Food Science and Engineering
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Research Article

Effect of Microwave Boiling and Ultrasound-Assisted Extraction on Curcumin from the Turmeric Rhizomes

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Received: 12 June 2023; **Revised:** 2 August 2023; **Accepted:** 11 August 2023

Abstract: The present work is proposed to study the effect of microwave boiling on the curcumin content of turmeric. Fresh rhizomes are subjected to different boiling methods i.e., pressure cooking, water boiling, and microwave boiling. Dried turmeric powder is used for the extraction of curcumin with the use of different solvents. Among the solvents used, ethanol to water ratio (70:30) has the highest extraction yields. Further, the curcumin extraction yields were studied using ultrasound technology at different time intervals. Ultrasound-assisted extraction (UAE) for 25 min treatment at 250 W was observed to have the highest yield. The rhizomes that are pressure cooked and tray dried at 90 °C followed by the ultrasound-assisted extraction have resulted in maximum curcumin content. The experimental curcumin value is compared with the calculated value obtained from Peleg's model. Ultrasound can be used as a green technology to have enhanced extraction yields of bioactive compounds.

***Keywords*:** turmeric, curcumin, solvent extraction, ultrasonication, ultrasound-assisted extraction

1. Introduction

In 2025 the population of planet Earth will reach 10 billion and there is a need for innovative and emerging food processing technologies to meet the new food demands [1]. One of the key objectives is a 'Better society' through risk assessment, present and new challenges in the food system. This can be achieved through the development of innovative scientific technologies. The gap between the old and recent food processing technologies can be decreased using non-thermal technologies. In recent days, non-thermal technologies emerged with a wide range of applications in different fields of food processing like meat, poultry, dairy and also in other fields of agriculture and medicine. Some of the nonthermal food processing technologies are Pulsed electric field, high-pressure processing, ultrasound, cold plasma, pulsed UV light etc [2-6]. Ultrasound refers to sound waves having frequencies beyond the audible range of human beings, i.e. > 20 kHz. High-intensity ultrasound having frequencies between 20 to 100 kHz is commonly used in food preservation. Ultrasonication involves the propagation of sound waves through the liquid matrix and generates a physical phenomenon known as cavitation resulting in the formation of bubbles [4]. Most of the research has been carried out on the application of ultrasound for the enhanced extraction of bioactive compounds like antioxidants [7], phenols [8], anthocyanins [9] and polysaccharides [10] etc. Although the results reported in the above works are quite convincing but the work done on ultrasound-assisted extraction of curcumin is very limited. A work reported by

DOI: https://doi.org/10.37256/fse.4220233207

Shirsath et al. [11] on ultrasound-assisted extraction of curcumin observed a 72% yield after 1 h extraction whereas 62% was observed at 8 h with the conventional extraction method. Another investigation carried out by Mandal et al. [12] reported higher yields of curcumin at 70 min of ultrasound-assisted extraction compared to long hours of solvent extraction.

Turmeric is obtained from the root of the Curcuma longa plant which belongs to the ginger family. Turmeric has been used as a spice for color and flavor for many years. The most useful part of the plant is the root containing the rhizome. Rhizome has tough brown skin and deep orange flesh. Patil et al. [13] reported that the three main active bioactive components of curcuminoids i.e. curcumin (70-74%), demethoxycurcumin (10-34%), and bisdemethoxycurcumin (2-10%) are observed in turmeric. Of those three components of turmeric, the functional and antimicrobial property is due to the curcumin. Curcumin has several potential applications in food and pharmaceuticals. In food processing, it is used as an antioxidant, coloring agent, antimicrobial agent, and spice. Traditional processing of raw turmeric includes boiling (hot water or steam) followed by the drying of the rhizomes. The dried rhizomes are then subjected to pulverization to a powder form that has many uses as a food and pharmaceutical ingredient.

