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Research paper



Process Development for the Effective Extraction of Curcumin from Curcuma Longa L (Turmeric)

Vinosh Muthukumar P[1]*, Vaishnavi M[1], Theepapriys S[1], Saravanaraj A[1]

[1]*Vel Tech High Tech Dr. Rangarajan Dr. Sakunthala Engineering College, Avadi, Chennai, Tamil Nadu - 600062.*
**Corresponding author E-mail: vinoz.15@gmail.com*

Abstract

Curcumin, the active, light sensitive agent, found in significant amounts in *Curcuma longa L*, is a potential therapeutic agent, possessing various pharmaceutical and industrial uses. The present extraction techniques, that are in application for many decades suffer from many economical and environmental demerits. This work is a prospective and noteworthy step towards conquering those disadvantages by means of optimizing the extraction process parameters and developing an energy efficient, environment friendly and economical process. In this study, various organic solvents were tested for their ability of curcumin extraction an concluded that acetone is most efficient. The process parameters like time, temperature and particle size were investigated and the minimum volume and concentration of solvent required were determined. Three potential pre-treatment process were analyzed for their capability to effectively distort the turmeric structure and aid in increasing the overall efficiency of the process. It was found that, employment of a mechanical distortion method by microwave assistance, and further followed by chemical disintegration, by means of enzymatic treatment, resulted in high attainable efficiency.

Keywords: *Acetone;Curcumin;Extraction;Soxhlet;Turmeric.*

1. Introduction

Curcumin is a hydrophobic poly phenolic compound derived from the rhizome of turmeric, *Curcuma longa L.* (Zingiberaceae), the plant that is considered as a rich source of phenolic compounds, called the curcuminoids. The curcuminoids constitute 4-6% of total turmeric content, of which 2-4% essential, fixed and volatile oils, such as turmerone, atlantone, and zingiberone [2]. However, the remaining constituents of curcuminoids include sugars, proteins and resins. Curcumin ($C_{12}H_{20}O_6$), approximately 0.5-6.0 % by weight of the root of turmeric and about 3-5% of the composition of turmeric, is the principal of the three curcuminoids, the other two being demethoxy curcumin (DMC), and bisdemethoxy curcumin (BDMC) [9]. CUR is the biologically active photochemical compound, primarily responsible for its bright yellow colour, making it a valuable additive in the food industry. it is also widely used in the medicine industry, for its anti-carcinogenic, anti-inflammatory and anti-microbial properties.

India is the largest producer and exporter of turmeric, with a figure of 4,87,000 metric tonnes in production, and 27,750 metric tonnes in export. The production of turmeric, which largely affects the curcumin production, is being regionally dominated by India, making it the largest manufacturer of curcumin, with its production exceeding 80% of global market. Based on its application, global curcumin market may be segmented into pharmaceutical, food and cosmetics, among which, pharmaceuticals industry accounts for a larger share, thus, boosting the growth of global curcumin market. In relation of the escalating demand, the curcumin industry is expected to reach USD 20 million by 2016 and 99.3 million by 2024 [4].

Isolation of curcuminoids is highly influenced by the wide range of biological activity exhibited by the metabolite and the complexity in its structure. Despite curcumin being an astounding panacea, its poor solubility in aqueous solvents, warrants for the development of an highly efficient and enhanced extraction method, with the incorporation of energy saving and environment friendly processes. The recent extraction methods can be categorized as - conventional and modern extraction methods. Conventional method are based on the extracting power of the solvents and it includes, Soxhlets', maceration and hydro distillation. The modern routes include, ultrasonic, microwave and enzyme assisted extraction methods [2].

The most conventional method of extraction, is the Soxhlet's, where the principle of operation is cell permeation followed by solubilizing the active constituents of the cell by the extracting solvent. Here, the solvent penetrates through the tightly packed cork cells, followed by the walls of oleoresin cells, which habitats the metabolite curcumin [8]. The U.S. Department of Health and Human Services Food and Drug Administration (FDA), Center for Drug Evaluation and Research (CDER), Center for Biologics Evaluation and Research (CBER), classified solvents into three categories, such as Class one, Class two and Class three solvents, based on its ability to impart unacceptable toxicity on the desired product and deleterious environmental effect, and thus, majorly impacting and limiting its application in pharmaceutical industries [5]. Table 1,2 and 3 provides the list of various solvents classified as Class I,II and III, in addition to its harmful effect and the concentration they must be restricted to, when used under unavoidable circumstances.

