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Supercritical carbon dioxide (SC-CO_2) extraction of essential oil from *Swietenia mahagoni* seeds

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Abstract. *Swietenia mahagoni* (Mahogany) is a traditional plant that is rich with bioactive compounds. In this study, process parameters such as particle size, extraction time, solvent flowrate, temperature and pressure were studied on the extraction of essential oil from *Swietenia mahagoni* seeds by using supercritical carbon dioxide (SC-CO2) extraction. *Swietenia mahagoni* seeds was extracted at a pressure of 20-30 MPa and a temperature of 40-60°C. The effect of particle size on overall extraction of essential oil was done at 30 MPa and 50°C while the extraction time of essential oil at various temperatures and at a constant pressure of 30 MPa was studied. Meanwhile, the effect of flowrate CO_2 was determined at the flowrate of 2, 3 and 4 ml/min. From the experimental data, the extraction time of 120 minutes, particle size of 0.5 mm, the flowrate of CO_2 of 4 ml/min, at a pressure of 30 MPa and the temperature of 60°C were the best conditions to obtain the highest yield of essential oil.

1. Introduction

Swietenia mahagoni is also known as 'tunjuk langit' in Malaysia is used traditionally to treat various diseases such as diabetes and high blood pressure [1]. *S. mahagoni* tree is 30 meters or taller [2] and the wood, usually being used for making furniture [3]. Meanwhile, the bark can be used for natural colorant [4]. The fruit of *S. mahagoni* is woody and consisting of capsules containing winged seeds [5]. Whereas, the seed of *S. mahagoni* can be obtained by removing the wing. In Malaysia, the raw seeds were used for treating hypertension and diabetes [6]. In addition, *S. mahagoni* seeds have been reported to have various biological activities such as anti-inflammatory activity, anticancer and antitumor activity [7] and also antidiabetic activity [8].

Also, carbon dioxide (CO_2) is the most frequent solvent used that is environmental friendly (fairly non-toxic), low cost and can be easily removed from the extract [9]. The elimination of CO_2 is easily achieved since CO_2 is in a gas state at room temperature. In addition, CO_2 in the supercritical state is in a moderate critical temperature (31.3°C) and pressure (7.38 MPa). Supercritical state is when gas and liquid are indistinguishable where at this state it is compressible but possessing a density of a liquid. In a word, supercritical CO_2 makes a good solvent because of the gas-like state that attributed the low viscosity and high diffusion coefficient and the liquid-like state that gave the solvating power [10].

2. Methodology

2.1. Materials and reagents

Swietenia mahagoni seeds were bought in the local market. The seeds were rinsed with tap water to remove any foreign particles and dirt prior to drying. Then, the cleaned seeds were cut into small pieces and dried by using oven at temperature of 50°C for a week to remove moistures. The seeds were ground by using a blender (Panasonic) and sieved to approximate 0.25, 0.50 and 0.75 mm of particle size. Commercial

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grade liquid carbon dioxide (purity 99.99%) used in supercritical carbon dioxide extraction was purchased from Kras, Instrument and Services, Johor, Malaysia.

2.2. Supercritical Carbon dioxide (SC-CO_2) extraction

CLEAR supercritical fluid extraction (SFE) machine in Center of Lipids Engineering and Applied Research (CLEAR), Universiti Teknologi Malaysia consisted of CO_2 gas cylinder, CO_2 controller pump (Lab Alliance), co-solvent pump (Lab Alliance), oven (Memmert, Germany), 10 ml stainless steel extraction vessel, pressure gauge (Swagelockk, Germany), automatic back pressure regulator (Jasco BP 2080- Plus) and restrictor valve. A schematic diagram of CLEAR SC-CO_2 unit is illustrated in figure 1.