The conventional methods are used to isolate and extract the curcumin from the turmeric powder. Some of the conventional extraction methods include steam distillation, hot and cold percolation, hydrotrope, alkaline, and organic solvents [13]. The solvent extraction method often involves the use of high temperatures, longer extraction periods, and lesser extraction efficiencies. Moreover, many of the bioactive compounds are heat sensitive that might degrade during the extraction. Curcumin is freely soluble in methanol, chloroform, ethanol, and acetone but practically less soluble in water. Various analytical methods have been developed in recent years for the quality control analysis of herbs and their formulation including High-performance liquid chromatography, High- performance thin layer chromatography, and Ultra Violet-Visible Spectrophotometry. The present work was carried out on the effect of different boiling methods i.e. hot water, pressure boiling, and microwave boiling followed by drying and pulverizing. The drying is done by natural (solar drying) and mechanical (tray drying) methods. In the second part of the work, the curcumin from the turmeric powder is extracted using different solvents.

The ultrasonication has led to enhanced mass transfer rate and cavitation involved in the portion of tissue cells that has facilitated the easy extraction of curcumin [11]. Carniel et al. [14] reported that the sound waves generated in the extraction medium resulted in the eddy current and turbulence formed due to the implosion of the bubbles attributed to an increase in the acceleration of solvent through the porous tissue cells responsible for the enhanced extractions. The bubbles implosion converts the potential energy to kinetic energy this energy accelerates the solvent towards the cells in the form of jets leading to the rupture and breakdown of the cell wall facilitating the easy leaching of curcumin [12]. Binello et al. [15] reported that ultrasound-assisted extraction is widely accepted because of higher yields and rates, low cost, limited use of solvents, and it can preserve heat sensitive compounds. The UAE decreases the working times, higher yields and the extract quality. This study includes the ultrasound-assisted extraction of curcumin from turmeric [16]. Milićević et al. [17] reported that Peleg's model is used to predict the extracted values of curcumin. This model is an empirical and classic hyperbolic model initially developed to describe moisture sorption curves.

2. Material and methods

2.1 *Materials*

The systematic approach adopted for the extraction of curcumin from turmeric powder is presented. Freshly harvested rhizomes which are of Guntur red variety collected from Regional Sugar & Rice Research Station, PJTSAU, Rudrur. As a control sample, branded turmeric powder was purchased from the local market for solvent optimization studies. The standard curcumin (assay > 98%) is purchased from Sigma-Aldrich. All the chemicals used in the study were analytical grade.

2.2 *Methods*

The freshly harvested rhizomes were immediately washed with clear water. The finger rhizomes were separated from the mother rhizomes. The rhizomes were cooked using 3 different methods i.e. 1. Water Boiling, 2. Pressure cooking, 3. Microwave heating.

2.2.1 *Water boiling*

The rhizomes are boiled in water for 1 h in the open vessel to allow complete gelatinization of the starch and even distribution of curcumin, avoiding raw odor, reducing drying time and yielding a uniformly colored product.

2.2.2 *Pressure cooking*

A pressure cooker is based on the principle that with the increase in pressure, the boiling point of water increases. The rhizomes were pressured cooked at 15 psi for 30 min that corresponds to a temperature of 121 °C [18].

2.2.3 *Microwave heating*

Microwave heating basically works on the principle of volumetric heating which means the material can absorb microwaves. The microwave boiling is done using a Samsung microwave oven for 30 min at 915 MHz operated at 750 W output capacity.

2.2.4 *Drying of rhizomes*

The drying operations were done using the method prescribed by Nithya et al. [19]. The boiled rhizomes are subjected to drying in solar drying (temperature 65 ± 5 °C) and tray dryer at 90 °C till desired moisture content is below 10% [20]. The airflow rate in the tray drier was adjusted to 3 m/s. The moisture of the dried rhizomes was determined using the hot air oven method given by AOAC [21]. The completion of drying was indicated when rhizomes become hard, brittle and would break with a metallic sound. After drying the rhizomes were pulverized and sieved to a size of 100 µm using a sieve shaker.

