COMPARATIVE STUDY BETWEEN HYDRODISTILLATION AND MICROWAVE-ASSISTED HYDRODISTILLATION FOR EXTRACTION OF CINNAMOMUM CASSIA OIL

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ABSTRACT

Microwave assisted hydrodistillation (MAHD) is an advanced technology extraction method that came up through the incorporation of microwave irradiation into the conventional method, hydrodiatillation (HD). In this research, both MAHD and HD methods have been compared and evaluated for their effectiveness in the extraction of essential oil from (cinnamon) barks. The effect of various parameters such as microwave power level (200W, 225W, 250W and 275W), water to raw material ration (6:1, 8:1 and 10:1) and extraction time (30 min, 60 min, 90 min, 120 min, 150 min and 180 min) on the yield of extraction and its major constituents were investigated and compared accordingly between MAHD and HD. At the optimum parameters of microwave power, 250W, water to raw material ratio of 8:1 and extraction time of 90 min, the yield obtained was 2.55% and 1.89% for MAHD and HD respectively. Gas Chromatography/Mass Spectrometric (GC-MS) used to further analyse the effect of both extraction methods by evaluating the content of its main constituents which is trans-cinnamaldehyde and oxygenated compounds. Results revealed that MAHD method possesses highly desirable features than HD and could serve as an efficient and convenient alternative to HD, especially in terms of its shorter extraction time, as well as the potentiality to produce reasonably good quantity and quality of cassia oil.

Keywords: cinnamomum cassia, microwave-assisted hydrodistillation, hydrodistillation.

INTRODUCTION

Essential oils are often defined as concentrated liquids with hydrophobic and lipophilic properties which are rich in volatile aromatic compounds and carries unique scent, flavours and essence when extracted from plant. These essential oil could be extracted from various parts of a plant such as flowers, seeds, leaves, roots and barks. In the past ages, essential oil were traditionally used in healing diseases where it was believed to have anti-bacterial, anti-fungal and anti-parasitic properties [1, 2].

However, nowadays, the demand for essential oil has increased in industries such as perfumes, cosmetics, flavouring foods, cleansing products as well as in pharmaceuticals. This was confirmed by the statistics from the United Nations comtrade in the year 2011, there was estimated 24 billion USD global market demand for flavors and fragrances from essential oils with growth of about 10% yearly [3]. Therefore, the interest in producing essential oil has been resuscitated in recent decades, due to its distinctive aromatherapy property, raising its demand especially in cosmetic industry. Also there is increasing demand for essential oils as alternative medicine due to the belief that specific aroma compounds present in essential oils has curative effects on certain diseases [4].

Essential oil could be extracted by various methods, e.g. hydrodistillation (HD), steam distillation, expression and organic solvent distillation. The most commonly used methods to extract essential oil is HD. However, monoterpenes compounds easily damage under steam distillation and conventional method HD tends to lose more volatile compounds during solvent removal [5]. Besides that, conventional methods have drawbacks of time-consuming as well as being energy intensive. Therefore, nowadays, a lot of advanced extraction technologies are being researched by many researchers to enhance the quantity and quality of the yield of essential oil. Essential oil had been reportedly extracted from different plant materials in times past through different methods. Among these plant material, Cinnamomum cassia is of particular interest and several techniques are under study to extract oils from this plant for both quantitative and qualitative advantage. Notable among these is the use of microwave-assisted hydrodistillation in extraction of Cinnamomum Cassia oil [6, 7]. This had been perceived to be a valid alternative to the conventional method, generally because the irradiation power of microwave tend to produce more yield and shorter operating time could also be achievable [8].