Solvents in Class 1 (Table 1) should not be employed in the manufacture of drug substances, recipients, and drug products because of their intrinsic toxicity and their detrimental effects on the environment. Solvents in Class 2 (Table 2) should be limited in their

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use in pharmaceutical products. Solvents in Class 3 (Table 3) are regarded as less toxic and of lower risk to human health and also includes no solvent known as a human health hazard at levels normally accepted in pharmaceuticals. The usage of higher concentration levels are considered acceptable, when they are used in unison with Good Manufacturing Practices (GMPs) and realistically enhance manufacture capability.

Table 1: CLASS I - Solvents to be avoided in pharmaceutical applications*The stated limit is based on review data

Solvent	Concentration (ppm)
Benzene	2
Carbon tetrachloride	4
1,2-Dichloroethane	5
1,1-Dichloroethane	8
1,1,1-Trichloroethane	1500*

Table 2: CLASS II - Solvents of limited use in pharmaceutical applications

Solvent	**Concentration (ppm)**
Acetonitrile	410
Chlorobenzene	360
Chloroform	60
Cyclohexane	3880
Cumene	70

Table 3: CLASS III - Solvents that can be used in pharmaceutical applications, but to be limited by GMP

Acetic acid	Heptane
Acetone	Isobutyl acetate
Anisole	Isopropyl acetate
1-Butanol	Methyl acetate
2-Butanol	3-Methyl-1-butanol
Butyl acetate	Methylethyl ketone
tert-Butylmethyl ether	Methylisobutyl ketone
Dimethyl sulfoxide	2-Methyl-1-propanol
Ethanol	Pentane
Ethyl acetate	1-Pentanol
Ethyl ether	1-Propanol
Ethyl formate	2-Propanol
Formic acid	Propyl acetate

The above mentioned extraction methodology is severely affected by the disadvantages such as, amount of solvent used, time and temperature necessary for the reaction to occur, and the environmental pollution caused by the overall process [3]. Thus, for the design of an appropriate extraction technique, factors such as high productivity, integrity and selectivity of the solvent towards the target compound, must be taken into prime consideration [7]. The present work spotlights on trouncing the existing disadvantages of using conventional organic solvents to the maximum possible extent, and development of an improved extraction process, by a combination of two or more extraction methodologies in the optimised order, in addition to the conventional Soxhlet extraction, for the efficient extraction of curcumin from turmeric. The critical parameters evaluated include, the choice of solvent, its concentration, and optimal material - solvent ratio, the retention time of the process, temperature, pressure and the particle size of the raw material.

2. Materials and Methods

2.1. Materials

Turmeric, dried rhizome of Curcuma Longa L, purchased from the local market, in Avadi, Tamil Nadu, India, was of high quality whose dark orange colour is a pointer of its high curcumin content. Commercial α-amylase and amylo-glocosidase enzymes, standard curcumin, ethanol, methanol, acetone, 1 - propanol, n - butanol, and other analytical or biochemical - grade solvents and chemical reagents that were used, were all of highest grade and commercially available.

2.2. Turmeric Preparation

The turmeric obtained from the local market was dried in hot air oven at 70 ±3°C or 24 hours and ground with the help of a high speed disintegrator. It was then sieved in a set of brass sieves of mesh number 35 - 7 such that, the particle size ranged from 0.5 mm- 3 mm. The powdered rhizome was then preserved in plastic containers in a cold freezer at 5±2°C and used whenever required.

2.3. Extraction Techniques

2.3.1. Enzyme Assisted Extraction

The enzyme assisted extraction of curcumin as carried out by taking 1 g of turmeric in 250 ml Erlenmeyer flask and mixed with 100 ml water and 50 ml McIlvaine's buffer at pH 5. The enzymes, α - amylase and amyloglucosidase, in the concentration of 4% w/w of turmeric of each of the enzyme, was added to the mixture, and let alone for 8 hours, after which the enzyme solution was separated from the precipitated mixture. The turmeric thus obtained, was dried at 60˚C overnight in a hot air oven and then subjected to Soxhlet's extraction.