2.2.5 *Optimization of solvents for curcumin extraction*

Different solvents i.e. methanol, ethanol, acetone and ethyl acetate were used to extract the curcumin content. 2 g sample is taken in a conical flask and 50 mL of different solvents were added to the flask at each time. The mouth of the flask is closed tightly with the cotton plug and sealed with paraffin tape and kept in a shaking water bath for 4 h. The samples were analyzed for curcumin content using a UV-Vis spectrophotometer.

2.2.6 *Optimization of solvent ratio for extraction*

The solvent that has resulted in the maximum curcumin extraction will be finalized and used for the extraction of curcumin at different proportions. Different solvent to water ratio (Solvent: water) 100:0 v/v, 70:30 v/v, 50:50 v/v, 30:70 v/v were used for the extraction. The extraction was done using a shaking water bath maintained at 37 °C for a period of 2-12 h. The samples were taken at a regular time intervals of 2 h and quantified for the curcumin content.

2.2.7 *Ultrasound-assisted extraction of curcumin*

Ultrasound-assisted extraction was carried out using the ultrasonication bath at a constant frequency. For the extraction of maximum curcumin from the turmeric different operating parameters of ultrasonication bath need to be optimized. Based on the available literature, different operating parameters are chosen for the present work. Parameters like ultrasound input power (250 W), treatment time (5, 10, 15, 20, 25 and 30 min), duty cycle (60%), solvent to water ratio (70:30 v/v) were used to study the enhanced extraction.

2.2.8 *Quantification of curcumin content using UV- visible spectrophotometer*

The absorbance of the standard and samples were observed using a spectrophotometer at 422 nm. A standard curve is prepared at a concentration of 0.1 mg/ml in solvent to water ratio (70:30). The quantification of curcumin content was done using the standard curve.

2.2.9 *Kinetic model*

The influence of ultrasound-assisted extraction time on the curcumin concentration is analyzed using Peleg's fitting model as per the procedure given by Shirsath et al. [11]. Peleg is a semi-emperical kinetic well-known model widely used to explain the extraction curves of the biological molecules extraction. There is a great similarity between the extraction curve and sorption curves the Peleg model is widely used. The equation of the Peleg kinetic model of extraction as follows

$$C(t) = C_0 + \frac{t}{K_1 + K_2 t}$$

Where

C_t is the concentration of curcumin at time t (mg curcumin/g turmeric powder);

C_0 is the initial concentration of curcumin which is zero, as fresh solvent is used;

K_1 is Peleg's rate constant (min. g/mg);

K_2 is Peleg's capacity constant (g/mg).

2.2.10 *Statistical analysis*

The results were statistically analyzed by one-way ANOVA using SPSS (IBM statistical analysis version 19). Duncan's new multiple-range test was used to determine significant differences. Statistical significance was given at $P < 0.05$ post-hoc comparison, SPSS 19 version. All the tests were conducted a minimum of triplicates.

3. Results and discussion

3.1 *Optimization of solvent for the curcumin extraction*

For the extraction of curcumin, four different solvents (methanol, ethanol, acetone, and ethyl acetate) have been used. After 4 h of extraction with 100% of the above solvents, ethanol extraction was observed to have more curcumin content. The values of curcumin content quantified using different solvents are given in Figure 1a. The highest curcumin content 1.99 mg/g was observed in ethanol extraction whereas the least content (1.48 mg/g) is observed was in ethyl acetate extraction. Shirsath et al. [11] observed a higher value of curcumin content in ethanol extraction than the other solvents used. The authors have also stated that the ethanol being a high polar, lower viscous, and has higher vapor pressure resulted in higher extraction of curcumin compared to the other solvents. The less polar curcumin requires a high polarity solvent like ethanol for better extraction efficiency [22]. Priyadarshini et al. [23] reported that among the several organic solvents, ethanol is widely used for the extraction of curcumin. Similarly, Sogi et al. [24] and Patil et al. [25] used ethanol as a solvent for the extraction of curcumin due to its higher extraction efficiency. Hikmawanti et al. [26] reported that ethanol is a protic organic solvent with a polarity index of 5.2 and dielectric constant of 24.55 and is less toxic than the other solvents. The polarity index of ethanol is more that of acetone and methanol. In this study, ethanol retained the maximum curcumin content among the other solvents. However, there is a necessity to optimize the ethanol concentration for better extraction efficiencies.

