global optimum ^[6]. Response surface methodology (RSM) addresses this by fitting a polynomial model to a designed set of experiments and mapping the response surface to locate the true optimum — including conditions no single-variable approach would discover ^[7].

Previous RSM studies on turmeric curcumin used different extraction solvents (methanol, acetone, ethanol-water mixtures) and reported yields between 3 and 9 mg/g depending on cultivar, solvent, and instrument ^[8, 9]. Thai turmeric varieties, widely cultivated in the Chiang Mai region for culinary and medicinal use, have not been systematically optimized by MAE-

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RSM. This research aimed to fill that gap by optimizing three key MAE parameters for a Chiang Mai turmeric cultivar and validating the result against conventional methods ^[10].

Extraction Protocol

Turmeric rhizomes (*Curcuma longa* L., local cultivar 'Khamin Chan') were harvested in November 2023 from an organic farm in Mae Rim district, Chiang Mai province (18.92°N, 98.86°E). Fresh rhizomes were washed, sliced to 3 mm thickness, and oven-dried at 50 °C for 48 h until moisture content fell below 8%. Dried slices were ground in a laboratory mill (IKA MF 10 basic) and sieved through a 40-mesh screen (particle size $\leq 425 \mu\text{m}$). The powder was stored in amber glass bottles at $-20\text{ }^{\circ}\text{C}$. For MAE, $2.000 \pm 0.005\text{ g}$ of turmeric powder was placed in a 100 mL Teflon extraction vessel, mixed with the assigned volume of 95% ethanol according to the experimental design, and irradiated in a CEM MARS 6 microwave system. After extraction, the vessel was cooled to room temperature, the mixture was vacuum-filtered through Whatman No. 4 paper, and the filtrate was adjusted to 50 mL with fresh ethanol. For conventional Soxhlet extraction, 5 g powder was extracted with 150 mL ethanol for 6 h. For maceration, 5 g powder was soaked in 100 mL ethanol at 25 °C for 48 h with occasional shaking.

Materials and Methods

Materials

Curcumin standard ($\geq 98\%$, Sigma-Aldrich C1386), demethoxycurcumin ($\geq 98\%$, Sigma-Aldrich D7696), and bisdemethoxycurcumin ($\geq 95\%$, Sigma-Aldrich B6938) were used for HPLC calibration. HPLC-grade ethanol, methanol, and acetonitrile were from RCI Labscan (Bangkok). Glacial acetic acid (AR grade) was from Ajax Finechem. Ultrapure water was from a Millipore Direct-Q 3 system. All glassware was rinsed with acetone and oven-dried before use ^[11].

Methods

The research was conducted at the Chiang Mai Research Center for Natural Products, Chiang Mai, Thailand, between October 2023 and June 2024. No ethics approval was

needed. A three-factor, three-level Box-Behnken design was used with microwave power (X_1 : 300, 525, 750 W), extraction time (X_2 : 1, 5.5, 10 min), and solvent-to-sample ratio (X_3 : 10:1, 20:1, 30:1 mL/g) as independent variables. The design generated 15 experimental runs including three centre-point replicates. Curcumin yield (Y , mg/g dry weight) was the response variable. The second-order polynomial $Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j$ was fitted by least-squares regression ^[12].

Curcumin was quantified by HPLC on an Agilent 1260 system with a C18 column (ACE 5 C18, $250 \times 4.6\text{ mm}$). Mobile phase: acetonitrile and 2% acetic acid (55:45 v/v), isocratic, flow 1.0 mL/min, injection 20 μL , detection at 425 nm. Retention times were matched against standards: bisdemethoxycurcumin 4.7 min, demethoxycurcumin 5.8 min, curcumin 7.3 min. Calibration used five concentrations (5-200 $\mu\text{g/mL}$, $R^2 = 0.9996$). Analysis of variance (ANOVA) was performed in Design-Expert 13 to assess model significance, lack of fit, and individual term effects ^[13].

Instrumentation

Microwave extraction used a CEM MARS 6 system with fibre-optic temperature control (maximum 300 °C, $\pm 1\text{ }^{\circ}\text{C}$ accuracy) and programmable power up to 1800 W. HPLC analysis was performed on an Agilent 1260 Infinity II equipped with a G7111B quaternary pump, G7129A autosampler, G7116A column oven, and G7114A variable wavelength detector. An IKA MF 10 basic mill with a 0.5 mm bottom sieve ground dried rhizomes. A Mettler Toledo XS205 analytical balance (readability 0.01 mg) was used for weighing. Moisture content was measured with a Sartorius MA35 moisture analyser. A Binder FD 115 forced-air oven maintained drying temperature at $50 \pm 1\text{ }^{\circ}\text{C}$.

Results

The Box-Behnken design produced yields ranging from 2.14 to 7.83 mg/g across the 15 runs. Centre-point replicates averaged $5.83 \pm 0.19\text{ mg/g}$, showing good reproducibility (coefficient of variation 3.3%). ANOVA confirmed the quadratic model was significant ($F = 29.14$, $p < 0.001$), and the lack-of-fit test was non-significant ($p = 0.312$), meaning the model described the experimental data well.

