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Technical factors affecting to extraction and drying of curcumin from turmeric (*Curcuma longa*)

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Abstract.

Curcumin is a potent bioactive compound present most commonly in the roots Curcuma longa plant, commonly known as turmeric plant. Traditionally curcumin is well known for its nutritional factors and medicinal values. Different aspects affecting to curcumin extraction were examined including blanching, solvent, solvent: solid, extraction time and temperature under ultrasound, carrier and temperature for drying. Optimal results were clearly shown that the turmeric should be blanched under 95^{o}C in 10 seconds; extraction should be conducted by ethanol 90%, ethanol: solid (2.0:1.0), 60^{o}C, 8 minutes; vaccum drying should be executed at 60^{o}C with the support of carrier (0.5 maltodextrin: 0.5 gelatin: 1.0 turmeric fluid). Ultrasonic had positive effect on curcumin extraction and this method could be utilized to obtain the most curcumin powder.

Keywords: *Turmeric, curcumin, ultrasonic, blanching, solvent, extraction, carrier, drying*

I. INTRODUCTION

Turmeric (Curcuma longa) belongs to the family *Zingiberaceae*. The most important part of the turmeric tuber is a group of bioflavonoids, i.e. curcumins (curcumin (77%), bisdemethoxycurcumin and demethoxycurcumin). The most common method for the isolation of curcumin is extraction with organic solvents, usually ethanol, by using Soxhlet, ultrasonic and microwave extraction, and more recently, due to increased use in the food industry (food supplements), triacylglycerol. Curcumin has significant anti-inflammatory, antioxidant, chemoprotective, anticancer, and gastroprotective properties (Ramirez-Bosca A et al., 1995; Zhang D et al., 2011; Helal MH et al., 2014; Prasad S et al., 2014; Boyanapalli SS, Kong AN, 2015; Zhang Y et al., 2015; Daria Jovičić et al., 2017; Gouda MM et al., 2017).

Curcumin is extracted from the tubers of a dried and milled turmeric by using a suitable solvent. The solvents which can be used for the extraction are: isopropanol, ethyl acetate, acetone, and ethanol. Curcumin is soluble in oils, insoluble in water at acidic and neutral pH, but soluble in alkaline conditions. The color of curcumin highly depends on the pH, at pH 2.5 to 7.0, it has an intense yellow color, and above pH 7.0 it is red. It is primarily used as an additive for coloring products. As an additive, curcumin is stable during thermal treatment and in dry food. Besides being used for food coloring, curcumin shows significant antioxidant properties and it prevents lipid peroxidation to a significant extent. The antioxidant properties are the result of the double carboxyl groups along with hydroxyl groups. Curcumin binds free radicals, and it becomes a short-lived, nonreactive free radical, and as such does not represent a health hazard. Commercially, turmeric can be sold either fresh or as dried powder. Dried turmeric powder is more often sold all around the world but the price varies depending on many factors including quality—ie moisture content, appearance (color), and phenolic contents (Jose and Joy 2009).

Sathira Hirun et al., (2014) investigated the effect of microwave power and drying times on the quality of dried turmeric in terms of colour, moisture content, water activity (aw), ash, antioxidant activity, total phenolic and curcuminoids content. Natural colorants from plant-based materials have gained increasing popularity due to health consciousness of consumers. Among the many steps involved in the production of natural colorants, pigment extraction is one of the most important. Soxhlet extraction, maceration, and hydrodistillation are conventional methods that have been widely used in industry and laboratory for such a purpose. Recently, various non-conventional methods, such as supercritical fluid extraction, pressurized liquid extraction, microwave-assisted extraction, ultrasound-assisted extraction, pulsed-electric field extraction, and enzyme-assisted extraction have emerged as alternatives to conventional methods due to the advantages of the former in terms of smaller solvent consumption, shorter extraction time, and more environment-friendliness. Prior to the extraction step, pretreatment of plant materials to enhance the stability of natural pigments is another important step that must be carefully taken care of (Luxsika Ngamwonglumlert et al., 2017).