Cinnamomum cassia belongs to Lauraceae family otherwise known as Chinese cinnamon, mainly distributed in Southeast Asia. [6]. The bark of Cinnamomum cassia is widely used as food flavouring, perfumes, and cosmetics and also acts as certain disease curative such as gastritis, dyspepsia, regulate blood circulation and inflammatory disease. Besides that, cinnamon oil rich in trans-cinnamaldehye is believed to have antimicrobial effects against animal and plants. The present research aimed to evaluate the effect of microwave assisted hydrodistillation on operational conditions such as extraction time, water to raw material ratio and microwave power for extraction of essential oil from Cinnamomum cassia. Quality and quantity of extraction yield shall also be investigated by comparing the active and oxygenated compounds. In addition, the applicability of MAHD technique shall be investigated as an alternative to conventional method, HD in extraction of *Cinnamomum cassia* oil.

MATERIAL AND METHODS

Plant Samples

The raw Cinnamomum Cassia (cinnamon) barks used for this experimental work was collected from A.Munisamy and Sons Sdn. Bhd. in Southern Peninsular Malaysia (Johor Bahru, Johor). The raw samples were washed in distilled water and dried in air at an ambient temperature of 25-30^{0}C and relative humidity of about 30-40%. After the drying process, cinnamon barks were grinded by help of a grinder machine (model-Retcsh Laboratory Knife Mills GRINDOMIX GM 200), and sieved with a 50 mesh sieve fitted to a mechanical sieve shaker. Grinding and sieving were carried out due to reports from literature which stated basically that as the particles size of cinnamon bark powder decreases, the extraction yield increases due to higher amount of essential oil released as the bark phloem are destroyed by grinding [9, 10]. Prior to extraction, 25 g of the powdered cinnamon bark was soaked for 30 minutes in distilled water, at different ratio of 6:1, 8:1 and 10:1 of water to cinnamon powder respectively.

Microwave-Assisted Hydrodistillation

Extraction of essential oil from Cinnamomum cassia (cinnamon) was carried out through Microwave-Assisted Hydrodistillation method. For this purpose, a microwave oven (model - Samsung MW71E), was domestically modified and connencted to a Clevenger-type apparatus. The microwave possesses a power consumption capacity of 1150 Watt with 800 watt maximum power output, power source (250v – 50Hz) as well as a microwave irradiation frequency (2450 MHz). The dimension of the microwave cavity is 306 x 211 x 320 mm. For the extraction process, the pre-soaked cinnamon powder was transferred into a 1 liter capacity reactor which was initially set up within the cavity of the microwave oven. During the microwave assisted hydrodistillation extraction process, the extracted cinnamon oil was decanted from its Clevenger-type condenser at an interval of about 30 minutes throughout the 150 minutes extraction period. Collection of the extracted essential oils was done using a Clevenger-type apparatus which was placed on top of the microwave oven. After extraction, the essential cinnamon oil was dehydrated over anhydrous sodium sulfate to remove excess water, after which the concentrated cinnamon oil was weighed and stored in vial at 4^{0}C for further analysis.

Hydrodistillation

The essential oil from Cinnamomum cassia (cinnamon) was extracted in accordance with hydrodistillation method described in the European Pharmacopoeia [11]. 25 g of cinnamon powder was weighed and mixed in distilled water in at a ratio of 6:1, 8:1 and 10:1 of water to cinnamon powder respectively. The cinnamon powder was pre-soaked for 30 min as in the case of MAHD and then transferred into 1 litre reactor placed in a heating mantle. The extraction time used for hydrodistillation method was 30, 60, 90, 120, 150 and 180 min until no further cinnamon oil was obtained. The distillate was collected using a Clevenger-type apparatus.

Optimization of Oil Extraction

The three extraction condition parameters which were optimized includes water to raw material ratio (w/w) (6:1, 8:1 and 10:1), extraction period (30, 60, 90,120 and 150 mins), and operating power (200 W, 225 W, 250W and 275W) for microwave-assisted hydrodistillation (MAHD).