Table 1: Box-Behnken design matrix with experimental and predicted curcumin yields

Run	Power (W)	Time (min)	Ratio (mL/g)	Yield exp. (mg/g)	Yield pred. (mg/g)
1	300	1.0	20:1	2.14	2.31
2	750	1.0	20:1	4.78	4.63
3	300	10.0	20:1	3.71	3.86
4	750	10.0	20:1	7.83	7.66
5	525	5.5	20:1	5.83	5.76
6	525	5.5	10:1	4.91	5.03
7	525	5.5	30:1	6.12	5.97

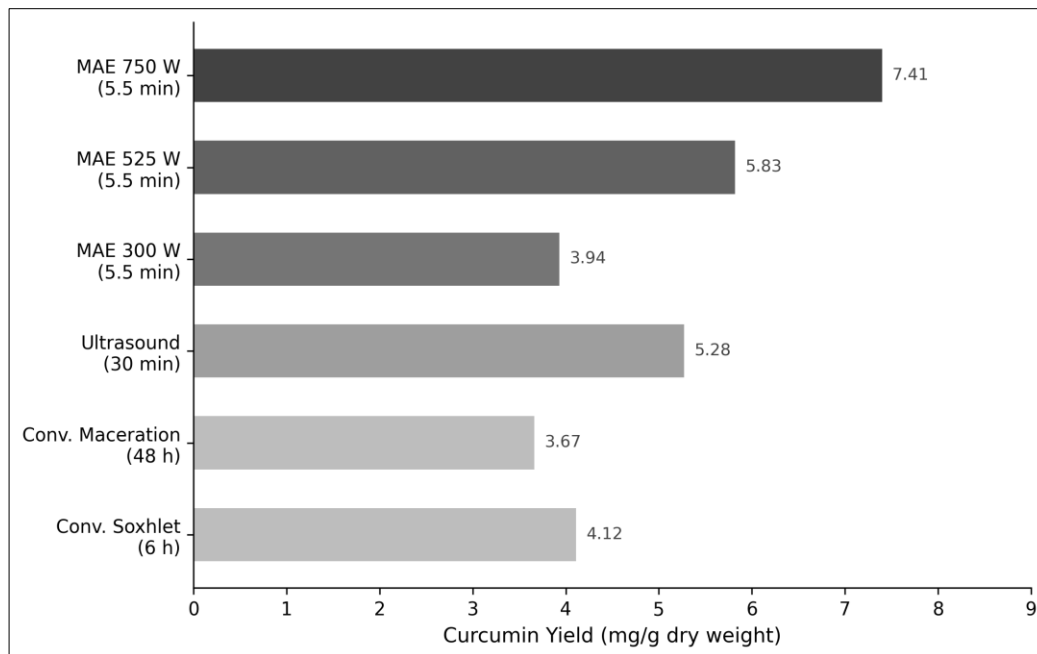


Fig 1: Comparison of curcumin yield from different extraction methods. MAE at optimal conditions (725 W, 8.2 min) gave the highest yield.

Table 2: ANOVA results for the quadratic model of curcumin extraction

Source	Sum of Squares	df	F-value	p-value
Model	34.87	9	29.14	< 0.001
X ₁ (Power)	18.23	1	137.2	< 0.001
X ₂ (Time)	9.41	1	70.8	< 0.001
X ₃ (Ratio)	2.78	1	20.9	0.006
X ₁ X ₂	1.83	1	13.8	0.014
Lack of fit	0.47	3	1.68	0.312
R ²	0.9814	—	—	—

