ARPN Journal of Engineering and Applied Sciences

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THE POTENTIAL OF MICROWAVE ASSISTED HYDRODISTILLATION IN EXTRACTION OF ESSENTIAL OIL FROM CINNAMOMUM CASSIA (CINNAMON)

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ABSTRACT

In this research, Microwave Assisted Hydrodistillation (MAHD) was used to extract essential oil from Cinnamomum Cassia (cinnamon). The effect of different parameters, such as water to raw material ratio (6:1, 8:1 & 10:1), microwave power (200 W, 225 W & 250 W) and extraction time (30 min, 60 min, 90 min, 120 min & 150 min) on the extraction yield and its major constituents were investigated. The essential oil was analysed by gas chromatography/ mass spectrometric (GC-MS) to evaluate the effect of extraction method on the content of its main constituent which was trans-Cinnamaldehyde. The optimum condition was found at water to raw material ratio of 8:1, microwave power of 250 W and extraction time of 90 min and the yield obtained under this condition was about 2.55%. The result obtained from GC-MS analysis revealed that the use of microwave irradiation did not adversely influence the composition of the essential oil. The main constituents found through MAHD was more desirable in terms of quality and quantity when compared to the conventional methods. The results obtained herein suggest that MAHD method could serve as a suitable and effective method for the extraction of essential oil from Cinnamomum Cassia (cinnamon).

Keywords: MAHD, cinnamon, extraction, GC-MS, optimization.

INTRODUCTION

Cinnamomum Cassia is an evergreen tropical tree, also named as Chinnese cinnamon, which is widely distributed in Southeast Asia. Cinnamon barks and leaves are widely used as spice and flavoring agent in foodstuffs, fragrances, herbicides, insecticides and for various applications in medicines. The essential oil obtained from the bark is rich in trans-Cinnamaldehyde with antimicrobial effects against animal and plant pathogens. Cinnamomum Cassia oil may also be used as potential repellents, antifungal, antioxidant and antitumor agents (Chang, Tak, Kim, Lee, & Ahn, 2006; Giordani, Regli, Kaloustian, & Portugal, 2006; Lin, Wu, Chang, & Ng, 2003; Shin *et al.* 2006). Reports of the statistics from the United Nations comtrade showed that in 2011, there was an estimated 24 billion USD global market for fragrances and flavors from essential oils with a yearly growth rate of about 10% (Ramu Govindasamy, 2013). This increasing demand for essential oils mainly has opened up wide opportunities for its global marketing, leading to the requirement of competitive product in market. These products are often expected to come with all the advantages in term of cost, quality and its production time. Since essential oil is a volatile component, it is vital to identify the best extraction technique, so that the higher yield of essential oil with good quality can be extracted.

The main methods used to obtain essential oils or extracts from the plant materials are hydrodistillation, steam distillation, steam and water distillation, maceration, empyreumatic distillation, and expression.

Among these methods, hydrodistillation is the most common approach used for the extraction of essential oils from medicinal herbs and plants. It had been noted however that this conventional method present several drawbacks such as long extraction time, potential loss of volatile constituents, high energy use, and so on, making it unsuitable for the demands of the current market (de Rijke *et al.* 2006). Incorporation of alternative extraction techniques that is rapid, sensitive, safe, and energy-efficient is therefore highly necessary. One possible solution towards the improvement on the existing extraction processes is to implore the potential benefits of the more active and efficient enhancement which can be achieved through the use of microwave.

In an attempt to take advantage of microwave heating with the conventional hydrodistillation, MAHD was then developed and used for the extraction of essential oils or extracts from some plants. With this method, the plant material placed in a Clevenger apparatus is heated inside a microwave oven for a short period of time to extract the essential oil where heat is produced by microwave energy. The sample reaches its boiling point very rapidly, leading to a very short extraction or distillation time. Using the microwave distillation technique, it is possible to achieve distillation with the indigenous water of the fresh plant material (Kürkcüoglu & Baser, 2010). Some of the extractions to which this technique had been applied includes but not limited to extraction of essential oils from Satureja hortensis and Satureja Montana (Rezvanpanah, Rezaei, Razavi, & Moini, 2008), Mango (Mangifera indica L.) flowers (Wang, Liu, Wei, Yan, & Lu, 2010) and from Thymus vulgaris L. (Golmakani & Rezaei, 2008). Although the effect of MAHD has been conducted on a number of plant materials as mentioned above, its effect on Cinnamomum Cassia has not yet been explored which makes it a good area for research.