2.3.2. Ultrasonic Assisted Extraction

The ultrasonic assisted extraction of curcumin was carried out by taking 1 g of turmeric in 20 ml water in an ultrasonic bath, and subjecting it to different temperatures varying from 25 - 40˚C, for time intervals of 10 - 40 min. During the process, the sample was covered with parafilm in order to prevent undesirable loss of solvent. After the completion of the process, the solvent and the precipitate was separated, and the turmeric thus obtained, was dried at 60˚C overnight in a hot air oven and then subjected to Soxhlet's extraction.

2.3.3. Microwave Assisted Extraction

The microwave assisted extraction of curcumin as carried out by taking 1 g of turmeric in 20 ml water in a microwave chamber. The process was carried out at a power capacity of 250 W, for a time period of 3 minutes, with 30 seconds intervals. The sample was subjected to intermittent microwave irradiation and cooling alternatively, as longer time and high power can cause undesirable boiling of the solvent. After the completion of the process, the solvent and the precipitate were separated, and the turmeric thus obtained, was dried at 60˚C overnight in a hot air oven and then subjected to Soxhlet's extraction.

2.3.4. Soxhlet's Extraction of Curcumin

The turmeric obtained from the above mentioned pre treatment processes was prepared and stored separately, as mentioned in section 2.2 and subjected to Soxhlet's extraction. 10 g ground turmeric powder was weighed and loaded into the Soxhlet apparatus by means of embedding it in a thimble. The desired extraction solvent was then loaded into the apparatus and the process was carried out at 60°C for 8 hours. Upon completion of the extraction, the solvent was separated from the extract using rotary vacuum evaporator at 35 °C and the residue was dried, weighed and taken for analysis.

2.4. Preparation of Solvent

Various organic solvents like methanol, ethanol, acetone, isopropanol and n-butanol were tested for their extraction capacity for curcumin from turmeric. These solvents were selected based on their commercial availability, solubility and their affinity to-

wards curcumin. To determine the minimum concentration of the solvent required for efficient extraction, each solvent was prepared in varying concentrations of 40 – 80 % by weight, and used in the process. Further, to determine a minimum volume of solvent required for efficient extraction and the maximum absorptive capacity of turmeric, the solvents were added in steps of multiple folds and the amount of solvent remaining unabsorbed after a particular time was determined and the results were tabulated.

2.5. Effect of Parameters

Various process parameters viz. time, temperature, and particle size were investigated for their influence on the extraction process and the results were analysed.

2.6. Estimation of Curcumin

The residue obtained after the Soxhlet extraction, was subjected to UV Spectrophotometric analysis. Here, the sample was qualitatively analysed, by taking 1 g of the sample, and mixing it with appropriate solvent and water in ratio of 1:1 and checking the absorbance of the mixture at 420 nm. The standard graph of calibration was separately obtained for each of the solvent tested. From the above results, the extraction efficiency was calculated.

3. Results and Discussions

3.1. Choice of Solvent

To determine the most suitable solvent for the extraction of curcumin, 5 different solvents were tested for its efficiency. Figure 1 shows the extraction efficiency of various solvents and it can be seen that, n-butanol is most effective with a yield percentage of 17.74. Though, the weight of curcumin obtained, when n-butanol was used is higher when compared to other solvents, it holds a disadvantage of having high solubility making the down streaming process more difficult. The next in order of extraction efficiency is acetone, (Yield = 17.14%), which has a lower boiling point and is more economical when compared to all other solvents [1]. Thus, acetone is selected as best suitable solvent for further analysis.

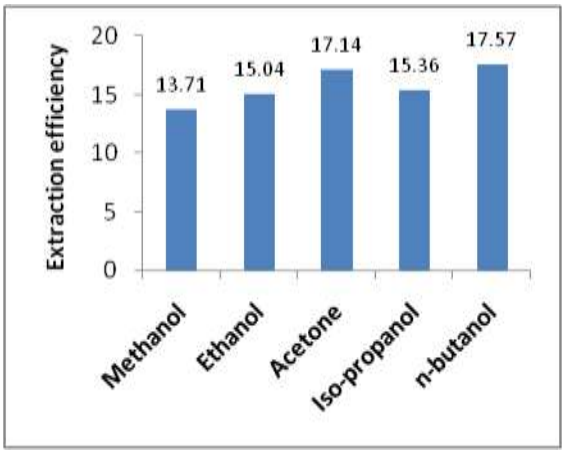


Fig 1: Extraction efficiencies of various solvents for extraction of Curcumin from *Curcuma Longa L*

3.2. Minimum Concentration and Volume of Solvent

To determine the minimum concentration of acetone required for effective extraction, solvent was prepared as specified in section 2.4 and the results obtained are shown in the figure 2. It can be seen that, maximum efficiency for extraction of curcumin was obtained at a concentration level of 80% acetone. This is due to the fact that, curcumin is least soluble in water [6], and as the concentration of water in the solvent decreases, the extraction efficiency increases. Also, water has a prime effect on the porosity and hardness of the turmeric.

