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Rapid Expansion Supercritical Solution (RESS) of Carbon Dioxide as a Green Technology Method for Leucaena Leucocephala Solid Oil Particle Formation

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Abstract: ***Rapid expansion supercritical solution (RESS) is a technology introduced that produces a small solid oil particle with organic solvent free particle. RESS method is a better method compared to the traditional method due to many advantages such are produce clean extraction yields, obtain free solvent traces, environmental friendly and short processing time can be used in various industries such as cosmetic and pharmaceutical. Therefore, RESS technique is applied in order to produce direct solid oil particle from Luecaena leucocephala (L.leucocephala) pods instead of oil. In this study, the operating condition that applied during the RESS process in order to obtain the highest solid oil particle from the L.leucocephala pods sample are extraction temperature of 40 ºC, 45 ºC, 50 ºC, 55 ºC and 60 ºC and extraction pressure of 3000 psi, 4000 psi, 5000 psi, 6000 psi and 7000 psi at the constant extraction time of 40 minutes. The highest yield of L.leucocephala solid oil particles that obtained is about 0.65% at 60 ºC and 7000 psi. The major components that obtained from the highest yield are 2,2,3-trimethyldecane, Cyclopentanone, propanoic acid and Cyclobutane, 1,2-diphenyl- by as identified by using Gas Chromatography Mass Spectrometry (GCMS).***

Keywords*: Extraction temperature and Pressure, Luecaena Leucocephala* **pods**, *Rapid Expansion Supercritical, Solid Oil Particles*

I. INTRODUCTION

Leucaena leucocephala (*L.leucocephala*) locally knows as ‘Petai Belalang’ is the Leguminose or Fabaceae family. This species found in tropical and subtropical areas that need the warm temperature for the optimum growth. The plant from the leguminosae family has been used for traditional medicines in Malaysia such as pastes for wound infection and decoction [1]. Health organization identified that 65% to 80% of populations rely on traditional medicine from leguminosae medicinal plants to treat various diseases [1].

There are various bioactivities like anticancer, anti-inflammatory, antimicrobial and antioxidant properties contain in the *L.leucocephala* plants which is traditionally to treat disease [1]. The pods of *L.leucocephala* are widely used in pharmaceuticals because high tocopherol content in the extract oil of the pods where the major compound is α-tocopherol with high vitamin E activity. Besides, pods oil has advantages to industrial application where it is useful for pharmaceutical, cosmetic, suspending agent and high quality of insecticides [2].

The seed of this species is significant as a medical treatment where it is used to expel intestinal worms and as a therapy for diabetes in Indonesia while in Philippines, it is a traditional medicine to increase the menstrual flow for women [3].

From the previous study, the traditional method is mostly applied in the pharmaceutical industry to produce the fine particles of pharmaceutical product. The traditional extraction methods commonly used worldwide, especially in the pharmaceutical industry where arises some problem due to lead of low bioavailability where a large number of drugs insoluble in water [4]. There are various traditional methods used to reduce particle size such as milling, grinding, high pressure homogenization and spray drying in pharmaceutical industry. Unfortunately, they are using a large amount of solvent, require of high energy and also chemical and thermal degradation product. Therefore, a new technology of extraction method which is RESS method is used in this study to produce the small particle size from *L.leucocephala*. RESS method is an efficient and appropriate method used in the pharmaceutical production due to have many advantages such as producing smallest particle size, solvent free product and controllable particle size [5]. In RESS method, supercritical carbon dioxide (CO_2) is consumed as a solvent which is environmental friendly, nontoxic, nonflammable and potentially used as a clean medium to replace the traditional solvent in industrial processes [6]. However, up to date no studies of particle preparation *L.Leucocephala* using the RESS method has been done.

The objective of this research is to explore the possibility of Rapid Expansion Supercritical Solution (RESS-CO_2) extraction method to acquire the formation of

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solid oil particle from *L.leucocephala* pods. Furthermore, determination on the best operating conditions for the highest solid oil particles yield and characterize the bioactive component of solid oil particle by using gas chromatography mass spectrometry (GCMS) will be done.

