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Parametric Evaluation of Microwave-Assisted Extraction of Phenolics from *C. domestica* Val.

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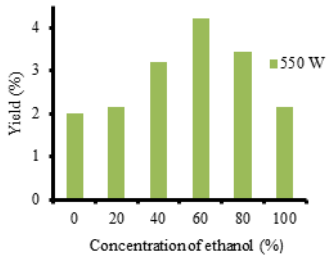
Graphical abstract



Abstract

Conventional methods for the extraction of natural products from plant are characterised by the consumption of large volumes of solvent, energy, lengthy extraction procedures and the potentially deleterious degradation of labile compounds. In the last two decades there has been an increasing demand for new extraction techniques, amenable to automation, with shortened extraction times, reduced organic solvent consumption, prevention pollution in analytical laboratories and reducing sample preparation costs. Unmodified domestic microwave oven is used in the extraction of *phenolics* from *C. domestica*Val. using water as solvent. The Microwave Assisted Extraction produced a better yield of crude extract of 48 mg compared to Soxhlet extraction methods with only an extraction yield 3.4 mg. Effects of extraction time, microwave power and solvent volume are evaluated on the extraction of phenolic compounds.

Keywords: Microwave assisted extraction; extraction; phenolics; Soxhlet; *C. domestica* Val.

Abstrak

Kaedah konvensional untuk pengekstrakan hasil semula jadi daripada tumbuhan dikategorikan dengan penggunaan sejumlah besar pelarut, tenaga, prosedur pengekstrakan yang lama dan berpotensi merosak kan sebatian labil. Dalam dua dekad yang lalu terdapat peningkatan permintaan untuk teknik pengekstrakan baru sesuai untuk automasi, dengan masa pengekstrakan dipendekkan, pengurangan penggunaan pelarut organik, pencegahan pencemaran dalam makmal analisis dan pengurangan kos penyediaan sampel. Ketuhar mikrogelombang domestik tanpa ubah digunakan dalam pengekstrak sebatian fenol daripada *C. domestica* Val. menggunakan air sebagai pelarut. Pengekstrakan bantuan mikrogelombang menghasilkan ekstrak mentah yang lebih baik, 48 mg, berbanding kaedah pengekstrakan Soxhlet dengan hasil pengekstrakan hanya 3.4 mg. Pengaruh masa pengekstrakan, kuasa gelombang mikro, dan isipadu pelarut dinilai daripada pengekstrakan sebatian fenol.

Kata kunci:Microwave Assisted-Extraction; pengekstrakan; sebatian fenol; Soxhlet; *C. domestica* Val.

1.0 INTRODUCTION

Turmeric is a member of Zingiberaceae family native to south Asia, which is a perennial herb with short and thick rhizomes with yellow flesh [1], grown in warm, rainy regions of the world. The essential oil is used for the manufacturing of cosmetics, drinks, foods [2], perfumes, and pharmaceutical drugs. Phenolic pigment is reported to have a medicinal value to remove blood stasis and alleviate pain [3], for the treatment of various skin diseases and common eye infection [4]. The principal colouring material in turmeric and its oleoresin are curcumin1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6heptadiene-3,5-dione with molecular formular of $C_{12}H_{20}O_6$ and two related minor curcuminoids; demethoxycurcumin and bisdemethoxycurcumin. A high degree of success in applying the microwave assisted extraction (MAE) technique based on solvent-sample duo-heating synergism (methanol and acetone), for the extraction of curcumin (phenolic) from *Curcuma longa l.* achieving 20 % of power, 2 g of sample, 4 mins of irradiation time with 40 mL of solvent [5]. As an alternative MAE provides considerable reduction in extraction time, solvent consumption with improved extraction rate and to some extent selective extraction [5]. The method showed drastic reduction in extraction time with much better precision when compared to conventional extraction methods. An attempt has been made to make use of microwave for extraction of two indigenous drugs *Emblica officinalis* and *Curcuma longa* using different solvent. Better result was obtained using MAE with water solvent with a recovery of 12 % with intensity of 450 W and time was 15 mins. The yield obtained in MAE is much more than the conventional extraction method and less time is required in MAE. In case of *Emblica officinalis* intensity was 560 W and time was not fixed but it was stopped when vigorous boiling of solvent started [6]. Moreover, the optimum extraction were achieved

at 700 W microwave power, at the irradiation time of 4 min. Recoveries of MAE-HS-SPME were between 86 to 93% [3].