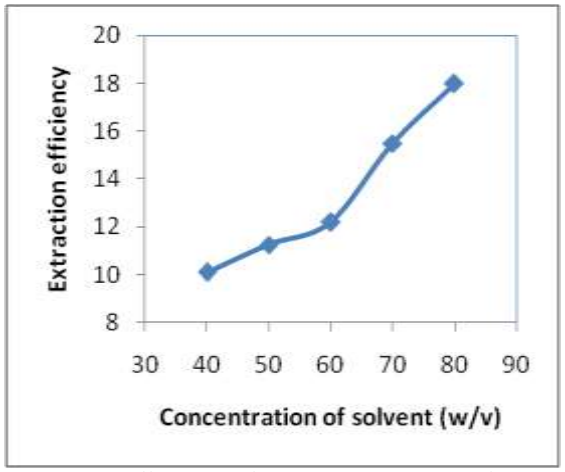


Fig 2: Extraction efficiencies of various concentrations of acetone for extraction of Curcumin from *Curcuma Longa L*

To determine the minimum volume of solvent required for the extraction of curcumin, ten folds excess of 80% acetone (w/v of turmeric) were added and the maximum absorptive capacity of turmeric was determined after a time interval of 30 min. Figure 3 represents the volume of solvent remaining across the time range of 10 - 60 minutes. It can be seen that, after a time of 40 minutes, the

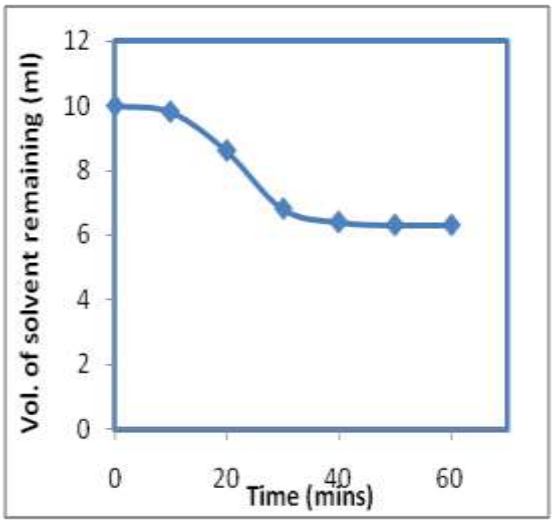


Fig 3: Volume of acetone remaining with time

Volume of the solvent remaining is almost constant thereafter. Thus, it can be inferred that, by 40 minutes, turmeric had fully imbibed in the solvent and the absorbed volume of solvent at this point provides the maximum absorptive capacity of turmeric, and was found to be in the ratio of 1:3.6. Further, usage of solvent more than this volume is unnecessary and futile.

3.3. Effect of Critical Parameters

During the investigation of critical parameters, each parameter was optimised individually, and the results were analysed.

3.3.1. Effect of Particle Size

The extraction efficiency was calculated for different particle sizes varying from 0.5 – 2.83 mm and the results were represented in figure 4. It can be seen that, as the particle size decreases, the efficiency increases, and this may be due to the fact that, disintegra-

tion of turmeric is more profound at very small particle size and the extraction process as a whole, for larger particles is governed by particle mass transfer. Also, the small structure enables the diffusion of solvent in to the turmeric matrix.

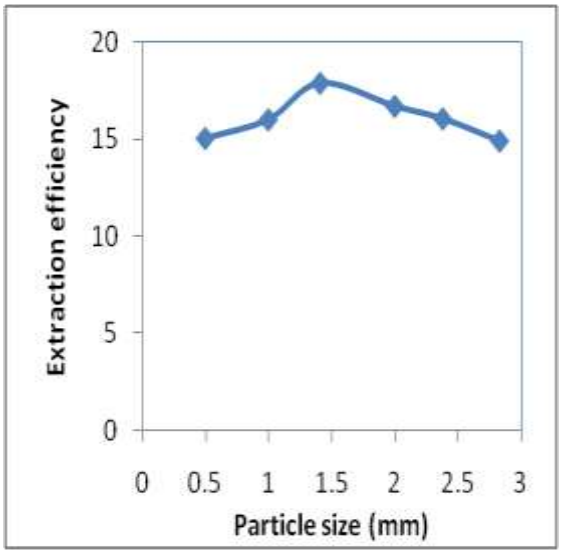


Fig 4: Effect of particle size on the extraction of Curcumin from *Curcuma Longa L*