Natural colorants have the potential to be used as acceptable additives in foods as they are natural and they also have potential health benefits. However, natural pigment compounds usually have poor stability as compared to artificial food colorants, which hinders their usage. In the present study, we examined different parameters influencing to curcumin extraction such as blanching, solvent, solvent: solid, extraction time and temperature under ultrasound, carrier and temperature for drying

II. Materials And Method

2.1 Material

We collected turmeric in Mekong river delta, Vietnam. They must be cultivated following VietGAP to ensure food safety. After harvesting, they must be conveyed to laboratory within 8 hours for experiments. They were washed thoroughly under turbulent washing to remove dirt, dust and adhered unwanted material. Besides turmeric we also used other materials during the research such as ethanol, acetone, ethyl acetate, maltodextrin, gelatin, DMSO. Lab utensils and equipments included oven, ultrasonicator, vaccum dryer, centrifugator, water bath.

Figure 1. Turmeric (*Curcuma longa*)

2.2 Researching procedure

2.2.1 Effect of blanching on curcumin extraction recovery (%)

Different blanching conditions were performed (100^{o}C, 5 sconds; 95^{o}C, 10 seconds; 90^{o}C, 15 seconds; 85^{o}C, 20 seconds) to verify the effectiveness (% recovery) of blanching to curcumin extraction.

2.2.2 Effect of different solvents on curcumin extraction recovery (%)

Different solvents (water, ethanol, acetone, ethyl acetate) were used to examine the effectiveness (% recovery) of curcumin extraction

500 g of dried turmeric is first grinded in a mixer grinder and then subjected to extraction. 2 g of sample is taken and mixed with 30 ml of solvents and 30 ml of water respectively, separately and then filtered. The concentration of each of the filtrate is kept same and then the absorbance is measured using spectrophotometer at 425nm. Curcumin content g/100 g is measured using this formula: (0.0025 x Absorbance at 425 nm x volume made up x Dilution factor x 100)/ (0.42 x weight of sample x 1000)

Since 0.42 absorbance at 425 nm =0.0025 g of curcumin (Anamika Bagchi, 2012).

2.2.3 Effect of solvent ratio: material on curcumin extraction recovery (%)

Different ratios of solvent: solid (1.0:1.0, 1.5:1.0; 2.0:1.0; 2.5:1.0; 3.0:1.0) were verified to demonstrate the effect of ratio between solvent and material on the effectiveness (% recovery) of curcumin extraction

Three sets of mixed solvents were used with 1.0:1.0, 1.5:1.0; 2.0:1.0; 2.5:1.0; 3.0:1.0 ethanol in ratio with water, keeping the total volume at 30 ml of each set constant respectively. The amount of curcumin extracted was again calculated using the gravimetric method mentioned earlier for the two different set of water and ethanol while another approach was taken into account for the mixed solvents. The filtrate from the mixed solvents were taken 10 ml each, same as that for the pure solvents, and were left to evaporate the ethanol in water bath and subsequently transferring the petriplates into hot air oven at 130°C for 1.5 hours and then repeating the previously mentioned method again (Anamika Bagchi, 2012).

2.2.4 Effect of extraction temperature by ultrasonic combination with solvent on curcumin extraction recovery (%)

Different temperature conditions (30^{o}C, 40^{o}C, 50^{o}C, 60^{o}C, 70^{o}C) under ultrasonic (37 kHz) combined solvent were examined to prove the effectiveness (% recovery) of curcumin extraction

2.2.5 Effect of extraction interval by ultrasonic combination with solvent on curcumin extraction recovery (%)

Different time intervals of ultrasonic treatment (2 minutes, 4 minutes, 6 minutes, 8 minutes, 10 minutes) were investigated to verify the effectiveness (% recovery) of curcumin extraction. 2g of sample is taken for further solvent extraction of turmeric using separately for solvents ethanol and water respectively. The interval of time being optimized to 2 minutes, 4 minutes, 6 minutes, 8 minutes, 10 minutes respectively. The amount of curcumin being extracted is then calculated using gravimetric method. After filtration 10 ml filtrate is taken from each set, using water as a solvent, and dried in hot air oven at 130°C for 1.5hours in a petriplate, where the weight of each empty petriplate was initially noted down. After taking them out of the oven they were kept in the dessicator to cool down and were measured until constant weight was obtained. Similarly for the set of ethanol extractives of curcumin, 10 ml of filtrate was taken and the solvent was evaporated under atmospheric pressure in water bath. The weight of petriplate with residual curcumin was noted down and the amount being extracted was calculated (Anamika Bagchi, 2012).