Secondly, the solvent to water ratio is optimized for maximum extraction yields. The solvent to water ratio (ethanol and water, 70:30) resulted in higher curcumin content than the 100% ethanol, 50% ethanol and 50% water, 30% ethanol and 70% water. The optimized solvent ratio resulted in higher curcumin extraction (2.3 mg/g). The least curcumin content (1.91 mg/g) was observed in the 30% ethanol and 70% water solvent ratio. The reason for the lowest yield with more water extraction is due to the hydrophobic nature carrying on the curcumin molecule [27]. The curcumin content and different solvent ratio are given in Figure 1b. The decrease in the ethanol percentage in the extraction media resulted in lower extraction yields. The yield of the bioactive compound also depends on the extraction time. In this study, different extraction times ranging from 2-12 h were used for ethanol and water (70:30) solvent ratio. The curcumin content found in each extraction time was given in Figure 1c. With an increase in extraction time, the curcumin content is increased and then decreased after a certain time. The maximum curcumin content of 5.02 mg/g was observed during the 8 h of extraction. The change in the extraction pattern may be due to the solvent saturation to extract further. In the batch process, the saturation point of the solvent plays an important role in the extraction of bioactive compounds. For the rest of the experiments, and ethanol solvent to water ratio (70:30) and 8 h of extraction was found to be suitable. A significant difference in the curcumin content is observed between the different solvents.