In this research water was selected as a solvent in the extraction of dry rhizomes of *Curcuma domestica* Val. because it is more economical, friendliness to the environmental, health and safety benefit [7] and also use in order to replace organic solvent and recognise as safe (GRAS) solvent.

In the last two decades there has been an increasing demand for new extraction techniques, amenable to automation, with shortened extraction times, reduced organic solvent consumption, reduced pollution and reducing sample preparation costs [8, 9]. These conventional methods for the extraction of natural products from plant material for instance Soxhlet, liquid-liquid, and solid-liquid extraction are characterised by the consumption of large volumes of solvent and energy, lengthy extraction procedures, and the potentially deleterious degradation of labile compounds [10].

Many research have been performed in this area of research but, little attention has been paid to the application of unconventional method such as microwave-assisted extraction (MAE) to the natural drugs especially using water as solvent. This paper examines the use of MAE methods on extraction of curcumin (phenolic) from *C. domestica* Val. using water as solvent as well as evaluation of the effects of extraction parameters.

2.0 EXPERIMENTAL

2.1 Material and Method

The plant *C. domestica* Val. (kunyit) was obtained at Pasar Peladang Skudai market Johor Bahru, Malaysia. The sample (rhizomes) was chopped into smaller pieces and air dried for two weeks, and ground into powder in a mill blender, finally stored in the sealed plastic container for further analysis. Ethanol used in the experimental for extraction purpose is analytical grade from QRec (Asia). All the aqueous solutions were prepared with distilled water from a distiller from Hamilton laboratory glass limited, (Sandwich, Kent, England). A Kubota 2420 Centrifuge (Japan) was used.

2.2 Microwave Assisted Extraction (MAE)

Experiments were carried out in a Samsung MW61F microwave oven 700 W. 2 g of dry turmeric powder were mixed with water in suitable ratio and pre-leach (soaking of matrix with solvent prior to irradiation at room temperature for 10 min, before extraction in MAE. The suspension was irradiated with microwave oven at regular intervals one minute radiation and two minutes off) to keep temperature below 100°C. The temperature was measured with thermo-coupled digital multimeter by inserting the wire inside the sample in which the temperature was measured at 98°C. The crude extract was centrifuged at 3800 rpm for 15 min and the supernatant was carefully decanted. It was then evaporated to dryness in a water bath at 50°C. It was stored at 4°C until used.

2.3 Soxhlet Extraction

50 g of turmeric powder was extracted in 1000 mL of the water for 15 h in a Soxhlet apparatus. The aqueous crude extract was filtered and dried on top of the water bath at 50°C.

3.0 RESULTS AND DISCUSSION

3.1 Microwave Assisted Extraction and Soxhlet Extraction

The extraction yield of 48 mg was obtained from the dried rhizomes of *C. domestica* Val using MAE methods while 3.4 mg was obtained using Soxhlet extraction methods.

3.2 Effect of Microwave Power on Extraction

Different irradiation powers, 100, 250, 400, 550 and 700 W were used for a time of 3 min, 4 min, 5 min and 6 min, respectively, as shown in Figure 1. The results show that the extraction yield of curcumin from rhizomes of *C. domestica* Val., generally increases with the microwave power. Moreover, electromagnetic energy was transferred to the extraction system quickly and improved the extraction efficiency when the microwave power increased from 100 - 550 W. However, further increases of energy above 550 W shows no significant increased in the extraction yield. This might be connected to the facts that, higher power might lead to risk of degradation of compound resulting in decreased of extraction yield.