2.2.6 Effect of ratio of carrier for drying to curcumin content in powder

Different ratios of carrier: turmeric (1 maltodextrin: 1 turmeric; 1 gelatin: 1 turmeric; 0.5 maltodextrin: 0.5 gelatin: 1 turmeric) were demonstrated to show the effectiveness of drying to curcumin recovery (%).

2.2.7 Effect of drying temperature to curcumin content in powder

Vaccum drying was applied at different temperature (50^{o}C, 55^{o}C, 60^{o}C, 65^{o}C, 70^{o}C) to the final moisture content of dried powder 6 ± 1%

2.3 Physico-chemical and biological analysis

Curcumin analysis (µg/g) analysis was performed through HPLC method by Shashidhar MD et al., (2018). The standard curcumin was purchased from sigma Aldrich. The stock solution 10mg/10ml was prepared and was stored in amberware bottles. Aliquots of curcumin 20, 40, 60, 80 and 100 ppm were prepared. The HPLC system included 2 LL 20 AT solvent delivery system system (dual pump), a SPD-20A UV/Vis detector and a 7725i rheodyne injector with a 20 µL loop volume coupled with CBM 20 Alite system controller. Separation was achieved using Luna Su C18 column. The solvent system consisted of 50mM potassium dihydrogen orthophosphate (3.5 pH): Acetonitrile (40:60 v/v) was pumped isocratically at a flow rate of 0.8mL/min. The detection was carried out using a SPD -20A UV/Vis detector.

2.4 Statistical analysis

The experiments were run in triplicate with three different lots of samples. Data were subjected to analysis of variance (ANOVA) and mean comparison was carried out using Duncan's multiple range test (DMRT). Statistical analysis was performed by the Statgraphics Centurion XVI.

III. Result & Discussion

3.1 Phytochemical composition in Turmeric (*Curcuma longa*)

Phytochemical composition (curcumin) in raw turmeric was analyzed. Result was clearly depicted in table 1 representing that turmeric was suitable for utilization to collect this healthy pigment.

3.2 Effect of blanching on curcumin extraction recovery (%)

Different blanching conditions were performed (100^{o}C, 5 seconds; 95^{o}C, 10 seconds; 90^{o}C, 15 seconds; 85^{o}C, 20 seconds) to verify the effectiveness (% recovery) of blanching to curcumin extraction. Results were depicted in table 2.

K. Pradeep et al., (2016) studied the influence of blanching and drying methods on the quality characteristics of fresh turmeric (Curcuma longa L.) Rhizomes. Different drying methods were investigated for dehydration of turmeric rhizomes (Curcuma longa L.) for getting good colour and quality. Sun drying normal [SDN, 30-37^{0}C and 30-35% relative humidity (RH)], sun drying coupled with black surface (SDB, 38-60^{0}C and 28-31% RH), hot air (HA, 50±2^{0}C and 58-63% RH), and low humidity air (LHA, 50^{0}C and 28-30% RH) were explored for their drying efficiency of turmeric rhizomes. The quality of unblanched sliced rhizomes dried under HA was superior based onphysico-chemical and CIE colour. SDB was energy efficient due to faster drying. Turmeric oil from blanched and unblanched rhizomes consisted of tumerone, 61.54 and 62.91%; curlone, 27.77 and 25.35%; and cyclohexane, 1.73 and 1.32%, respectively. The HPLC of turmeric oleoresin had curcumin 81.7%, and its analogues de-methoxy curcumin, 12.9% and bis-demethoxy curcumin 5.4%. Among all treatments, hot air dried, unblanched samples were superior followed by SDB drying which was energy efficient.

3.3 Effect of different solvents on curcumin extraction recovery (%)

Different solvents (water, ethanol 90%, acetone, ethyl acetate) were used to examine the effectiveness (% recovery) of curcumin extraction. Results were elaborated in table 3.