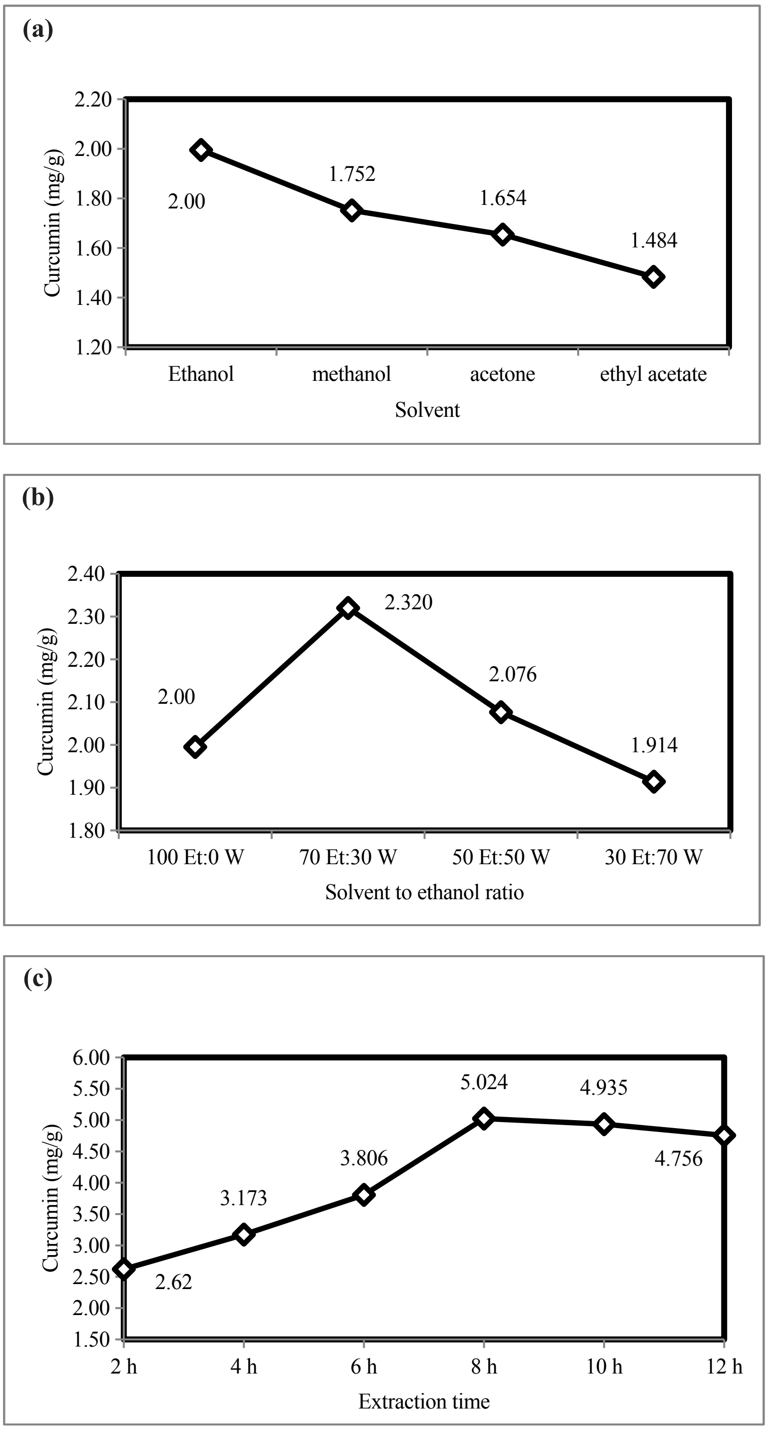

Figure 1. Effect on the curcumin content (a) Different solvents, (b) Solvent to ethanol ratio (ethanol: water), (c) Extraction time

3.2 *Effect of processing conditions on curcumin content*

Three methods were employed to cook the freshly harvested rhizomes for the extraction of curcumin. From Figure 2, it can be observed that the microwave-cooked samples were completely shrunken and wrinkled compared to other samples. The heat generated rapidly by the absorption of electromagnetic waves has completely ruptured and disintegrated the intact tissues of the turmeric. Dandekar and Gaikar [28] reported that electromagnetic heating increased the internal cell wall pressure resulting in rupturing and degradation of the cellulosic cell walls. Monisha et al. [29] observed a significant weight loss in turmeric rhizomes after microwave boiling. The authors have also observed the decrease in the weight of turmeric to increase with an increase in power level and treatment times. The rhizomes that are water boiled was observed to be least affected. There is only slight shrinkage in the cooked samples when compared to pressure cooking and microwave boiling. As the rhizomes were boiled in open vessel without the application of any pressure there is less effect on the rhizome integrity. The cooked rhizomes are dried as per the conditions given in above section 2.2.1, 2.2.2, 2.2.3. The dried samples were then pulverized using a hammer mill, sieved, and stored for further experiments.

Figure 2. Rhizomes boiled using different methods (a & b) before and after pressure cooked, (c & d) before and after microwave cooked, (e & f) before and after water boiled

The rhizomes were dried using a solar dryer and a tray drier (90 °C) till the moisture content reaches below 8-10%. The time taken for the rhizomes to reach the desired moisture level is 6.5 and 16 h in tray dryer and solar dryer respectively. More retention of curcumin was observed in the tray dried samples compared to the solar-dried samples. There are a few reasons that have led to a decrease in the curcumin content of solar-dried turmeric. Raza et al. [30] reported that direct sunlight exposure can significantly reduce curcumin content. The above authors also stated that curcumin is a heat and light sensitive pigment and direct exposure to sunlight significantly decreases the curcumin concentration. Nithya et al. [19] observed a decrease in the curcumin recovery in the solar-dried rhizomes than in the mechanical dried. The longer heating period and non-uniform heat transfer of rhizomes during sun drying have led to photosensitization light reactions that might have decomposed the curcuminoids. Similarly, Geethanjali et al. [31] also stated that curcumin is a light-sensitive bioactive component of turmeric powder. Heger et al. [32] reported that the chromophore group of curcumin absorbs visible light and easily undergoes photochemical decomposition with other phenolic groups. In the presence of UV radiation the chromophore component of the curcumin changes to auxochrome that exhibit brown color [33]. Lee et al. [34] reported that light induced the decomposition of yellow turmeric pigment. A similar observation of light-induced decomposition of curcumin was also reported by Komonsingh et al. [35]. The above authors have also stated that both direct and indirect solar radiation can decompose the curcumin pigment. Raza et al. [30] observed more curcumin retention in hot air-dried samples than the conventional drying and sun drying. However, the drying efficiencies depend on the drying temperature, drying air velocity, and thickness of turmeric placed in the drying cabinet.