II. METHODOLOGY

A. Material

The green pods of *L.leucocephala* were collected in Klang, Selangor, Malaysia. The fresh green pods chosen and washed thoroughly with distilled water to remove any impurities and dirt on the pods surface [7]. The pods were cut into small slices with the length is about 1 to 2 cm. Then, the small slices of *L.leucocephala* pods are dried in a universal oven model CSC91000 at 40°C for 7 hours to remove moisture content up to 90% [8]. The dried pods were ground using laboratory blender to obtain the powder sample and then were sieved using Endecotts Octagon 2000 Digital Sieve Shaker to get desire particle size of 125 µm [9]. Lastly, the sample powder of *L.leucocephala* pods were stored at 2 to 4°C until further use [10].

B. Moisture Content Determination

Moisture content is defined as the amount of water that contained in the sample. The determination of moisture content was conducted to identify the period of time required for the drying process. Initially, the weight of the empty glass dish and the glass dish with 1.41 g of pods were measured before undergo the drying process. The pods was placed in the oven at the temperature of 40 °C for 7 hours [9]. The weight of the pods sample was measured until the constant of moisture content was achieved. The percentage of the moisture content (MC) on the wet-weight basis (wb) was calculated using (1):

$$\text{Moisture content, \%} = (m_i-m_f)/(m_i-m_o)\text{x}100\% \quad (1)$$

Where, m_0 = the weight of empty dish (g), m_i = the weight of the dish and pods sample before drying (g), m_f = the weight of the dish and pods sample after drying (g).

C. Rapid Expansion Supercritical Solution Carbon Dioxide Extraction (RESS-CO_2)

The experimental study of the *L.leucocephala* pods extraction was carried out using RESS extractor modified from supercritical fluid extractor model SFT-100 from Supercritical Fluid Technologies, Inc. Company. The apparatus used in RESS process was shown in Fig. 1.

6 g of grinded *L.leucocephala* pods sample was filled into the cotton bag and inserted this cotton bag of sample into the 25 mL of extraction vessel. The top of an extraction vessel was sealed tightly before closed with the top cover and opened the dynamic valve while restrictor valve was closed during the extraction time. In this process, CO_2 was used as a solvent with 99.99% of purity with the constant flow rate of 24 ml/min. The pump of CO_2 was run to enter the liquid of CO_2 with the high pressure of the liquid and then will be converted to the supercritical condition of CO_2 [11]. The precipitation of the solute in the collection chamber resulted from the rapid expansion and depressurization of supercritical fluid of CO_2 through the nozzle to ambient condition [3]. Then, the extracted product will be collected in the collection vials.

The restrictor valve was opened quickly to decrease the pressure of the supercritical solution in order to separate the solute particle from the solvent through an expansion nozzle after 40 minutes of the extraction time. The solid oil particle yield of the RESS process was collected in the collection vials. The weight of the collection vials will be measured before and after the RESS process in order to calculate the extraction yield of solid oil particle from the *L.leucocephala* pods sample.

Fig. 1. RESS apparatus.

D. Gas Chromatography Mass Spectrometry (GCMS)

The characterization of the solid oil particles constituent of the RESS process of *L.leucocephala* pods were determined using Gas Chromatography Mass Spectrometry (GCMS), model Agilent Technologies, 7890A equipped with 5975G inert MSD with triple axis detector a 7683B series automatic injector made from the USA. The compounds of *L. leucocephala* pods were detected by the mass spectral library search by comparing their retention time [12]. The identification of mass spectrum of each component of *L.leucocephala* pods were used the database of the National Institute of Standard and Technology (NIST) [13].

In GCMS sample preparation, the collection vial that obtained the highest solid oil particle of *L.leucocephala* pods sample was selected after the RESS-CO_2 extraction process. A single dilution of the extracted of solid oil particle was carried out by diluted with 2 mL of hexane as a solvent for the sample preparation to undergo GCMS [14]. The dilution solution of the sample was placed in a glass vial with septa.