Sample Analysis

Analysis of the dehydrated cinnamon oil was done through Gas Chromatography-Mass Spectrometry (GC-MS) analysis. The yield of the essential oils were analyzed to evaluate the performance of MAHD and HD in cinnamon oil extraction. Yield of the essential oil was determined tripilicate, and the results were presented as a mean value. Yield of the essential oil was obtained using following equation:

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

For this analysis, a GC-MS instrument (5973N, Agilent Technologies, Wilmington, DE, USA) equipped with a mass selective detector operating in the electron impact mode (70eV) was used to study the composition of the essential oil at the various conditions of their extraction. The GC part (6890N, Agilent Technologies, Palo Alto, CA, USA) was equipped with HP-5MS column (Agilent BTechnologies) capillary column (30 m long, 0.25 mm id and 0.25 mm film thickness). Temperature-programming of the oven included an initial hold at 100 °C for 4 min and a rise to 130 °C at 5 °C min-1 and maintained for 20 min at the same temperature. A final hold for 3 min was allowed for a complete column clean-up. The samples were diluted with Dichloromethane (1/10, v/v) and a volume of 1.0 μl was injected to the GC with the injector in the split mode (split ratio: 1/30). Carrier gas, He, was adjusted to a linear velocity of 1.2 ml min-1. EI mode (70 eV) at 230 °C used to analyse the mass spectral. The mass analyser was set from 30 to 550 amu for scan at 1s scan time. In order to identify the chemical constituents present in the extracted essential oil, their mass spectra was either compared with those of standard compounds from the National Institute of Standard and Technology (NIST) library data from GC-MS, database (Wiley/ NBS library) or with published mass spectra [12, 13]. Close similarity in retention time of the compound from both techniques were observed when the components were quantified. The percentage of each constituent was calculated using the normalization technique, based on the area of each peak from a total area peak value estimated to be 100%.

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RESULTS AND DISCUSSION

Effect of Microwave Power on Yield

Irradiation intensity of microwave power is a vital element which often influence the efficiency of MAHD extraction. Microwave power supplies the sufficient energy to the sample where this energy is converted into heat energy in the dielectric material such as water, and consequently increases the temperature of the solution. Figure-1 illustrates the effect of microwave extraction power on yield and induction time of MAHD and HD extraction at a fixed water to raw material ratio of 8:1 as well as at a constant extraction time of 90 min. It can be obviously seen from Figure 1 that the yield of extracted cassia oil increased with an increasing microwave power level until it reached a maximum point at after which it began to reduce. In a previous research work by Hu [14], similar observation was also reported. Reason for this was accrued to the fact that higher microwave power would supply more energy to the immersed cinnamon, produce rapid generation of heat, and subsequent formation of higher pressure gradient inside the Cinnamomum cassia solution such that the extraction is enhanced [15-17].

However, it can be seen that when the microwave power was further increased beyond 250W, the cassia oil yield started to decrease. There is the possibilty of a negative effect from higher microwave power, which might tend to overheat the product by elevating the temperature too high, leading to the compound breakdown or product damage [15]. This could be the reason for the decrease in yield from 2.55% to 2.21% at microwave power of 250W and 275W respectively. On the other hand, by comparing the highest yield of 2.55% cassia oil obtained at 250 W for a period of 90 min for MAHD with conventional method of hydrodistillation (HD) at the same extraction time of 90 min and water to raw material ratio of 8:1, a lesser yield of 1.89% cassia oil was obtained. Thus, the yield obtained from MAHD was about 26% higher than in HD at similar extraction conditions.

On the other hand, it can be seen shorter time was taken to reach the boiling point of the mixture (induction time) for higher microwave power levels. At constant water to raw material ratio of 8:1 and at period of 90 min, the induction time for 200 W, 225 W, 250 W and 275 W were 20 min, 15 min, 9 min and 5 min respectively. In the case of HD, the induction time was 17 min at same conditions. This indicates that MAHD use shorter time to reach the boiling point, as well as began to produce the essential oil earlier, with the possibility of up to about 47% total extracted oil even before HD began the distillation process.

