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Effect of Pressure on Essential Oil Yield of Chromolaena Odorata Leaves Extract using Supercritical Fluid Carbon Dioxide

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ABSTRACT

Most of the previous researchers used conventional extraction technique such as steam distillation and solvent extraction method in Chromolaena Odorata extraction. Therefore, in this study a clean extraction method that is Supercritical Fluid Extraction (SFE) technology using supercritical carbon dioxide as a solvent (SC-CO2) was introduced for Chromolaena Odorata leaves extraction to produce oil of bioactive components. The objective of this research was to find the optimum pressure for the highest oil yield extracted. The extraction was conducted within a range of temperature (40, 45 and 50°C) and pressure (3000, 3500, 4000, 4500 and 5000psi), at a constant flowrate (24 ml/min) for 10, 20, 30, 40, 50 and 60 minutes of extraction time. From the studies, it was found that extraction pressure is an important factor that can affect the extraction yield where the oil yield increased as the pressure increased due to the increasing amount of CO2 consumed where it boost the density of the solvent to interact with the solute. The highest oil yield is 51.20% at pressure of 4000psi and temperature 50°C. Whereas at the highest pressure of 5000psi, the oil yield is the lowest. This shows that the extraction is not suitable at pressure 5000psi.

1. Introduction

Chromolaena odorata is a sort of herbal plant with the height of 1.5 to 2.0m that can form bushes [1]. Several researches have been made on Chromolaena odorata plant in early 1990s until now. The studies on Chromolaena odorata were done in order to extract the essential oil and to find the main components exist in the oil. The parts of this plant such as leaves, stem and even roots have been used to do some researches. Globally, Chromolaena odorata has been used as a traditional medicine with 80% of world population in Asia and Africa used this plants as traditional medicine [2]. For example, it is used to treat colds, coughs, skin diseases, heal wound and as an antiseptic agent [3,4]. Nowadays, Chromolaena Odorata extract received great attention due to the increasing application of natural antioxidant in pharmaceutical products. Many studies acknowledged the high amount of

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natural antioxidant content in Chromolaena odorata as a valuable component to replace synthetic antioxidant that can be used in anticancer activity for cancer treatment. Besides that, antioxidant components such as α-pinene, β-pinene stored in Chromolaena odorata leaves are beneficial to pharmaceutical industries. Moreover, the artificial antioxidants are doubtful on their safety issues, hence for this reason attentions are focused on natural antioxidants. Table 1 shows the main components exist in Chromolaena odorata in several studies. The table shows that the leaves have the highest amount of main components detected in the oil extracted.

Table 1
Main Components in Oil Extraction from Chromolaena Odorata

Plant parts	Main Components	References
Leaves	α-pinene β-pinene Germacrene D Caryophyllene Pregeijerene	Owolabi *et al.* [3], Félicien et al. [5]
Roots	Himachol 7-isopropyl-1,4-dimethyl-2-azulnol Androeencecalinol 2-methyoxy-6-(1-methoxy-2-2-propenyl)	Joshi [4]
Stem	α-pinene β-pinene Bicycle[7.2.0]undecene Caryophyllene Germacrene D	Olusegun and Musa [6]

Usually, conventional extraction method such as hydrodistillation and solvent extraction method are used in extracting oil from Chromolaena odorata [3,4,7]. However, there are disadvantages for each of these long-established methods for oil extraction. As stated by Illés *et al*., [8], hydrodistillation extraction method chemically altered the essential oil of the plants extracted. Moreover, the compounds that are sensitive to heat can be destroyed easily which make the quality of the oil to be low. In the case of soxhlet extraction, the disadvantages are that agitation is impossible to take place, the valuable volatiles can be lost during evaporation of solvent, the chances of thermal decomposition must not be ignored due to lengthy extraction time and the oil extracted is not solvent-free [8,9]. In addition, these two extraction methods are using chemical like hexane which may leave residue and have some level of environmental contamination in the extract [10].