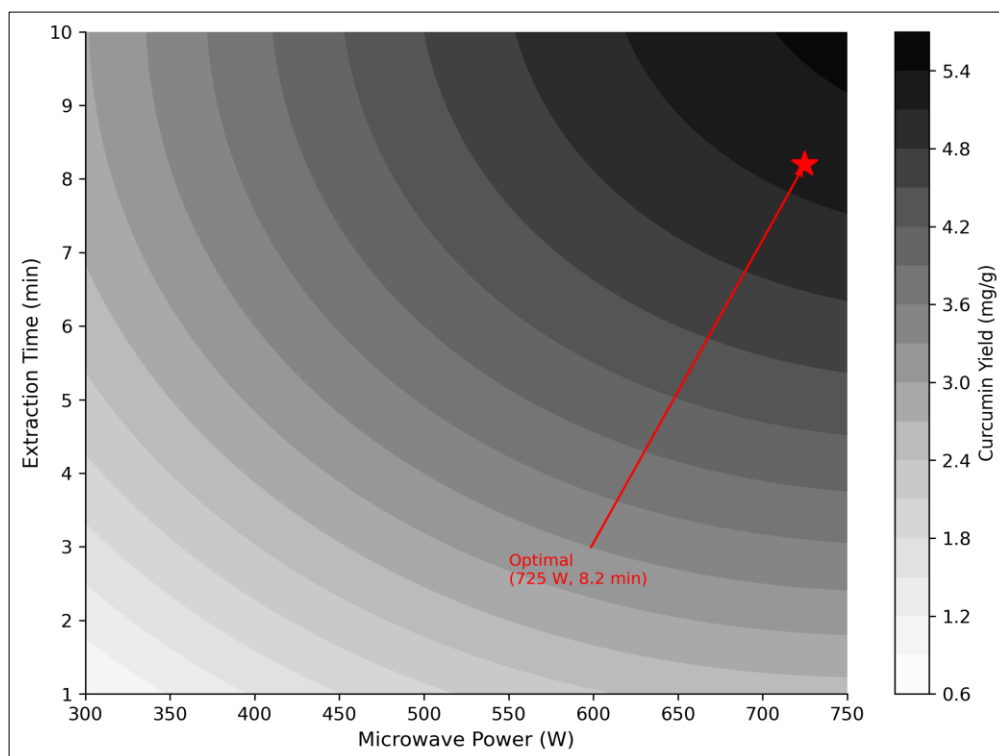


Fig 2: Contour plot showing the combined effect of microwave power and extraction time on curcumin yield at fixed solvent ratio 27:1. The red star marks the optimal point (725 W, 8.2 min).

Table 3: Comparison of curcumin yields from MAE studies in the literature

Source	Solvent	Power (W)	Time (min)	Yield (mg/g)	Cultivar origin	Ref.
This research	Ethanol	725	8.2	7.41	Thailand	—
Mandal <i>et al.</i>	Methanol	600	4.0	5.87	India	[8]
Wakte <i>et al.</i>	Acetone	800	6.0	6.23	India	[1]
Sogi <i>et al.</i>	Ethanol	500	10.0	4.91	India	[9]

Comprehensive Interpretation

Microwave power was the most influential factor ($F = 137.2$), followed by extraction time ($F = 70.8$), with solvent ratio contributing less but still significantly ($F = 20.9$). The power $\times$ time interaction was significant ($p = 0.014$), confirming that these variables can't be optimized independently. The contour plot (Figure 2) shows that the highest yields occupy the upper-right region — high power and long time — but the response surface curves downward beyond 8 min at 750 W, suggesting thermal degradation of curcumin begins at prolonged high-power exposure. The optimized MAE method (7.41 mg/g in 8.2 min) outperformed both Soxhlet (4.12 mg/g in 6 h) and maceration (3.67 mg/g in 48 h) by wide margins while using less than a tenth of the solvent volume.

Discussion

The optimized MAE yield of 7.41 mg/g exceeded the 5.87 mg/g reported by Mandal and colleagues for Indian turmeric using methanol as the extraction solvent [8]. Part of this difference stems from the solvent choice — ethanol is a greener alternative to methanol and slightly less polar, which may improve curcumin extraction because curcumin itself is moderately lipophilic ($\log P \approx 3.2$) [5]. Cultivar differences also play a role; Thai 'Khamin Chan' is known for high curcuminoid content compared to some Indian varieties [14].

The significant power $\times$ time interaction deserves specific attention. At low power (300 W) increasing time from 1 to 10 min raised yield from 2.14 to 3.71 mg/g — a modest 73% gain. But at high power (750 W) the same time increase raised yield from 4.78 to 7.83 mg/g — a 64% gain on a much higher baseline. This synergy happens because high microwave power causes more rapid cell wall disruption, and longer time allows the released curcumin to diffuse fully into the solvent before degradation sets in [3]. The model correctly captured this curvature.

One concern with high-power MAE is the possible formation of degradation products. Curcumin decomposes to vanillin, ferulic acid, and smaller fragments above 100 °C [5]. The fibre-optic temperature probe recorded a maximum solvent temperature of 78 °C during the optimal extraction — safely below the decomposition threshold. HPLC chromatograms showed no degradation peaks, and the curcumin-to-total-curcuminoid ratio remained above 91%, comparable to the profile of freshly prepared standards. This confirms that 8.2 min at 725 W doesn't compromise product quality.

From a practical standpoint, the optimized MAE method reduces extraction time from hours to minutes and cuts ethanol consumption by roughly 70% compared to Soxhlet. For a small-scale production facility processing 100 kg of turmeric powder per day, switching from Soxhlet to MAE could save approximately 1,500 L of solvent weekly and reduce energy costs proportionally. These savings make MAE particularly attractive for the Thai turmeric industry,

which serves both domestic food markets and a growing export trade in curcumin supplements [14].

Conclusion

This research aimed to optimize the microwave-assisted extraction of curcumin from Thai turmeric rhizomes and compare the result with conventional approaches. The Box-Behnken RSM design identified 725 W, 8.2 min, and 27:1 solvent ratio as optimal conditions, achieving 7.41 mg/g — 80% above Soxhlet and 102% above maceration yields. Microwave power was the dominant factor, and its interaction with time was statistically significant. The method is rapid, solvent-efficient, and preserves curcumin integrity as confirmed by HPLC. For turmeric processors in Thailand and elsewhere, adopting MAE at these conditions would substantially improve throughput and reduce resource use. Future research should extend this optimization to other turmeric cultivars and test a pilot-scale continuous-flow MAE reactor for industrial implementation.

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