This could be related to the effect of microwave on water which is a polar solvent and possesses high dielectric constant. Hence, the induction time might tend to be longer in lower microwave power, due to the lower density waves at lower power level [16]. Whereas for HD, the heat is supplied paricularly to the bottom of the reactor and the heat energy might be localized at one particular place. Subsequently molecules of cinnamon at the bottom of reactor will begin to gain heat energy and convert it into kinetic energy. This kinetic energy will vibrate and rotate the molecules inside the cinnamon solution and it might influence the neighbour molecules to join in the reaction. As a result, it will take longer time to reach the induction time. Based on the above observations, 250 W was determined as the optimum microwave power for further experiment.

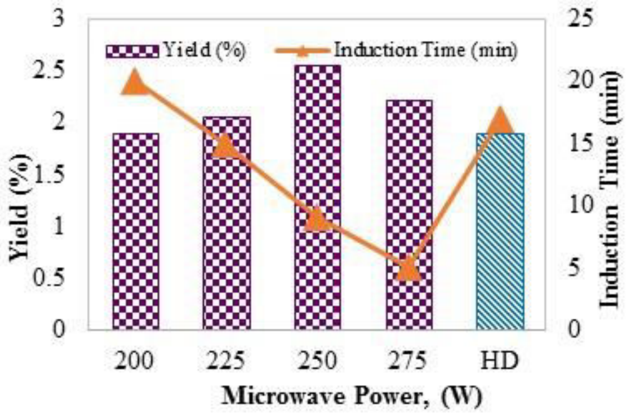

Figure-1. Variation of cinnamon oil yield and induction time (min) of MAHD and HD at different microwave power levels in water to raw material ratio of 8: 1 and extraction time of 90 minutes

Effect of Water to Raw Material Ratio on Yield

Figure-2 illustrates the effect of various water to raw material (cinnamon powder) ratio on cassia oil yield at constant extraction time of 90 min microwave power level of 250W (for MAHD), and the corresponding yield from HD during the 90 min extraction time at the different water to raw material ratios. Throughout the experiment, the mass of cinnamon powder was kept constant at 25g while the amount of solvent (water) was varied such that the ratio of water to raw material was 6:1, 8:1 and 10:1 respectively. The yield extracted from Cinnamomum cassia at 6:1, 8:1 and 10:1 for a period of 90 min were 2.06%, 2.55%, and 2.06% respectively for MAHD. On the other hand, the cassia oil yield extracted at 6:1, 8:1 and 10:1 at period of 90 min were 1.68%, 1.89% and 2.10% respectively for HD. Generally, the purpose of the water in distillation process is to prevent the cinnamon powder in the solution from being thermally degraded. It also serves as a carrier for cinnamon oil during the evaporation process. However, the usage of large volume of solvent might lead to more energy and time being consumed to condense the extraction solution.

Moreover, rapid heating of water could lead to excessive thermal stress [18]. In contrast, smaller volume of solvent may cause incomplete extraction of the target substance. This is because when high microwave intensity is radiated for a long time, the solvent would evaporate faster and the plant material could be burnt off [15, 19]. This could be the reason for the observed trend in the case of MAHD. However, it is worthy of note that whereas there was an initial increase followed by a decrease in yield as the volume of solvent was increased for MAHD, there was a progressing increase in yeild as volume of solvent was increased for HD.

In essence, while the optimum yeild for MAHD was obtained at water to raw material ratio of 8:1, the highest yield for HD was only obtained when the water to raw material ratio was 10:1. This suggests that MAHD requires less solvent to produce high yield of essential oil compared to HD. Thus, optimum yield of the extracted oil from Cinnamomum assia was obtained at water to raw material ratio of 8:1 (ml:g) and this was used as the criteria for further analysis.