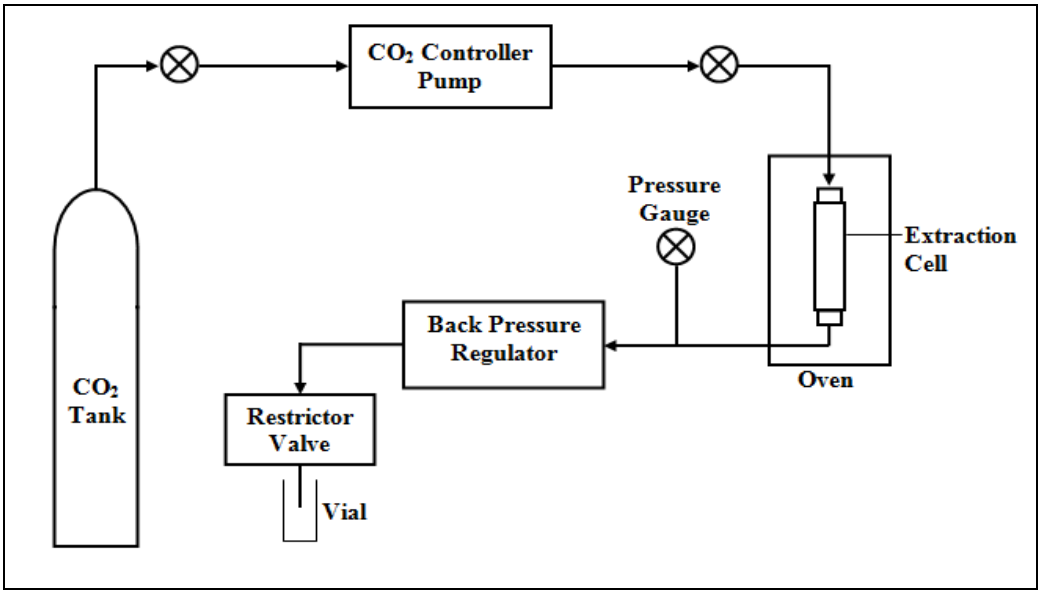


Figure 1. The schematic design of the SC-CO_2 unit

Five gram of sample was placed in 10 ml stainless steel extraction vessel and sealed tightly in the oven. Set all the parameters (temperature, pressure and flowrate of CO_2), the extraction process started after all the parameters were attained. Lastly, depressurized the system and the oil yields were collected. The parameters used in extraction process is presented in table 1.

Table 1. The process parameters for SC-CO_2 extraction

Parameter	Range/ value
Temperature (°C)	40-60
Pressure (MPa)	20-30
Flowrate of CO_2 (ml/min)	2.00-4.00
Particle size (mm)	0.25-0.75
Mass of sample (g)	5.00
Extraction time (min)	0-180

Collected oil yield was calculated as percentage of oil yield by using equation below:

$$\text{Oil yield (\%)} = \frac{\text{Mass of oil extract (g)}}{\text{Mass of sample (g)}} \times 100 \quad \text{(Eq. 1)}$$

3. Result and Discussion

3.1. Particle size

Particle size is one of the important factors to be considered in the extraction of *S. mahagoni* seeds. In a word, as the surface area increases, the extraction rate also increases. Hence, smaller particle size will increase the extraction yield because the mass transfer will increase with smaller particle size of the sample. But the downside is as the sample is ground to small, the extraction rate may decrease due to channeling

effect inside the extraction vessel [11]. Channeling effect in the extraction vessel is due to over fine particle size of the sample that reduce the contact between the sample and solvent. Supported by Salleh (2010), fine powder with smaller particle size can increase the extraction rate but it is also lead to the difficulty in maintaining the flow rate to obtain a good optimization of SC-CO_2 extraction [12].