Consequently, in this study a clean extraction method which is Supercritical Fluid Extraction with CO2 (SFE-CO_2) as a solvent was introduced for the extraction of Chromolaena odorata oil. Recent developments in extraction technology such as Supercritical Fluid Extraction (SFE) method allowed extraction of bioactive components of herb to be produced in form of essential oil. SFE with CO_2 as a solvent offers a lot of beneficial compared to conventional extraction method such as nontoxic, non-flammable, environmental friendly method, easy to separate the solvent, mild operating conditions, short extraction time, and offers high quality of final product [10-13]. As stated by Handa [14] SFE is used as an alternative extraction method which intend to reduce the use of solvents while increasing the sample. Pourmortazvi and Hajimirsadeghi [15] stated that more than 90% of SFE is carried out by using carbon dioxide (CO_2) due to its low critical temperature (31.1°C) and low critical pressure (7.38MPa). It is where CO_2 in liquid and gas phase disappear and form one single supercritical phase.

The parameters on conducting SFE were referred by previous studies. As an example, study conducted by Yamini *et al*., [16] on oil extraction of Salvia Mirzayanii was done within the range of temperature 35-70°C and pressure 1464-5149 psi for a duration of 15-35 minutes. Extraction of cardamom oil by SC-CO2 were conducted by Hamdan *et al*., [17] at the range of temperature 35-50°C and pressure in between 1450-4351 psi. In recent times, Zeković *et al*., [18] have done an optimization of coriander seed using SC-CO2 at which they chose to use temperature 40, 55 and 70°C and pressure of 1450, 2175.6 and 2900 psi. By using these ranges of temperature and pressure, the above researchers have found optimum performance of oil yield and solubility

As for yield, it is important to find the optimum parameters for extracting the best quality oil. Based on study done by Liu *et al*., [19], the oil yield increases when the pressure is increased along with the extraction time. Besides that, the oil yield will also increases as the temperature increases. According to Grosso *et al*., [20], oil yield is the percentage in gram of weight to the gram of dry weight (%g oil/g sample).

2. Methodology

2.1 Sample Preparation

The *Chromoalaena odorata* leaves used in this study were obtained from local of several batches. The leaves were cleaned thoroughly with water to remove any impurities. After that, the leaves were air dried to remove any moisture before grinded using Herb Grinder Medical Industry 2 HP from Buyamag Inc. to obtain powdered *Chromoalaena odorata* leaves. By using Sieve Shaker Model Retsch AS200, the powdered leaves were then sieved to obtain the size of 0.25mm.

2.2 Supercritical Carbon Dioxide (SC-CO_2) Extraction

SC-CO_2 extraction of *Chromolaena odorata* leaves was performed using SFE Model Thar SFC from Thar Process (USA) available in Faculty of Chemical Engineering, UiTM Pulau Pinang. The studies SC-CO_2 extraction were carried out at operating temperature of 40, 45 and 50°C and pressure of 3000, 3500, 4000, 4500 and 5000 psi. The maximum mass of sample that can be loaded in the extraction vessel is 10g. This Thar SFC equipment was designed specifically to operate at maximum temperature of 90°C and maximum pressure of 5800 psi.

Desired extraction temperature and pressure were set, and 10g of the *Chromolaena odorata* sample was inserted in a cotton bag. The cotton bag containing the sample was inserted in the extraction vessel and made sure that the cotton bag did not touch the top of the extraction vessel before sealing the top tightly. On/off valve (MV1) and CO_2 cylinder valve were fully opened, and valves (CO_2 bleed valve) MV2, (drain valve) MV3 and (co-solvent release valve) MV4 were fully closed during the extraction time. MV4 were closed because no co-solvent was used in this study.

When the extraction temperature achieved the desired value, CO_2 pump was run to feed high pressure liquid CO_2 (99 % purity provided by MOX Linde Gases Sdn Bhd) continuously in the extraction vessel at a fixed solvent flowrate of 24 ml/min. Basically, liquid CO_2 will be converted to a supercritical condition when it is pump into the extraction vessel (heated zone). To achieve the desired pressure set point, CO_2 pump will continuously actuate. After reaching the extraction time, all the pumps were stopped and valve MV1 was closed. The extraction product was collected in a beaker by opening valve MV3 slowly until the pressure drop to 0 psi. After all the extract was collected, valve MV1 and MV2 were opened to release the CO_2. The connections of valve MV1 to the extraction vessel top were disconnected and the sample was taken out. Depressurized CO_2 at the ambient pressure will convert into gaseous form and purged into the ambient. Extraction product will be collected in collection vials.