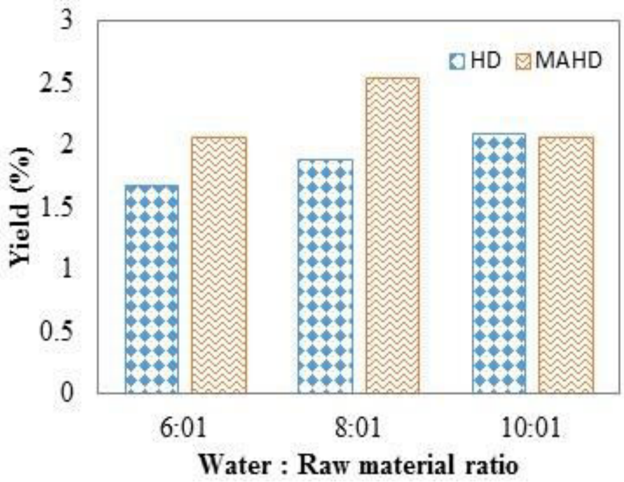

Figure-2. Various water to raw material ration at fixed extraction time for both MAHD and HD.

Effect of Extraction Time

Figure-3 shows the variation in extracted cassia oil yield with extraction time at a fixed microwave power of 250 W, and at water to raw material ratio of 8:1. As can be seen from the graph, there was an initial progressive increase in yield of Cinnamomum cassia, as the extraction time increases from 30 up till 90 min. At extraction time of 30, 60 and 90 minutes, the extracted cassia oil yields are 1.82%, 2.18% and 2.55% respectively. However, when the extraction time became extended beyond 90 min to 120 min and further, there is notably decrease in the yield of cassia oil. Similar observation was reported in literature for for MAHD of Radix Astragali [20].

On the other hand in the case of HD, an increasing trend can be seen for the cassia oil yield as the extraction time was increased form 30-150 mins, after which a decrease was detected when the extraction was raised from 150-180 mins. The yield for at 150 and 180 min are 2.3% and 2.13% respectively. It is worthy of note that whereas the highest yield for MAHD was obtained at 90 min extraction time, the highest yield from HD was not obtained until after about 150 min extraction time. Similar observation had been reported in literature and the result was reported to indicate that MAHD requires a shorter time to produce high yield of essential oils compared to conventional HD method [21].

In essence, MAHD could reduce the extraction time needed to obtain the highest yield of essential oil on one hand, as well as enhance rapid production of essential oil on the other hand with the possibility for higher energy saving [22]. It should be noted however that the initial increase in yield of MAHD in the range of 30-90 min extraction time could be due to the polar solvent (water) which possesses high dielectric properties that tends to contribute higher heating rate, when they are expose to higher temperature. However, after 90 min the observed decrease in cassia oil yield could be related to the possible plant material degradation as the extraction time becomes prolonged [23]. This is because at a prolonged extraction time, volatile component in the cinnamon oil might be evaporated, leading to a decrease in the extraction yield of Cinnamomum cassia as reported in literature [16, 19, 20].

Thus, for MAHD, 90 min was selected to be the optimum time in extracting cassia oil. It should be noted however that the difference in extraction time between the two methods could be related to the influence of microwave on the MAHD extraction. With microwave assisted hydrodistillation, there is the possiblity to attain the induction time faster compared to HD. Furthermore, the overall extraction time for MAHD can be seen to be lower than for HD.

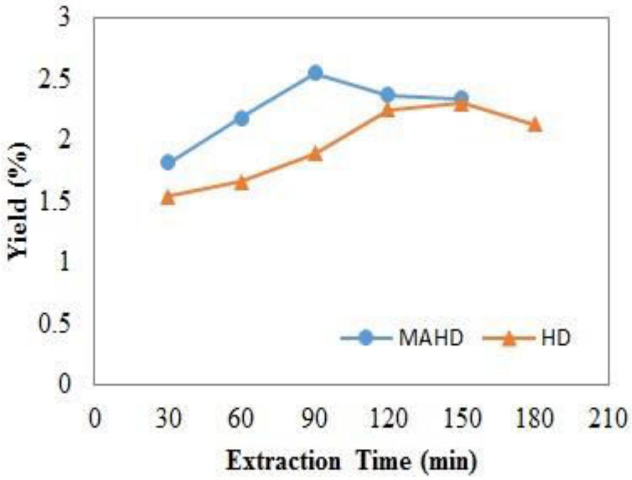

Figure-3. Variation of yield with extraction time for MAHD and HD at water to raw material ratio of 8:1.