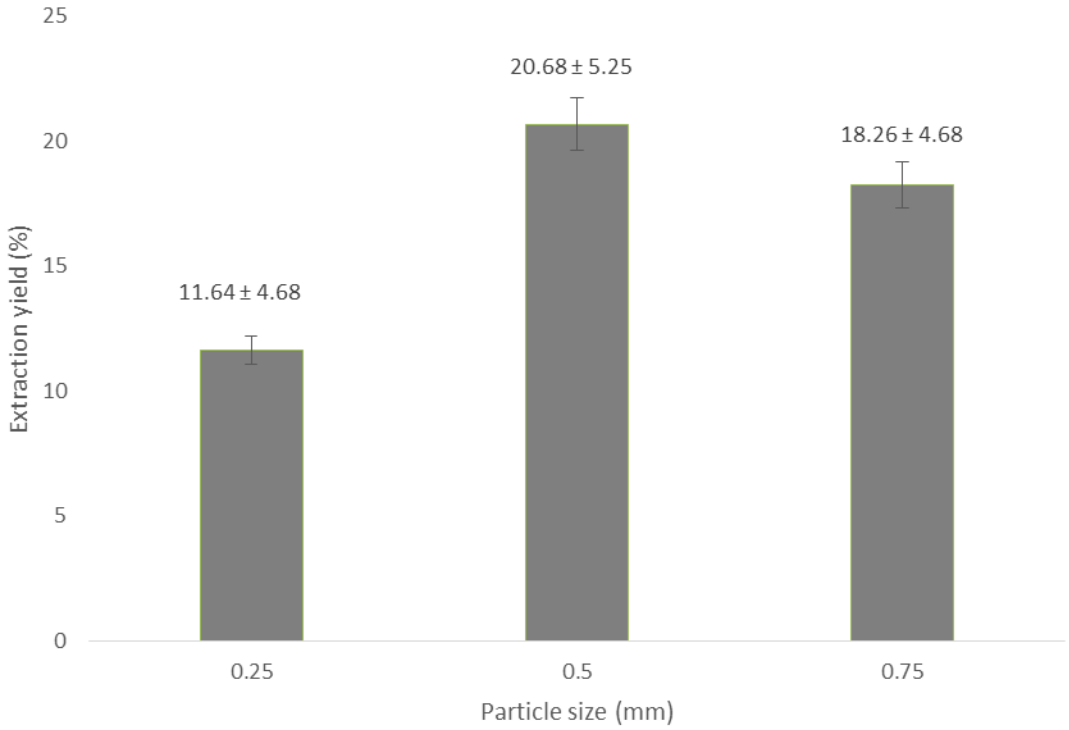


Figure 2. Effect of particle size on extraction yield

Figure 2 shows that particle size of 0.5 mm gave the highest percentage of oil yield which is 20.68% ± 5.25 compared to 0.75 mm and 0.25 mm. From the figure, it shows that when the particle size is too small, the extraction yield is decreased. This is because when the particle size is too fine, the porosity of the sample is reduced and slower CO_2 penetration into the sample, hence lower the extraction yield. In this study, *S. mahagoni* seeds are naturally oily thus it may easily clumped together in the extraction vessel hence reduce the surface area. Similarly with the study in the extraction of rubber seed, smaller particle size (≤0.355 mm) can easily clump together, however larger particle size (0.50mm) do not clump together easily and provide larger surface area [13]. It is resulted that the particle size of 0.5 mm is considered for this SC-CO2 extraction.

3.2. Extraction time

The determination of the extraction time for the extraction of *S. mahagoni* seeds by using SC-CO_2 extraction was performed. The conditions used were pressure of 20, 25 and 30 MPa, with the temperature of 50°C. The flow rate used in this study was 2 ml/min. The yield of extract was collected every 20 minutes over 180 minutes of extraction time. The results of the best extraction time are presented in Figure 3.

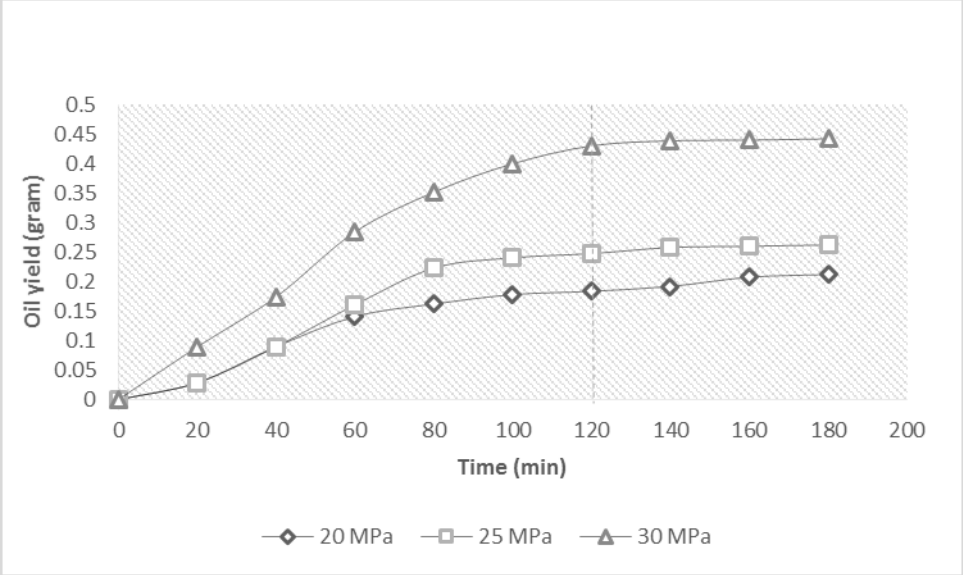


Figure 3. The effect of extraction time on extraction yield of SC-CO_2 at 20, 25 and 30 MPa and the temperature of 50°C

The figure displays the oil yield in percent versus the extraction time in minutes. At pressure of 20 MPa, it took more than 120 minutes to achieve the asymptomatic value. The highest yield (0.4306%) was at the highest pressure of 30 MPa within 120 minutes. Lina *et al.*, (2010) also obtained similar result with this study where the seed oil of *Microula sikkimensis* increased as extraction time increased [14]. The same trend was found in the study of saffron extract by Lozano *et al*. (2000) [15]. In conclusion, the extraction time of *S. mahagoni* seeds was performed at 120 min constantly for each sample throughout this study in order to obtain asymptotic value to ensure maximum extraction yield was extracted.