Table 1. Effect of different boiling methods on the curcumin content of turmeric

Boiling methods	Curcumin content (mg/g)	
	Solar-dried	Tray dried (90 °C)
Hot water boiled	5.57 ± 0.02^{b}	5.75 ± 0.04^{b}
Pressure boiled	5.88 ± 0.04^{c}	6.18 ± 0.03^{c}
Microwave boiled	2.49 ± 0.04^{a}	2.64 ± 0.01^{a}

All the data are expressed as mean ± standard deviations. Means with the different superscript letters in a column are differ significantly (P < 0.05)

Of the three cooking methods employed, the pressure-cooked rhizomes that were dried using the tray drier were found to have the highest curcumin content (6.18 ± 0.03 mg/g). The curcumin content of different samples ranged from 2.49 ± 0.04 to 6.18 ± 0.03 mg/g. The curcumin content values of different cooked and dried samples are given in Table 1. A significant difference in curcumin content is observed between the cooked samples. Nithya et al. [19] reported that the pressure boiling of turmeric resulted in higher curcumin retention compared to the water boiled. Chandrasekaran et al. [36] observed an increase in the curcumin content in pressure-cooked turmeric (5.21%) compared to water-boiled turmeric (3.94%). The steam developed during the cooking might have loosened the turmeric tissue that has liberated during the solvent extraction. Likewise, the least curcumin content was observed in microwave cooked samples followed by solar drying. The microwave boiling resulted in the decrease in color b* which corresponds to the yellowness that suggests the degradation of curcumin [29]. Similarly, we also found a decrease in the curcumin content due to the longer exposure of turmeric to microwave radiation. Jung et al. [37] observed a reduction in the curcumin content after the microwave treatment. The microwave dried rhizomes showed a low curcumin yield than the tray dried samples [19]. Cortez et al. [38] reported that microwave boiling at 650 W decreased the curcumin content compared to water boiling. Although there are advantages of the microwave process like ease of operation, speed, leaching effect and fewer residues after the treatment resulted in the degradation of curcumin content.

3.3 *Ultrasound-assisted extraction of curcumin*

Compared to the conventional method of extraction, ultrasound-assisted extraction resulted in higher extraction efficiencies. In this study, the pressure-cooked and tray-dried samples were taken to quantify the curcumin content during ultrasound-assisted extraction. The curcumin content was increased from 6.18 ± 0.03 mg/g in the conventional extraction method to 7.801 ± 0.02 mg/g after 25 min of ultrasound-assisted extraction. The curcumin content of 5 min of UAE is more than the highest content observed in conventional extraction. The results of ultrasound-assisted extraction at different time intervals are given in Figure 3. With the increase in the extraction time beyond 25 min, there is a decrease in the curcumin content. The curcumin content was found to be decreased from 7.801 ± 0.02 mg/g to 7.529 ± 0.04 mg/g at 30 min of extraction. So, the optimized time for the maximum extraction of curcumin in 70% ethanol solvent is 25 min at 250 W. A significant increase in the curcumin content was observed in the ultrasound extracted samples. The improvement in the mass transfer rate could be one of the reasons for the enhanced extraction. The explosion of bubbles results in the rupture of intact tissues leading to cell poration that is attributed to easy leaching out of intracellular compounds. The explosion of bubbles results in the micro-jetting of the extraction medium towards the solids leading to cell rupture and cell permeabilization. Mandal et al. [12] reported that the ethanol due to its lower vapor pressure resulted in a larger number of bubble formations leading to improved mass transfer. However, the extraction efficiency depends on the solvent and its proportion, solvent to solids ratio, and power applied. Strieder et al. [39] stated that for enhanced extraction of a few bioactive compounds, the ethanol solvent could be suitable due to the formation of acidified ethanol during ultrasonication. In a similar kind of investigation, Shirsath et al. [11] observed a 9.18 mg/g of curcumin retention with the application of ultrasound. The above authors have also reported that at 250 W powers, the larger amplitude sound waves resulted in the intense collapse of bubbles, an increase in cell disintegration, more solute diffusion, and high local energy dissipation. Binello et al. [15] reported that ultrasound-assisted extraction of curcuminoids is a fast, efficient, and cost-effective method.