The significant parameters were used for GCMS analysis in order to detect the compound efficiently and accurately are a selection of capillary column, sample injection condition, column temperature conditions (Ministry of Environment, 2002). GCMS analyzer was equipped with a non-polar Perkin Elmer Elite 5 ms column. The capillary column was used with 30 m length, 0.25 diameter and 1µm film thickness [15]. The carrier gas used was helium with the constant flowrate of 1.0 ml/min. The temperature oven was at 50 °C was held for 5 min and then 5 °C/min to 250 °C. The injector of this analyzer was operated at 250 °C with 1 µL of injection volume and the split ratio was 1:10 [15].

The bioactive component of solid oil particle was analyzed using the NIST.

III. RESULTS AND DISCUSSION

A. Effects on Particle Size

Particle size is an important aspect that effected on the supercritical CO_2 extraction of the plant material. During the preliminary studies, the best particle size of 150 µm is chosen. The smallest particle size of 150 µm is selected from Fig. 2 due to the highest mass of solid oil particles that obtained is 0.1997 g (0.033%) at the selected extraction time of 40 minutes by comparing to other particle size, which are 300 µm and 500 µm that attained 0.1861 g (0.030%) and 0.1315 g (0.022%), respectively. According to Aris et al. [16], the highest extraction yield is obtained from the smallest particle size due to the higher of mass transfer. In addition, the smallest particle size is selected as it will be increased the extraction rate because of the larger surface area [16].

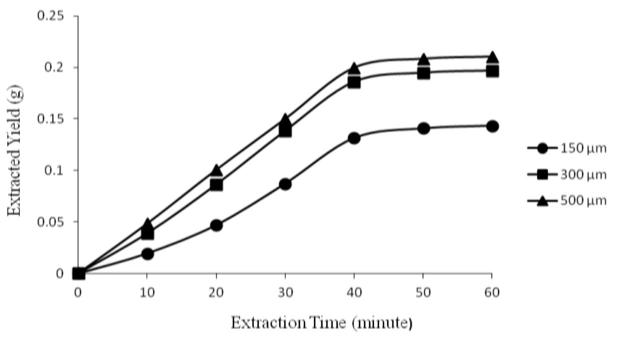

Fig. 2. Preliminary studies for the best extraction time selection.

B. Effect of Moisture Content

During the drying process, the moisture content of the *L. leucocephala* pods sample should be removed to be less than 10% in order to achieve the best CO_2 extraction. The moisture content of the *L.leucocephala* pods sample was found in this experiment is about 8.57% and the time taken for drying process is 7 hours at oven temperature of 40 °C. According to Mouahid, et al. [17], the moisture content affects to the process of supercritical CO_2 extraction due to the mass transfer of solutes in solid phase depends on its water content. The range of moisture content that should be contained in the sample is about 8 to 9% in order to obtain the optimum supercritical CO_2 extraction. The presence of the water content in the sample can act as a diffusion barrier of supercritical CO_2 extraction [18].

C. Effect of Pressure on Extraction Yield

In this section, the effect of different extraction pressure on the solid oil particle yield (%) was determined at 3000, 4000, 5000, 6000 and 7000 psi which run constantly at different extraction temperature of 40, 45, 50, 55 and 60 °C, respectively. The extraction time in the process of RESS-CO_2 takes about 40 minutes that have been selected during the preliminary study.

The effect of pressure on extraction yield at the constant temperature is graphically shown in Fig. 3. Overall, the solid oil particle yield increased as the pressure increased from 3000psi to 7000psi at the constant temperature. Rahman et al. [19] stated that the highest extracted yield from *P.speciosa* of leguminosae family gains from the increasing of extraction pressure from 3000 psi to 8000 psi at the constant temperature. The extracted yield will be increased when the pressure is increasing at constant temperature [20].

Based on Fig. 3, it can be seen clearly at the constant temperature, the extraction yield of *L.leucocephala* increased with the increase of pressure while decreasing at the lower pressure. This trend is caused by the enhancement of solvent density with the extraction pressure during the RESS-CO_2 process [21]. At the constant temperature of 60 °C, the percentage of extraction yield that attained at a different extraction pressure of 3000, 4000, 5000, 6000 and 7000 psi were 0.105, 0.16, 0.2117, 0.4667 and 0.65%, respectively. The analyte solubility and extraction yield of plant material are increasing if the increasing of extraction pressure with the fluid density is increasing at the constant temperature [22]. This because of the increasing of the density and solvating power which is due to the highest extraction forces between solute molecules of plant material and supercritical fluid [19].