3.3.2. Effect of Temperature

The effect of temperature on extraction efficiency of curcumin was investigated over the temperature range of 25 – 65 ˚C and the results are represented in figure 5. It can be inferred that, high extraction efficiencies are achieved at moderate temperature of 35˚C, which is due to the fact that, high temperatures promote high diffusion rates which decreases the extraction process from turmeric.

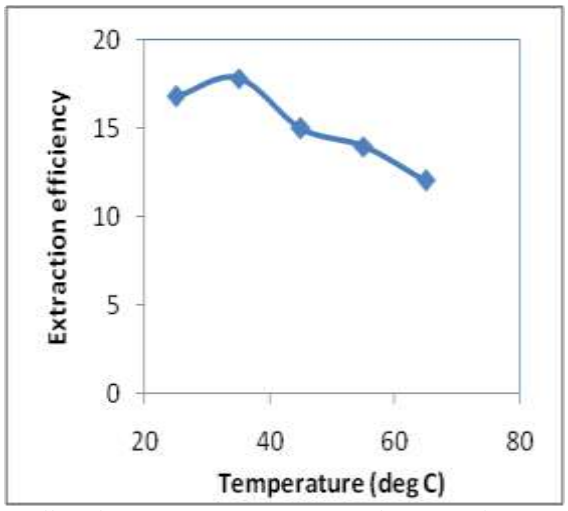


Fig 5: Effect of temperature on the extraction of Curcumin from *Curcuma Longa L*

3.3.3. Effect of Time

The extraction efficiency of acetone for curcumin, for different lengths of time was tested and the results (Figure 6) indicated that, the extraction efficiency increases with time, though prolonged periods of time, did not have any significant effect on the extraction efficiency. This is due to the fact that, long periods of extraction time and difference in equilibrium time, is caused by the polymerisation, solubility and the interaction between the poly phenolic compounds causing degradation.

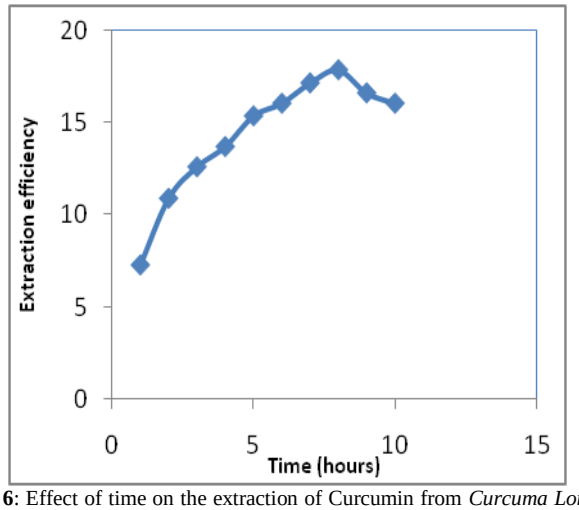


Fig 6: Effect of time on the extraction of Curcumin from *Curcuma Longa L*

3.4. Process Development

To develop a process for efficient extraction of curcumin from turmeric using acetone, three pre-treatment methods were considered and each was employed separately prior to the Soxhlet extraction. The efficiency of extraction was calculated at each stage and the results are tabulated (table 4).

Table 4: The extraction efficiencies after the pre treatment (Stage 1) and after Soxhlet extraction (Stage 2)

Pre treatment process	η_{ext} (Stage1)	η_{ext} (Stage 2)	Difference in η_{ext}
NIL	-	17.89	0
Microwave assisted extraction	10.5	20.25	13.19
Ultrasonic assisted extraction	9.28	23.45	31.08
Enzyme assisted extraction	11.23	28.56	59.64