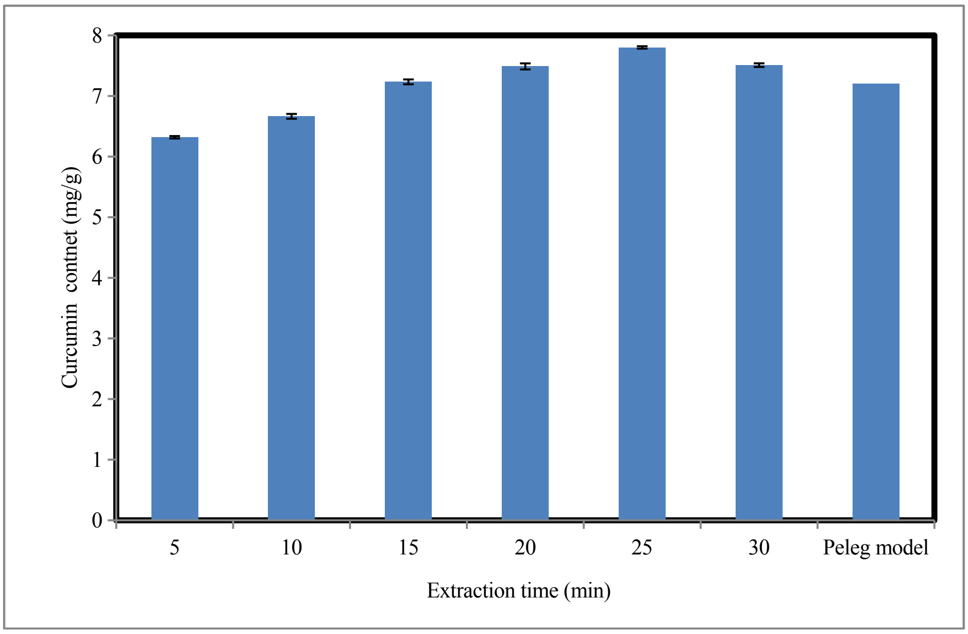

Figure 3. Ultrasound-assisted extraction of curcumin content and peleg's model calculated value

From Peleg's model, the calculated curcumin content was found to be 7.2 mg/g which falls in between the values obtained from the experimental data. The calculated value of curcumin content using Peleg's fitting model is given in Figure 3. The K_1 is related mass transfer rate and the lower values indicate the higher extraction rates. The t/C_t value with respect to the time of extraction is in accordance with the experimental values. The peleg model ($R^2 > 0.9914$) value indicated a good fit for the experimental data. The value of R^2 shows that the model can predict the experimental data with accuracy.

4. Conclusion

Alternative to conventional extraction techniques, ultrasound-assisted extraction has more extraction efficiency. Though a good amount of work has been carried out in the use of UAE of curcumin, the present work has been conducted to study the microwave boiling of turmeric rhizomes and UAE on curcumin extraction. The microwave boiling resulted in greater degradation of curcumin compared to the hot water boiling and pressure cooking. After boiling, the tray dried samples retained more curcumin than the solar-dried samples. The light-induced degradation of curcumin could be the possible reason for the lower values in the solar-dried samples. For the curcumin extraction, ethanol to water ratio (70 V/V) was found to be more suitable for the extraction. The extraction time was optimized by using a shaking water bath and ultrasound to get maximum curcumin extraction. A maximum curcumin yield of 7.801 mg/g was found in 25 min of ultrasound extraction at 250 W. The calculated value obtained from Peleg's model is in agreement with the experimental value. We can conclude that the use of ultrasound as a green technology could be beneficial for the extraction of natural compounds as a cost-effective and lesser extraction time.

Conflict of interest

The authors declare no competing financial interest.

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