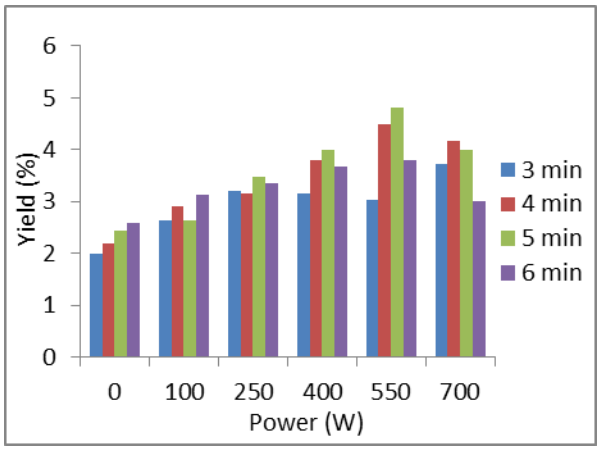


Figure 1 Variation of percentage yield with power at various irradiation times

Therefore, 550 W microwave powers would be adopted for the subsequent experiments as the optimum. Hence, it is in agreement with the result obtained by Joshi *et al.* [6] in the extraction of phenolics from *C. longa* but achieved the same % recovery using the same solvent.

3.3 Effect of Extraction Time on the Extraction

Extraction time is also a factor that affects the extraction yield of the analytes in the MAE. Extraction was carried out at different extraction times of 3 min, 4 min, 5 min and 6 min.

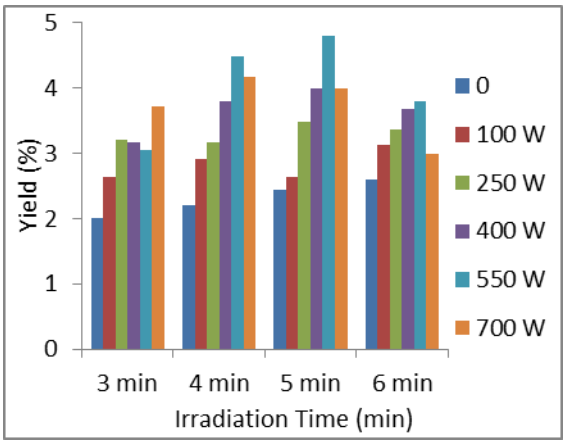


Figure 2 Variation of percentage yield with irradiation times at various powers

The results were shown in Figure 2. The experimental results indicate a pronounced increase of the extraction recovery of analytes from 3 to 5 min but decrease from 5 to 6 min. The significant of the extraction time can be assumed to be related to the time required for the desorption process to take place. Therefore, 5 min were considered as the optimum for the extraction of analytes. It is in agreement with data on the extraction of polyphenol by Wei *et al.* [11] and also the result obtained by Joshi *et al.* [6] in the extraction of compounds from *C. longa* though it is different plant but having the same compounds.

3.4 Effect of Solvent Volume on the Extraction

The influence of solvent volume on extraction yield from *C. domestica* Val., was investigated by performing an experiment. The extraction of solvent volume was increased from 10 to 50 mL, for the same mass of turmeric. The extraction yield of analytes from *C. domestica* Val. increased rapidly with an increased of solvent volume with optimum yield at 15 mL/g as shown in Figure 3. There is then drastic decreased in extraction yield at solvent volume above 15 mL/g. This is attributed to higher solvent which may not give better yield due to non-uniform distribution and exposure to microwaves energy.

On the other hand also, this might be as a result of larger volume causes excessive swelling of the material by water and absorbing the effective constituent of plant material. Therefore a volume of 15 mL was enough for the extraction of phenolics with is contrary to results by Vivekenanda *et al.* [5] where the extraction of phenolics was achieved with 40 mL of solvent volume, although is extracted from *C. longa*. Earlier studies found that higher solvent volume may give lower recoveries according to Uppendra *et al.* [12] and Eskilsson *et al.* [13].