Bagchi Anindya et al., (2015) extracted curcumin by different solvents (methanol, chloroform, acetone). They found that chloroform: methanol (95:5) was suitable for curcumin extraction. This finding was also similar to data collected by Gokhul. V et al., (2015).

Shital Patil et al., (2018) proved that among the different solvent (ethanol, methanol, acetone, ethyl acetate) used for extraction, ethanol shows maximum extraction yield of curcumin than other solvents

3.4 Effect of solvent ratio: material on curcumin extraction recovery (%)

Different ratios of solvent: solid (1.0:1.0, 1.5:1.0; 2.0:1.0; 2.5:1.0; 3.0:1.0) were verified to demonstrate the effect of ratio between solvent and material on the effectiveness (% recovery) of curcumin extraction. Results were elaborated in table 4.

Shital Patil et al., (2018) examined different ratio of solute and solvent (1:2, 1:5 and 1:10) was used for extraction and its effect on curcumin yield was recorded. The solute to solvent ratio was 1:10, highest yield were obtained, whereas it was lesser when the ratio was 1:2.

Table 1. Phytochemical composition in Turmeric (*Curcuma longa*)

Major composition in turmeric	Curcumin
Value (mg/g)	5.41±0.02

Table 2. Effect of blanching to curcumin extraction recovery (%)

Blanching	100^{o}C, 5 seconds	95^{o}C, 10 seconds	90^{o}C, 15 seconds	85^{o}C, 20 seconds
Curcumin extraction recovery (%)	74.22÷0.01^b	76.48÷0.02^a	70.11÷0.02^c	67.44÷0.02^d

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 3. Effect of different solvents on curcumin extraction recovery (%)

Solvent	Water	Ethanol 90%	Acetone	Ethyl acetate
Curcumin extraction recovery (%)	44.19÷0.01^d	79.47÷0.02^a	74.29÷0.01^c	76.83÷0.02^b

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 4. Effect of solvent ratio: material on curcumin extraction recovery (%)

Solvent: solid	1.0: 1.0	1.5: 1.0	2.0: 1.0	2.5: 1.0	3.0: 1.0
Curcumin extraction recovery (%)	66.39÷0.02^e	68.41÷0.01^d	81.67÷0.03^a	79.11÷0.01^b	71.22÷0.02^c

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 5. Effect of extraction temperature by ultrasonic combination with solvent on curcumin recovery (%)

Extraction temperature (^oC)	30^oC	40^oC	50^oC	60^oC	70^oC
Curcumin extraction recovery (%)	69.36÷0.02^e	74.22÷0.03^d	78.41÷0.01^c	83.44÷0.01^a	81.19÷0.02^b

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 6. Effect of extraction interval by ultrasonic combination with solvent on curcumin recovery (%)

Extraction time (minutes)	2	4	6	8	10
Curcumin extraction recovery (%)	74.12÷0.03^d	78.04÷0.02^c	81.49÷0.01^b	86.02÷0.02^a	86.17÷0.02^a

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 7. Effect of ratio of carrier for drying

Carrier: Turmeric	1 maltodextrin: 1 turmeric	1 gelatin: 1 turmeric	0.5 maltodextrin: 0.5 gelatin: 1 turmeric
Curcumin drying recovery (%)	81.37÷0.01^c	84.11÷0.01^b	89.23÷0.02^a

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

Table 8. Effect of drying temperature on curcumin recovery (%)

Drying temperature (^oC)	50	55	60	65	70
Curcumin drying recovery (%)	85.06÷0.02^d	88.12÷0.03^b	91.15÷0.01^a	87.32÷0.02^c	84.11÷0.02^e

Note: the values were expressed as the mean of three repetitions; the same characters (denoted above), the difference between them was not significant (α = 5%).

3.5 Effect of extraction temperature by ultrasonic combination with solvent on curcumin extraction recovery (%)

Different temperature conditions (30^{o}C, 40^{o}C, 50^{o}C, 60^{o}C, 70^{o}C) under ultrasonic (37 kHz) combined solvent were examined to prove the effectiveness (% recovery) of curcumin extraction. Results were elaborated in table 5.