It can be inferred that, the enzyme assisted pre - treatment provided the maximum efficiency for extraction of curcumin. The highest attained efficiency was 28.56 percent, which was 59.64 percent higher than the efficiency attained when no pre-treatment was used. Further, when a microwave assisted pre - treatment step was added prior to the enzyme activity, it was found that, the extraction efficiency advanced to 39.40 percent. This efficiency of the overall process, after the addition of a second pre treatment step was 38 percent higher than the obtained with a single pre treatment step and 5.3 times better than the one where no pre-treatment was employed. This is due to the fact that, when the turmeric sample undergoes microwave treatment in the first stage, mechanical disintegration of the cell wall takes place. Further, when the same sample is subjected to enzyme treatment in the second stage, chemical dissolution of the turmeric wall and the surrounding cork cells happens, resulting in complete distortion of the structural barrier posed by the turmeric, aiding nearly complete cell permeability. Thus, Soxhlet's, when employed as the third stage of extraction, the solvent vapours gain free unperturbed entry, and the extraction process occurs at the highest possible efficiency.
Figure 7 gives a block diagram for the extraction of curcumin from the rhizome turmeric.

4. Conclusion

The obtained results showcase the potential of different organic solvents for the extraction of curcumin form *Curcuma longa L*. Among them, it was concluded that, acetone was as the most economical, easily available and handle-able and the most efficient solvent for curcumin extraction. The minimum volume and minimum concentration of the solvent required for the extraction process were obtained and the optimal material to solvent ratio was

arrived at, soaring the prospective of an economically stable and reliable process. Further, the critical parameters of the extraction process viz., time, temperature and particle size, were optimised, to make the process more sustainable and beneficial. The process was furthermore optimised by means of adding a microwave assisted and an enzyme assisted pre-treatment step, prior to the Soxhlet's extraction which increased the overall efficiency of the process 5.3 times.

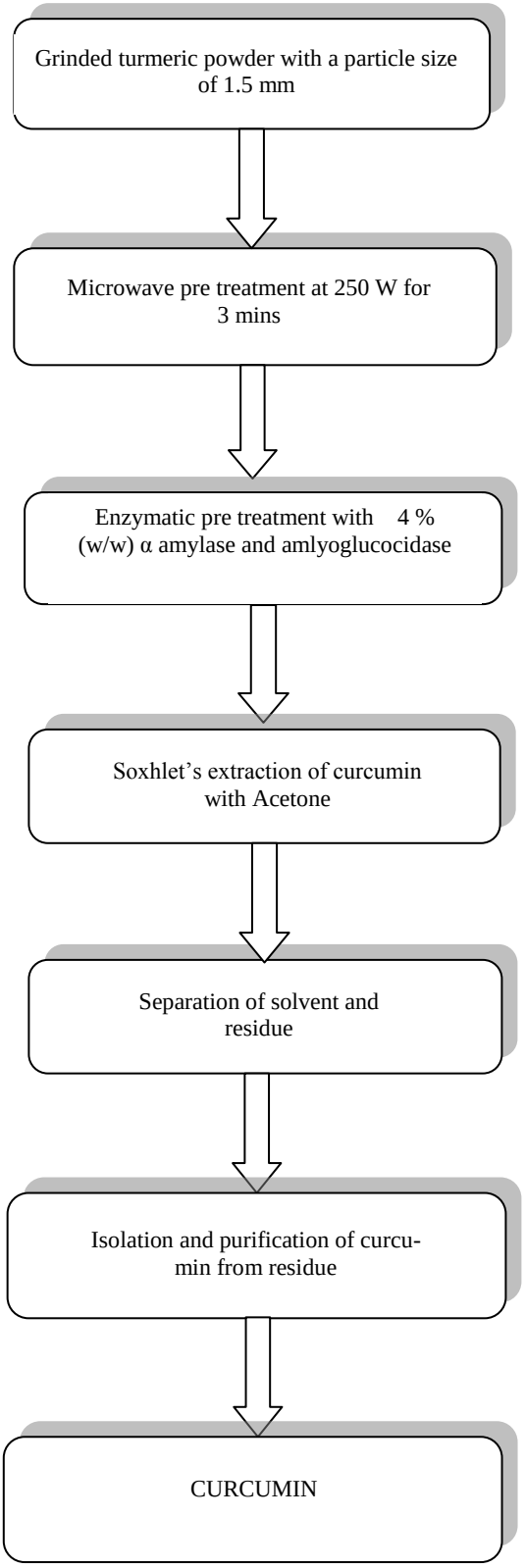


Fig. 7: Block diagram for the extraction of Curcumin from *Curcuma longa L*

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