3.3. Flowrate

Flow rate of solvent (example: CO_2) is another factor affecting the extraction process to produce a high yield of extract by using supercritical carbon dioxide (CO_2) [16]. By increasing the flow rate of solvent, also reduce the extraction time and mass transfer resistance.

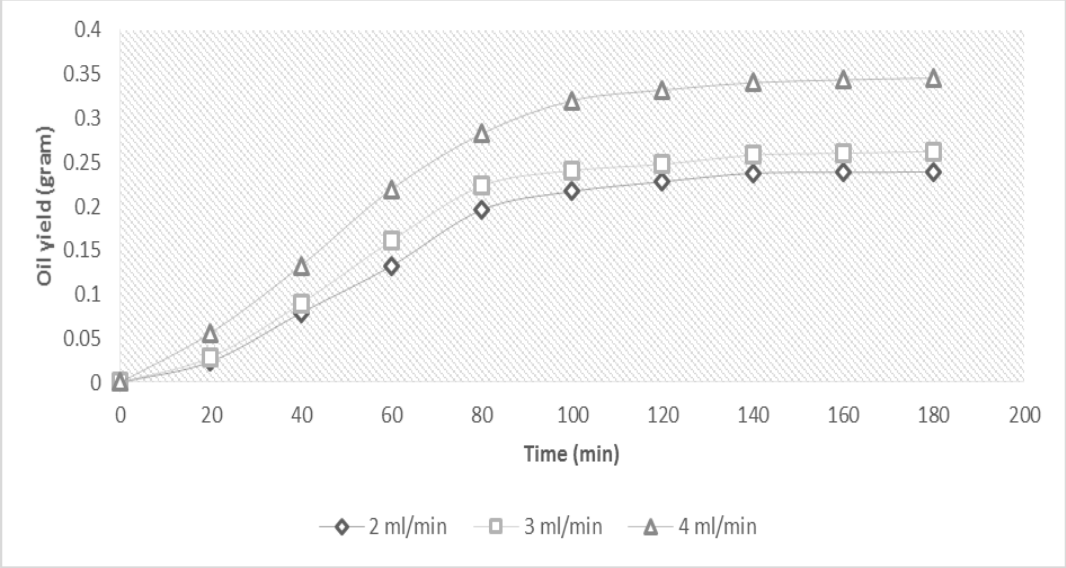


Figure 4. Effect of flow rate on the overall oil yield against extraction time at the pressure of 30 MPa and temperature of 50ºC

As shown in figure 4, the overall oil yield increase with the increasing of flow rate at constant pressure of 30 MPa and temperature of 50ºC. The extraction of borage oil by SC-CO_2 extraction showed higher flow rate gives higher yield of borage seed oil [17]. Similarly, in the extraction of flaxseed showed that at higher

flow rate (3 L/min) recovered higher oil yield [18]. Furthermore, by increasing the SC-CO_2 flowrate, the intermolecular interaction between solvent and the solute increase due to the increase of CO_2 molecules per volume in the extraction vessel [19]. In this condition, the mass transfer increases as the flowrate increases. Hence, 4 ml/min of CO_2 was selected as the best flowrate for the extraction of *S. mahagoni* seeds.

3.4. Temperature and Pressure

The extraction of *S. mahagoni* seeds by using SC-CO_2 extraction shows that the highest yield (14.45 %) obtained was at the maximum level (30MPa, 60°C) meanwhile lowest (1.49%) at (20MPa, 60°C) as shown in Table 2. At higher temperature, the decomposition of cell walls occurred thus extracted oil produced is higher [19]. Operating at higher pressure will influence the solvent density, thus enhanced the solvating power (the interaction of inter-molecules and solutes increase) [11]. Similar result was reported in the extraction of *S. mahagoni* seeds by using SC-CO_2 extraction [20].