2.3 Determination of Oil Yield

To determine the extraction yield, weight of the collection vials will be measured before and after SC-CO_2 process. The extraction product was weighed using the analytical balance model Mettler Toledo AB204-S for oil yield determination. The extraction yield is defined as the mass of the extracted oil divide by the mass of the ground sample loaded in the extraction column. The oil yield was determined using equation (1) according to Oliveira *et al*., [12].

$$Oil\ yield\ (\%) = \frac{Mass\ of\ oil\ extract\ (g)}{Initial\ Chromolaena\ odorata\ sample\ (g)} \quad (1)$$

3. Results and Discussion

In this study, the effect of different pressure (3000, 3500, 4000, 4500 and 5000psi) on extraction oil yield was investigated at three different constant extraction temperatures of 40, 45 and 50°C. The oil extract was collected at 10, 20, 30, 40, 50 and 60 minutes of extraction time. According to Sapkale *et al*. [10], the time for extraction using SFE is quick which is mostly takes less than 60 minutes compared to soxhlet extraction that take up to 6 to 48 hours to extract oil. By using equation (1.1), the oil yields of every sample were collected on every 10, 20, 30, 40, 50 and 60 minutes. Table 2 below shows the extraction oil yield percentage obtained at different extraction pressure with three different temperatures. The effects of extraction pressure on oil yield were plotted as shown in Figure 1, Figure 2 and Figure 3.

Figure 1 shows the effect of pressure on extraction oil yield at a constant temperature of 40°C. In the figure, the oil yield increased with the extraction time. It can be observed from Figure 1 that the highest oil yield was 27.60% which was obtained at extraction pressure 4000psi and the lowest oil yield was 6.20% at extraction pressure 3500psi. The extremely low oil yield may be due to low CO_2 density during the experiment.

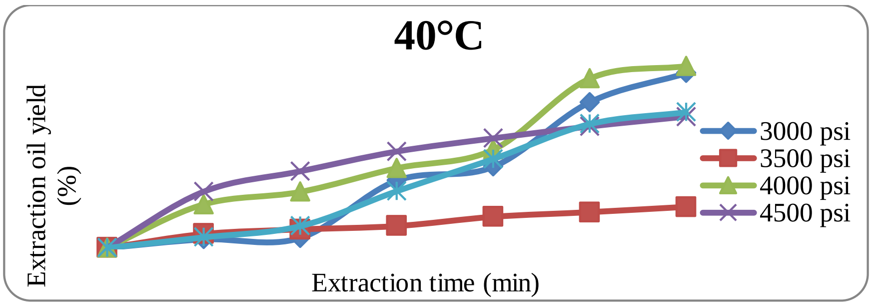

Fig. 1. Effect of Different Pressure on Extraction Oil Yield at constant temperature of 40°C

For a constant temperature of 45°C, the effects of pressure on extraction oil yield were shown in Figure 2 where the highest oil yields was 48.90% obtained at pressure of 4000psi. Whereas the lowest oil yield was 30.70% which was obtained at extraction pressure 5000psi. On the other hand, the effects of different pressure on extraction oil yield at constant temperature, 50°C are shown in Figure

3. From the figure, the extraction oil yield at 4000psi is the highest at extraction time of 60 minutes that was 51.20%. In contrast, the lowest oil yield at 60 minutes was 32.40% at pressure 5000psi. Based on the figures, it can be observed that the overall highest oil yields are obtained at extraction pressure of 4000psi for temperature 40, 45 and 50°C. Based on the study done by Liu *et al*. [19], the oil yield increased with pressure as there were improvement in the solubility of the oil which affected the solvent density for it to increased. However, the lowest oil yields are shown at pressure 5000psi for both temperatures 45 and 50°C which may be due to the variations of oil content in *Chromolaena odorata from the different batches* as stated by Hamdan *et al*., [17]. Besides that, the oil yield for every pressure increased as the temperature increased. As an example, the highest oil yields are at pressure 4000psi with the value of 27.60%, 48.90% and 51.20% for temperature 40, 45 and 50°C respectively.