Eugenio Torres Rodríguez et al., (2014) showed the use of ultrasound in the extraction of curcumin from its natural source. Shital Patil et al., (2018) proved that ultrasound assisted extraction of curcumin. The experiments were carried out in a high-intensity probe system of 200 W and 33 kHz. Ultrasonic probe was immersed in samples and placed in water bath to keep constant temperature. Sample was treated with different ultrasound power i.e. 25, 50 and 75 W powers for 30 min and their effect on extraction yield was monitored. Highest yield was obtained when the ultrasonication power was 25 w.

3.6 Effect of extraction interval by ultrasonic combination with solvent on curcumin extraction recovery (%)

Different time intervals of ultrasonic treatment (2 minutes, 4 minutes, 6 minutes, 8 minutes, 10 minutes) were investigated to verify the effectiveness (% recovery) of curcumin extraction. Results were elaborated in table 6.

Binta Hadi et al., (2015) optimised extraction parameters for ultrasonic-assisted extraction (UAE) with aqueous extraction solvent for curcuminoids were amplitude of 100, particle size of 0.30–0.60 mm, extraction time of 20 min, extraction solvent volume of 10 mL and extraction temperature of 60 °C. The applications showed remarkable improvements in terms of reduced extraction time, solvent consumption, extraction yield and the quality of extracts. The turmeric oleoresin was successfully solubilised in aqueous solution by forming inclusion complex with methyl-β-cyclodextrin (Mβ-CD). Phase solubility studies used curcumin as a marker compounds to represent turmeric oleoresin. In the presence of Mβ-CD, the curcumin was enhanced. Foozie Sahne et al., (2016) showed that the curcumin extraction yield using Soxhlet method (6.9%) was considerably higher than those obtained from microwave-assisted (3.72%), ultrasound-assisted (3.92%) and enzyme-assisted (4.1%) extractions. Zaibunnisa Abdul Haiyee et al., (2016) compared the supercritical fluid extraction and ultrasonic assisted extraction of curcumin. Ultrasonic assisted extraction was able to produce significantly the highest yield (6.40 %, dry weight basis) and the highest curcuminoids concentration (0.1020 mg/100 g). Kimthet Chhouk et al., (2017) used ultrasonic assisted supercritical carbon dioxide (USC-CO_2) to extract curcumin from turmeric and compared to conventional method. The effect of operating conditions on extraction, including temperature (40 - 60^{o}C), pressure (15 - 25 MPa), extraction time (30 - 120 min), CO_2 flow rate (2 - 4 mL/min) and percentage of cosolvent (10 - 20% v/v) were also studied. The result shows that the high extraction yield of 7.17% w/w and curcumin content of 1.69% w/w were achieved at temperature of 50^{o}C, pressure of 25 MPa, extraction time of 90 min, CO_2 flow rate of 3 mL/min with 10% cosolvent. Compared to conventional method, USC-CO_2 could provide higher curcumin content in extraction yield in a shorter extraction time.

3.7 Effect of ratio of carrier (maltodextrin) for drying

Different ratios of carrier: Turmeric (1 maltodextrin: 1 turmeric; 1 gelatin: 1 turmeric; 0.5 maltodextrin: 0.5 gelatin: 1 turmeric) were demonstrated to show the effectiveness of drying to curcumin recovery (%). Results were elaborated in table 7.

D.M. Cano-Higuita et al., (2015) investigated the effects of different formulation wall materials (gum arabic, a binary mixture of maltodextrin and modified starch, and a ternary mixture of gum arabic, maltodextrin and modified starch) and different drying methods (for spray and lyophilization) on the stability of microcapsules of turmeric oleoresin. The drying method affected retention curcumin powder in the drying process and storage of the microcapsules under incident light. Curcumin retention during lyophilization was greater than spray drying, but showed the opposite behavior during storage; spray-dried capsules had a higher retention of curcumin after 8 weeks under light exposure. As a result, the ternary mixture of gum arabic, modified starch and maltodextrin was more effective to prevent loss of curcumin and color changes in the microcapsules.

Deivis de Moraes Carvalhd et al., (2015) prepared a suspension with curcumin nanoparticles in tween 80, the testing of pure curcumin solubility and of a simple mixture of curcumin with tween 80 and nanosuspension in water and ethanol as solvents, and fnally the assessment of the antioxidant activity.