VOL. 11, NO. 4, FEBRUARY 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

In this research, the potential of MAHD for the extraction of essential oil from Cinnamomum Cassia (cinnamon) was studied. The effects of operating parameters, namely extraction time, microwave irradiation power and proportion of water to raw material, on the extraction yields of Cinnamomum Cassia (cinnamon) essential oil was also investigated. Additionally, the chemical composition of the essential oil was analysed by GC–MS at optimum condition in order to compare the properties of essential oil extracted through MAHD with those extracted using the conventional hydrodistillation at both quantitative and qualitative level.

MATERIAL AND METHODS

Plant Materials

The Cinnamomum Cassia barks used for this research were collected as experimental raw materials from A.Munisamy and Sons Sdn. Bhd. in Southern Peninsular Malaysia (Johor Bahru, Johor). After drying, they were grinded and sieved using a mechanical sieve shaker fitted with a 50 mesh sieve. Prior to the experiment, 25 g of the powdered cinnamon bark was soaked in water at different ratio of 6:1, 8:1 and 10:1 of water to cinnamon powder respectively.

Microwave-Assisted Hydrodistillation

Essential oil from Cinnamomum *Cassia* (cinnamon) was extracted by using MAHD method. For the extraction, a modified domestic microwave oven model Samsung MW71E connected to a Clevenger-type apparatus was used. Experimental set up for the extraction stage is illustrated in Figure-1. The microwave has 1150 W power consumption, maximum output power of 800 W with 250 v - 50 Hz power source and 2450 MHz of microwave irradiation frequency. The cavity dimensions of the microwave oven were 306 mm x 211 mm x 320 mm. A 1 liter reactor was set up within the microwave oven cavity, and the pre-soaked cinnamon solution was transferred into the reactor. After extraction, the essential oil was collected using a Clevenger-type apparatus.

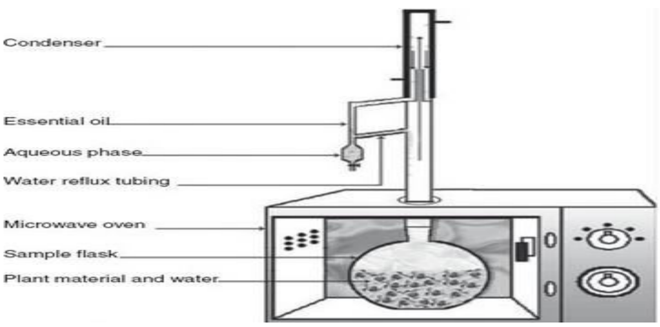

Figure-1. Schematic diagram of microwave-assisted hydrodistillation.

Optimization of Cinnamon Oil Extraction

Three extraction parameters were optimized. These are water to raw material ratio (w/w) (6:1, 8:1 and 10:1), extraction period (30, 60, 90,120 and 150 min), and operating power (200 W, 225 W and 250 W).

Sample Analysis

The extracted essential oil was dried over anhydrous sodium sulphate, weighed and stored in amber vial at 4^{0}C for further analysis. The yield of the essential oil was analyzed to evaluate the performance of MAHD in cinnamon oil extraction. Yield of the essential oil was determined tripilicate, and the results were presented as a mean value. The yield of the extracted essential oil was obtained using following equation (1):

$$\text{Yield (\%)} = \frac{\text{Amount of essential oil (g)obtained}}{\text{Amount of raw material (g) used}} \text{ x } 100\% \quad (1)$$

The extracted essential oil samples were analyzed with the help of a gas chromatography/ mass spectrometry (GC-MS), model Agilent 6890 gas chromatography instrument coupled to an Agilent 5973 mass spectrometer and an Agilent Chem in order to identify their chemical constituents. This is an essential method to evaluate the quality of the oil samples. For the analysis, the following operating condition was used: system operating in EI mode (70 eV), equipped with splitness injector at 270^{0}C, using HP-5MS column (30 .00 mm x 0.25 mm i.d x 0.25 mm). The column temperature was kept at 100^{0}C for 4 min, increased to 230^{0}C at a rate of 5^{0}C/min and held for 5 min. Helium was used as carrier gas at a flow rate of 1 mL/min (Li, Kong, & Wu, 2013).

RESULTS AND DISCUSSION

The experiment was run at different parameters in order to identify the optimum condition. The various effects of the different operating parameters are as presented in the following sections.

Effect of Microwave Power on Yield

Effect of microwave power on cinnamon oil yield at fix water to raw material ratio of 8:1 is shown in Figure-2. Three different microwave power levels were used in the extraction which are 200 W, 225 W and 250 W. From Figure-2, it can be seen that when the microwave power increases, the yield of extracted cinnamon oil also increased for all categories of the extraction power. Noteworthy is the highest yield of 2.55% which was obtained when cinnamon oil extracted was conducted at 250 W for a period of 90 min. In a similar research which was carried out by other researchers using the conventional method of hydrodistillation, report showed that the highest yield which was obtained from Cinnamomum Cassia was about 2.38% (Li *et al.* 2013).