Table 2. Extraction of *S. mahagoni* seeds at different conditions (pressure, P and temperature, T)

Condition (s)	Oil yield (%)
P= 20MPa, T= 40°C	6.556
P= 20MPa, T= 50°C	3.676
P= 20MPa, T= 60°C	1.490
P= 25MPa, T= 40°C	6.640
P= 25MPa, T= 50°C	4.954
P= 25MPa, T= 60°C	4.558
P= 30MPa, T= 40°C	7.024
P= 30MPa, T= 50°C	8.612
P= 30MPa, T= 60°C	14.45

Figure 5 shows the effect of pressure and temperature on oil extraction. At constant temperature, oil yield increases as pressure increases (20-30 MPa). This is due to the increase of density and thus increase the solvating power of SC-CO_2. Effect of pressure influenced both solvating power and intermolecular interaction strength [21,22]. Similar result was reported in SC-CO_2 extraction of green coffee oil [23], wheat bran [24] and *S. mahagoni* seeds [20].

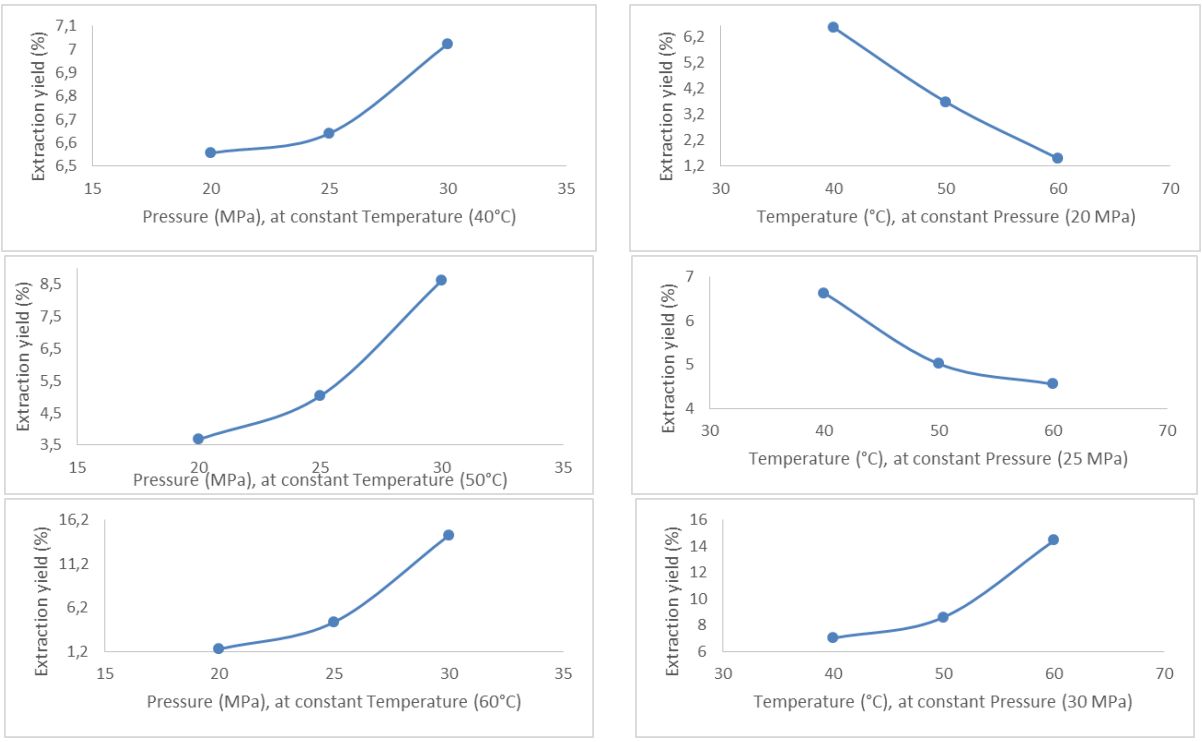


Figure 5. The effect of pressure and temperature on extraction yield

Next, at constant pressure (20 and 25 MPa), oil yield decreases as temperature increases (40-60 ºC). But at constant pressure (30 MPa), oil yield increases as temperature increases. Effect of temperature in extraction process is much more difficult to evaluate unlike pressure. This is due to the dual effect of temperature toward extraction process depending on the domination of solute vapour pressure or solvent density [19,25,26]. Decreasing solvent density with the increase in temperature resulted in higher extracted yield because of the increase in vapour pressure. Similar effect were reported on extraction green coffee oil [23] and silkworm pupal oil [26].

4. Conclusion

Supercritical carbon dioxide (SC-CO_2) extraction applied to extract *Swietenia mahagoni*. From the experimental data, the extraction time of 120 minutes, particle size of 0.5 mm, the flowrate of CO_2 of 4 ml/min, at a pressure of 30 MPa and the temperature of 60°C were the best conditions to obtain the highest yield of essential oil.

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