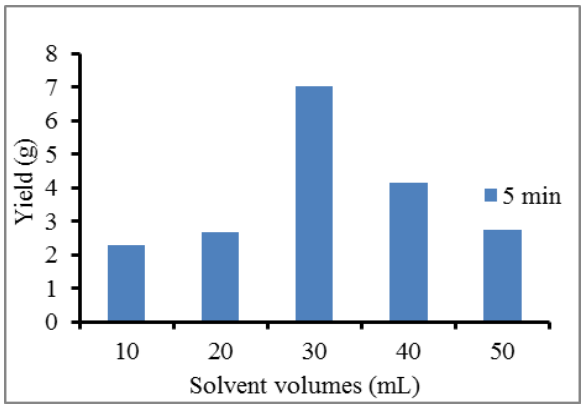


Figure 3 Variation of percentage yield with solvent volumes at 5 min time, 550 W and 2 g of sample material (turmeric)

3.5 Effect of Concentration of Ethanol

Among various solvents, ethanol was selected as a right choice because it is environmentally benign and relatively safe to human health. In addition, it is non-toxic and inexpensive solvent. The ethanol concentration was set at 20, 40, 60, 80 and 100% (v/v), respectively. Extraction of phenolic compound in *C. domestica* Val. is greatly influenced by the volume of ethanol concentration. When the percentage of ethanol concentration in the solvent increased from 20 - 60% (v/v), extraction yield increased significantly. Further increase of ethanol concentration affects the extraction yield slightly. However, as shown in Figure 4, when ethanol concentration is between 80 - 100 % (v/v), extraction recovery decreased.

The colour of the extracts deepened gradually when the volume of ethanol percentage was higher than 60% (v/v), which indicated that more undesirable compounds were also extracted. So, it was decided to adopt ethanol concentration of 60% (v/v) as extraction solvent. The results agree with the result obtained from Wei *et al.* [11].

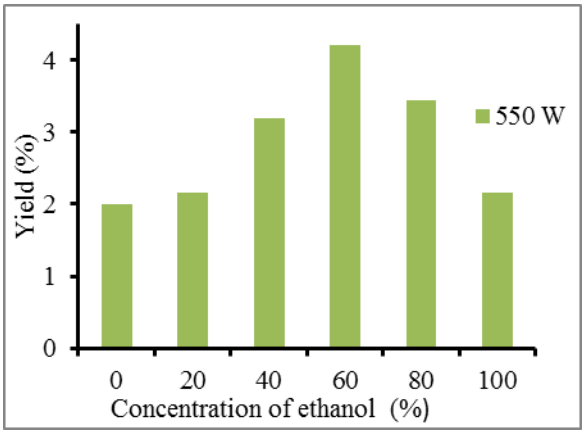


Figure 4 Variation of percentage yield against concentration of ethanol

4.0 CONCLUSION

Unmodified domestic microwave oven was used in the extraction of phenolic compound from *C. domestica* Val. using water as solvent. The MAE produced a better yield of 48 mg compared to Soxhlet extraction methods with only 3.4 mg yield. Microwave of energy above 550 W shows no significant increased in the extraction yield; this means is the optimum extraction power. 5 min was considered as the optimum for the extraction of analytes corresponding to the maximum extraction yield. The extraction yield of phenolics increased rapidly with an increase of solvent volume with optimum yield at 15 mL/g. There is then drastic decreased in extraction yield at solvent volume above 15 mL/g. Therefore, the economical solvent volume of extraction is 15 ml/g.

The colour of extracts deepened gradually when the volume of ethanol percentage was higher than 60% (v/v), which indicated that more undesirable compounds were also extracted. The ethanol concentration of 60% (v/v) is adopted as an optimum concentration for the extraction *C. domestica* Val. The MAE demonstrated shortened extraction times, reduced organic solvent consumption, prevention pollution in analytical laboratories and reducing sample preparation costs.

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