Quantity and Quality of Cinnamomum Cassia Oil

At the optimum conditions of MAHD as stated in the previous sections, the effect of microwave evaluated on the extraction yield of cassia oil, were compared with conventional method of HD in terms of quantity and quality of the oil. The amount of cassia oil yield obtained at optimum conditions for the MAHD and HD techniques were 2.55% and 1.89% respectively. This indicates that at the optimum conditions, MAHD produced higher yield compared to conventional HD method. For the qualitative anlalysis, the identified components in cassia oil are represented in Table-1.

The major active compund found to be in the Cinnamomum cassia oil was trans-cinnamaldehye which was found be of relatively equal amount in both extraction techniques with a total value of 81.00% and 79.94% respectively for MAHD and HD. It was can be seen however that there tends to be more oxygenated compounds in the cassia oil which was extracted by MAHD method compared to HD method. Some of the oxygenated compounds with their constituent values are presented in the Table-1. These includes linalool, benzaldehyde, caryophyllene, cis-cinnmaldehye, anethole and 2-propen-1-ol, 3-phenylacetate all of which were found to show higher relative peak area percentage on MAHD compared to traditional method, HD.

Generally the essential oil contains high percentage of organic compounds which can easily absorb radiation of microwave energy. Organic compound which are oxygenated are often classified as high dipolar moment compound which tend to absorbed more microwave energy [4]. This is due to the potentiality of the oxygenated organic compounds present in these essential oil to absorb the intensity of the microwave energy through polar solvent (water). When the oxygenated compounds absorbed sufficient energy from microwave irradiation, there is more possiblity for these oxygenated compounds to easily released from oil glands of cinnamon cell through phloem of cinnamon bark [24].

This could be the reason for the higher amount of oxygenated compounds observed in MAHD compared to HD, conforming to reports from a previous research work [4]. These results indicates that the advance technique (MAHD) has the capability to support rapid extraction process for the production of larger quantity of essentila oil with good quality, without much observable damaged to the volatile composition in the cassia oil.

Table-1. Composition of Cinnamomum Cassia (cinnamon) oil obtained by MAHD and HD.

	Relative peak area (%)	
Compounds	**MAHD**	**HD**
Trans-cinnamaldehyde	81.00	79.94
Linalool	1.02	0.76
Benzaldehyde	1.29	0.88
Caryophyllene	1.02	0.69
Cis-cinnamaldehyde	1.19	-
Anethole	1.97	-
2-propen-1-ol, 3-phenylacetate	2.15	-

CONCLUSIONS

This research concentrated on effect of microwave on microwave power, water to raw material ratio and extraction time in comparison with conventional method, hydrodistillation. The yield of extracted cassia oil and its constituents in the cassia oil were evaluated and compared for the both methods. The optimum conditions for MAHD obtained were 250 W, 90 min of extraction time and 8:1 of water to raw material ratio respectively. The optimum yield of Cinnamomum cassia obtained at the optimum conditions were 2.55% and 1.89% for MAHD and HD respectively.

This indicates that advance technology, MAHD has better potentiality than conventional method, HD. MAHD which had been reported for its capacity of rapid essential oil production, had also been proven herein as result showed that MAHD requires a shorter extraction time to produce essential oil compared to conventional method.

However, the chemical constituents present in the cassia oil by both methods shows no significant differences in the quantity of the active compounds, trans-cinnamaldehyde. It is worthy of note however that MAHD offers additional benefits of better microwave intensity for isolation of oxygenated compounds.

Thus, microwave assisted hydrodistillation possesses highly desirable features compared to the conventional method. Some of these features includes but not limited to time saving during essential oil of extraction, invariably leading to reduced cost and energy. Furthermore, there is the possibility for obtaining better quality and quantity of cassia oil through MAHD technique. This therefore suggests that MAHD is one of the potential techniques which can conveniently, effectively as well as efficiently serve as an alternative to the conventional HD method for extracting essential oil both at laboratory and industrial scale productions.

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