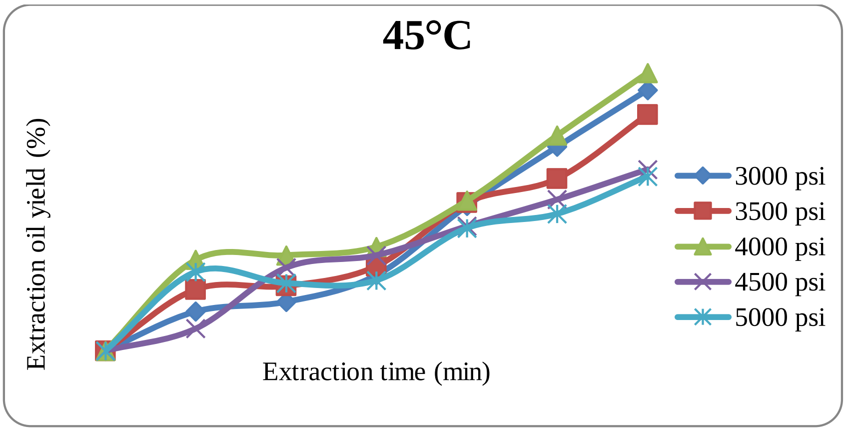

Fig. 2. Effect of Different Pressure on Extraction Oil Yield at constant temperature of 45°C

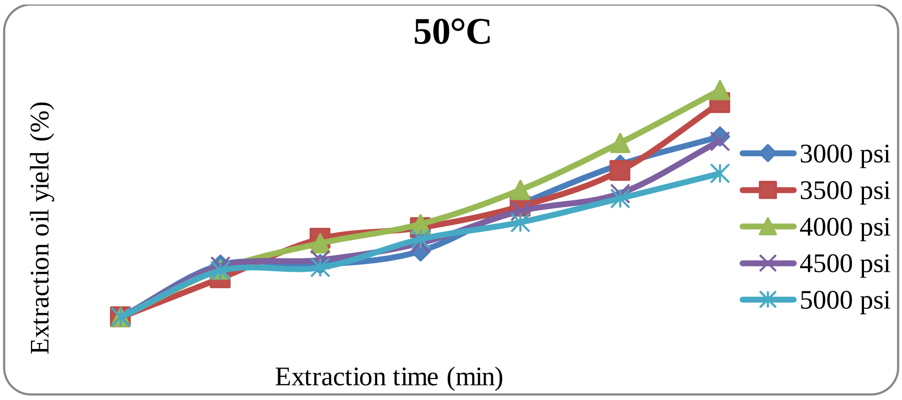

Fig. 3. Effect of Different Pressure on Extraction Oil Yield at constant temperature of 50°C

Table 2
Percentage of Oil Yield Obtained at Different Pressure with Constant Temperature

Temperature (°C)	Pressure (psi)	Oil yield (%)					
		10 min	20 min	30 min	40 min	50 min	60 min
40	3000	1.30	1.50	10.20	12.30	22.10	26.50
	3500	2.10	2.80	3.30	4.70	5.40	6.20
	4000	6.60	8.50	12.10	14.90	25.70	27.60
	4500	8.50	11.60	14.60	16.60	18.40	19.90
	5000	1.60	3.30	8.60	13.50	18.80	20.60
45	3000	6.90	8.60	13.30	25.50	35.90	45.90
	3500	10.80	11.40	14.90	26.10	30.30	41.60
	4000	16.00	16.80	18.30	26.40	37.90	48.90
	4500	3.90	14.60	16.80	21.90	26.60	31.90
	5000	13.90	11.90	12.40	21.60	24.10	30.70
50	3000	11.80	11.90	14.90	25.40	34.40	40.70
	3500	8.80	17.70	20.20	25.00	33.00	48.30
	4000	10.80	16.70	21.00	28.70	39.30	51.20
	4500	11.50	12.90	16.70	23.90	27.90	39.70
	5000	10.50	11.20	17.60	21.40	26.80	32.40

4. Conclusion

The aim of this study is to extract the *Chromolaena odorata* oil from dried *Chromolaena odorata* leaves using Supercritical Extraction Solvent-CO_2 technique and to determine the effect of extraction pressure on the extraction yield. The influence of pressure and temperature on extraction yield was investigated at 3000, 3500, 4000, 4500 and 5000psi; and at 40, 45 and 50°C. From the conducted studies, it was proven that extraction pressure is an important factor that can affect the extraction yield, since increasing the extraction pressure will increase the extraction yield at constant temperature. The highest oil yield is 51.20% at pressure of 4000psi and temperature 50°C. Whereas, at the highest pressure of 5000psi, the oil yield is the lowest due to the varieties of oil content in the raw materials since it is from different batches. This shows that the extraction is not suitable at pressure 5000psi and the most suitable at 4000psi.

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