A.A. Ochoa et al., (2016) develop a curcumin nanoemulsion by ultrasonication, containing a high curcumin load, small droplet size and good physical stability. The composition and preparation method effects on entrapment efficiency, droplet size, polydispersity index, and zeta potential of the nanoemulsions were evaluated. Curcumin nanoemulsions were successfully prepared by combined thin-film hydration emulsification and ultrasonication methods, employing 50 % of glycerol in the aqueous phase, and 10 % of soybean lecithin as emulsifier; at 20 % amplitude for 12 min in the sonicator. Nanoemulsions with 2.5 mg curcumin per g, 100 % entrapment efficiency, mean droplet size of 108 nm, and stable for 120 days at 4°C were obtained.

Tri Yuni Hendrawati et al., (2017) studied the influence of concentration maltodekstrin to rendemen, the water level, and time the solubility of flour turmeric by using dryer spray (spray blow dryer). Variation composition maltodekstrin used consisting of 6 %, 8 %, 10 %, 12 %, 14 % at the temperature inlet spray blow dryer 120^{o}C. A method of making flour saffron done by means of the pollen from saffron in grated, then sari saffron obtained added maltodekstrin with a variety composition and in stirring use homogenizer that homogeneous. We do drying in spray blow dryer at the temperature inlet 120^{o}C.

3.8 Effect of drying temperature and time

Vaccum drying was applied at different temperature (50^{o}C, 55^{o}C, 60^{o}C, 65^{o}C, 70^{o}C) to the final moisture content of dried powder 6 ± 1%. Results were elaborated in table 8.

Rodrigo Molina et al., (2013) evaluated the effects of the spray drying on curcuminoid and curcumin contents, antioxidant activity, process yield, the morphology and solubility of the microparticulated solid dispersion containing curcuma extract using a Box Behnken design. The microparticles were spherical in shape, and an increase in outlet temperature from 40 to 80 °C resulted in a significant increase in the yield of microparticles from 16 to 53%. The total curcuminoid content (17.15 to 19.57 mg/g), curcumin content (3.24 to 4.25 mg/g) and antioxidant activity (530.1 to 860.3 μg/mL) were also affected by the spray drying process. The solubility of curcuminoid from *C. longa* remarkably improved 100-fold in the microparticles, confirming the potential of the ternary solid dispersion technique to improve the dyeing and nutraceutical properties of these compounds.

D. S. Aniesrani Delfiya et al., (2014) reported on the microencapsulation of turmeric oleoresin by spray drying using different ratios of maltodextrin and gum arabic (100:0–0:100) as wall material. Solvent-extracted and commercial oleoresin microcapsules prepared using gum arabic alone as wall material and spray dried at the inlet air temperature of 175C showed higher encapsulation efficiency of 71.74 and 73.67%, maximum curcumin of 3.41 and 3.39 g/100 g and oleoresin content of 7.16 and 7.29 g/100 g, respectively. Stability of microcapsules against light, oxygen and heat was higher than the nonencapsulated oleoresin.

Aura Y. Coronel-Delgado et al., (2017) evaluate the operation of spray drying to obtain powder curcumin from turmeric extract blended with maltodextrin. An experimental design (central composite design) with two statistical factors was used. These factors were the inlet air temperature (140-160ºC) and outlet air temperature (75-95ºC), with the rotation speed kept constant (28000 RPM). Statistical optimization was established by considering the response surfaces analysis where the hygroscopicity was minimized and the curcumin concentration was maximized. The optimal conditions for the spray drying process were inlet and outlet air temperatures of 149ºC and 75ºC, respectively, at an atomization speed of 28000 RPM. These settings provided the following results: hygroscopicity (11.71% ± 0.03) and fnal concentration of curcumin (9.03 ± 0.44 mg/g), which were statistically signifcant.

IV. CONCLUSION

Nutritional intake of curcumin could be effective against several problems such as cellular microbial infection, oxidation, free-radical scavenging, inflammation and cancer. We have successfully optimized different parameters influencing to curcumin extraction such as blanching, solvent, solvent: solid, extraction time and temperature under ultrasound, carrier and temperature for drying.

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