This indicates that the highest extraction yield (2.55%) obtained from MAHD was considerably high compared to conventional methods. As can be seen from the graph, the initial extraction rate increased as the microwave power level was increased. Similar observation was also reported in a previous research work (Hu, Cai, & Liang, 2008). The reason for this could be associated with the rapid genaration of heat inside the immersed cinnamon with the absorption of microwave energy and subsequent formation of a higher pressure gradient inside the plant material (Ma *et al.* 2009; Rezvanpanah *et al.* 2008). However, with a prolonged heating for a period of about 120 to 150 min, the extraction yield can be seen to decrease for all the three microwave power levels. The reason for this, shall be discussed in the next section.

On the other hand, it was observed that the time taken to reach the boiling point of the mixture (induction time) was shorter at higher microwave power level. Induction time for 200 W, 225 W and 250 W were 20 min, 15 min and 9 min respectively at constant water to raw material ratio of 8:1 and at an extraction time of 90 min. These observations could be attributed to the effect of microwaves on polar solvent, water, which has high dielectric constant. Therefore, the lower microwave power might tend to extend the induction time, perhaps due to the lower density of waves at such power level. Owing to these observations, the optimum microwave power for further experiment was selected to be 250 W (Rezvanpanah *et al.* 2008).

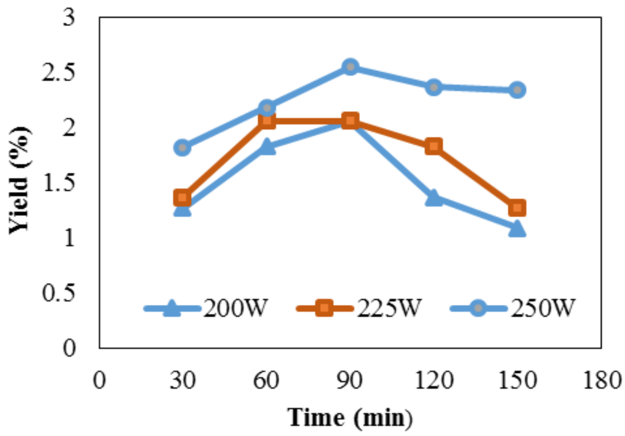

Figure-2. Varition of essential oil yield of cinnamomum cassia (cinnamon) at different microwave power level in water to raw material ratio of 8:1

Effect of Extraction Time on Yield

Figure-3 illustrates the yield of essential oil extracted from Cinnamomum Cassia (cinnamon) at different extraction time at a constant microwave power of 250 W, and at water to raw material ratio of 8:1. From the graph, it can be seen that the amount of yield increases as the extraction time was increased from 30 min to 90 min. The yield obtained at 30, 60 and 90 min are 1.82%, 2.18% and 2.55% respectively. Obviously, MAHD was able to produce the highest yield of 2.55% w/w when the extraction time was 90 min. However, as the extraction time was increased beyond 90 min, the yeild can be seen to be notably reduced. Similar observation was reported for MAHD of Radix Astragali (Xiao, Han, & Shi, 2008).

The initial increase in extraction yield could be accrued to the higher heating rate contributed by the high dielectric properties of polar solvents such as water, when they are subjected to temperature increase. The decrease in yield after 90 min extraction time could however be associated with the possible degradation of the plant material as the extraction time becomes prolonged (Chen, Xie, & Gong, 2007). This might therefore lead to undesired evaporation of the volatile component in the cinnamon oil which could invariably lead to a decrease in the extraction yield as observed from the graph. Similar observation was also reported in literature (Rezvanpanah *et al.* 2008; Xiao *et al.* 2008). Thus, based on the results obtained herein, 90 min had been selected as the optimal time for extracting cinnamon oil by MAHD.