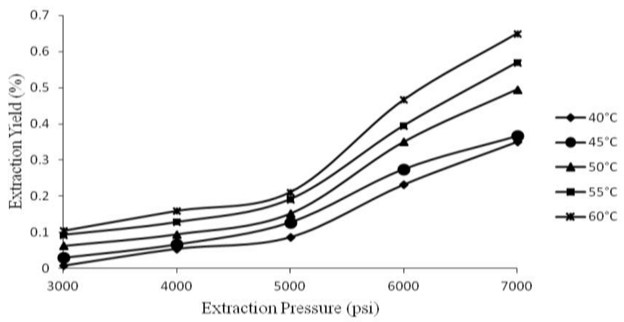

Fig. 3. Effects of Different Pressure on Solid Oil Particle Yield at Constant Temperature.

As shown in Fig. 3, the highest extraction yield of *L.leucocephala* pods sample was obtained about 0.65% at the maximum extraction condition which are pressure of 7000 psi and temperature of 60 °C. At the highest extraction pressure, the solvating power of interaction between inter-molecules and the solute is increased due to the increased of solvent density that caused by extraction pressure [16].

In the range of extraction pressure between 3000 psi and 5000 psi, extraction yield increased slowly at the constant temperature of 40 °C and 45 °C. The extraction yield increased slowly due to the reduction of diffusion rates of the extracted yield to the supercritical fluid CO_2 [23]. Meanwhile, the extraction yield increased rapidly from extraction pressure of 6000 psi to 7000 psi at the constant temperature. Sovilj [24] have studied on the extracted oil of almond seed where the extraction yield increased quickly when the pressure increased from 6000 psi to 8000 psi due to the higher solubility of extracted yield in supercritical fluid CO_2.

At the extraction pressure range between 6000 to 1000 psi, the supercritical fluid CO_2 involves heating process and in form as a dense fog where CO_2 is applied in the dense liquid state. During RESS-CO_2 process at high pressure, the high amount of fine particle produced due to the increased solubility of CO_2 with increasing density [25].

In addition, at the lower extraction pressure at 3000 psi in Fig. 3 displayed the lower extraction yield. According to Norulaini, N [19], the supercritical CO_2 extraction is poor when the extraction pressure and temperature are lower.

D. Effect of Temperature on Extraction Yield

The effect of different extraction temperature on solid oil particle yield (%) was determined at 3000, 4000, 5000, 6000 and 7000 psi at different extraction temperature of 40, 45, 50, 55, and 60 ^{o}C, respectively for 40 minutes of extraction time. The results of the temperature effect on extraction yield are illustrated in Fig. 4 on the constant pressure at extraction time of 40 minutes.

As shown in Fig. 4, the extraction yield increased with the increased of extraction temperature, which is from 40 ^{o}C to 60 ^{o}C at constant extraction pressure. The extraction temperature influence the isomerazation and degradation of extracting yield from plant material and also the increasing of extraction temperature from 40 ^{o}C to 70 ^{o}C will be increased the percentage of extraction yield [16]. Based on previous studies from Sovilj [24], the extracted yield increase when the increasing of the extraction temperature at the fixed condition of pressure and flowrate of supercritical CO_2.

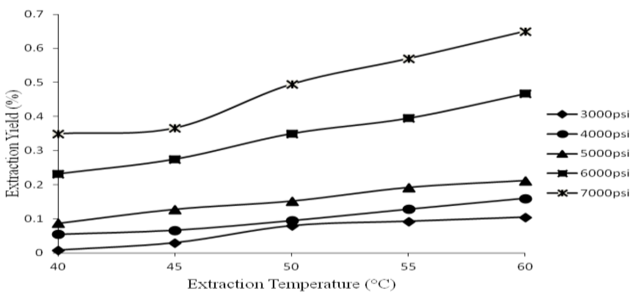

Fig. 4. Effects of different temperature on solid oil particle yield at constant pressure.

Fig. 4 shown, at the constant extraction pressure of 7000 psi, the extraction yield that attained at the extraction temperature of 40, 45, 50, 55 and 60 °C are 0.35, 0.3667, 0.495, 0.57 and 0.65%, respectively. According to Durante et al. [23], the increasing of extraction temperature at the constant pressure will decrease the supercritical CO_2 density but the vapor pressure of extracted compound will be increase that causes the increasing of extraction yield and analyte solubility. At the constant extraction pressure of 3000 psi, 4000 psi and 5000 psi, the extraction yield increase moderately in the temperature range between 40 ^{o}C to 60 ^{o}C. The slow increase of extraction yield because of the reduction in solute solubility due to the decrease of solvent density while the temperature increased [21]. Besides that, the extraction yield increased rapidly at the constant pressure of 6000 psi and 7000 psi when the temperature increased. This happens due to mass transfer of solvent is accelerated faster and thus improve the extraction yield enhancement [23]. At the constant pressure of 4000 psi, the extraction yield increases as extraction temperature increases. The higher of extraction yield resulted from the decreasing of solvent density with the increasing of temperature. The lowest extraction yield is obtained at the lowest of the constant pressure of 3000 psi with the increasing of extraction temperature. This is because of solvent density and solvent power of the supercritical fluid CO_2 decreases with the increase of temperature at low pressure [26].

E. Component Identification

The component identification of *L.leucocephala* solid oil particle has been analyzed by using Gas Chromatography Mass Spectrometry (GCMS), model Agilent Technologies. The component was determined by using the NIST database as shown in Fig. 5. In the component analysis, the highest yield of solid oil particle was selected to undergo the GCMS analysis which was operated at the temperature of 60 ^{o}C and pressure of 7000 psi.

In this study, the major component detected through GCMS analysis are 2,2,3-trimethyldecane, cyclopentanone, propanoic acid and cyclobutane, 1,2-diphenyl. According to Mohd Azizi et al. [27], the major compound that found *in P.speciosa* seeds extracts was propanoic acid that used as an antifungal agent in food and present naturally in dairy product at low quantities.

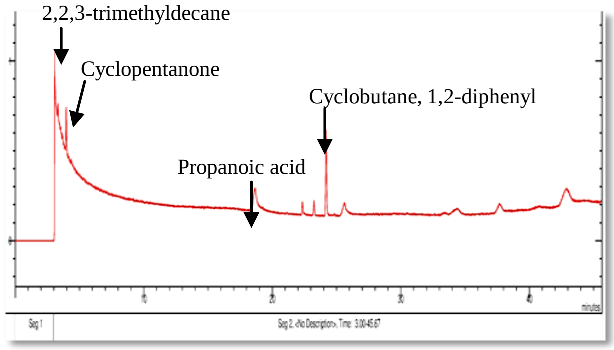

Fig. 5. Components Identification of L.leucocephala Solid Oil Particle using GCMS.

IV. CONCLUSION

The main objective of this study is to investigate the potential of RESS-CO_2 extraction to obtain the solid oil particle from the *L.leucocephala* pods. This objective was achieved successfully by determining the best operating conditions for the smallest particle and the highest solid oil particle yield that attained from RESS process. Then, the component characterization of solid oil particle yield was conducted using GCMS. From this preliminary analysis, the smallest particle size of 150 µm was selected as a best particle size because it gained the highest extraction yield of 0.2105 g. Meanwhile, the best extraction time has been determined at 40 minutes by plotting the graph of time versus extraction yield where this graph shown clearly that the extraction yield is constant and became a steady line after 40 minutes. Besides that, the solid oil particle obtained from the RESS process by determining the effect of extraction pressure and temperature on the extraction yield. This study was conducted at the extraction pressure of 3000 psi, 4000 psi, 5000 psi, 6000 psi and 7000 psi and extraction temperature of 40 ºC, 45 ºC, 50 ºC, 55 ºC and 60 ºC. In conclusion, the highest yield that attained from this study is about 0.65% at 60 ^{o}C and 7000 psi.

From the GCMS analysis, the major component detected were propanoic acid, 2,2,3-trimethyldecane, cyclopentanone and Cyclobutane, 1,2-diphenyl.

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