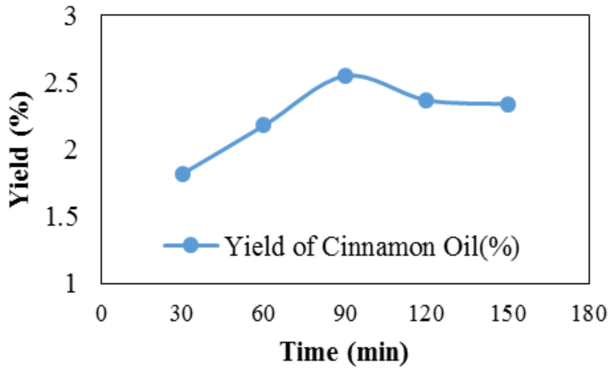

Figure-3. Extraction yield of essential oil from cinnamomum cassia (cinnamon) at different extraction time by 250 W in water to raw material ratio of 8:1

Effect of Water to Raw Material Ratio on Yield

The effect of different water to cinnamon powder ratio on yield of cinnamon oil at specific power level of 250 W is shown in Figure-4. The mass of cinnamon powder used for the experiment was kept constant at 25 g of Cinnamomum Cassia (cinnamon). On the other hand, the amount of distilled water added as solvent was varied such that the ratio of water to powdered cinnamon was 6:1, 8:1 and 10:1 respectively. As illustrated in Figure-4, the highest yield was obtained at ratio of 8:1 followed by 6:1 and 10:1. The highest yield at 6:1, 8:1 and 10:1 for cinnamon oil were 2.55%, 2.55% and 2.06% respectively.

Generally, the main function of water in distillation is to prevent the raw material from being thermally degraded and also act as the carrier of essential oil during the evaporation before the condensation process took place. Larger amount of solvent may consume more energy and more time would be needed to condense the exraction solution. Furthermore, it might cause excessive thermal stress due to rapid heating of solvent (Dhobi Mahaveer & 2009). On the other hand, smaller volume could lead to incomplete extraction of the target substance. This could be in form of failure on the part of the solution to withstand high microwave intensities especially if the extraction time is prolonged. Invariably this could lead to the burning off of the plant material in the flask (Gao, Song, & Liu, 2006; Ma *et al.* 2009). This explains the reason while the water to raw material of 8:1 (ml:g), was efficient to produce the optimum yield of the extracted oil from Cinnamomum Cassia.

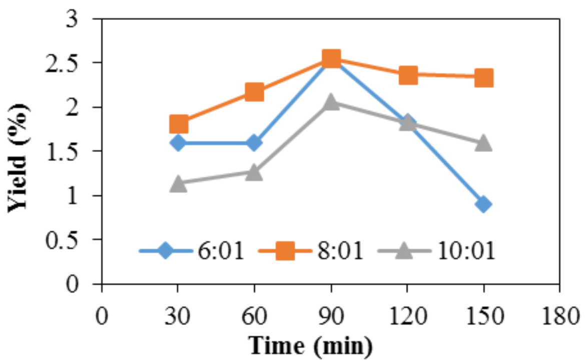

Figure-4. Variation of essential oil yield of cinnamomum cassia (cinnamon) with time in different water to raw material ratio at 250 W.

Identification and Quantification of Extracted Essential Oil

At optimum condition of water to raw material ratio of 8:1, microwave power level 250 W and extraction period of 90 min, a total of about 31 components were observed. The most abundant and active component of the cinnamon oil was trans-Cinnamaldehyde (81.00%). In addition, the cinnamon oil also contained cis-cinnamaldehyde (1.19%), linalool (1.02%), benzaldehyde (1.29%), anethole (1.97%), caryophyllene (1.02%) and 2-propen-1-ol, 3-phenylacetate (2.15%). The area percentages of other observed volatile compounds were generally less than 1.00%. From a previous research, it was reported that the maximum content of trans-Cinnamaldehyde which was obtained from Cinnamomum Cassia through conventional methods, was only around 33.95 – 76.40% (Geng *et al.* 2011). Results from this present research showed up to about 81.00% of trans-Cinnamaldehyde. This suggests that MAHD is more effective in essential oil extraction from Cinnamomum Cassia compared to other conventional methods.

CONCLUSIONS

The potential of microwave assisted hydrodistillation (MAHD) method in the extraction of essential oil from Cinnamomum Cassia (cinnamon) was investigated. The optimum condition for the lab scale MAHD extraction method was found to be 250 W microwave power for extraction time of 90 min and water to raw material ratio of 8:1. The maximum yield obtained was 2.55%. At the optimum condition, trans-Cinnamaldehyde (about 81.00%) was the major constituent found in the Cinnamomum Cassia oil. This component is well known for its antitumor properties and also its desirable use as potential repellent.

Based on the results obtained, shorter extraction time and power level with optimum water to raw material ratio have been used by MAHD to produce good quality and quantity of cinnamon oil. This indicates that MAHD could serve as a cost effective and time conserving method which could offer great economic value to industries that produce essential oils.

ACKNOWLEDGEMENTS

This research was sponsored by the Ministry of Education Malaysia through the research grant RDU 140397. The authors also appreciate the efforts of Mr.Jeyakhantan and Mr.Jeyakartik for their assistance in collection of Cinnamomum Cassia (cinnamon) samples.

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