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Kinetics of cinnamon oleoresin extraction using Microwave-Assisted Extractor

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Abstract. Cinnamon oleoresin can be produced from the solvent extraction of cinnamon (*Cinnamomum burmannii*) bark. Among many extraction method, Microwave Assisted Extraction (MAE) is one method that gives many advantages, especially in reducing the extraction time. This research will compare the kinetic models of MAE extraction process, using first and second order model. Extraction were carried out from 5 to 40 minutes with eight cycles and analyzed its oleoresin content. For comparison, soxhlet extraction method was also done for 1 and 4 hours. From the analysis, it was shown that the second order kinetic model was more suitable to explain the MAE of cinnamon oleoresin. The 40 minutes MAE process gave 9.55 g L⁻¹ oleoresin, while soxhlet extraction gave 3.76 g L⁻¹ and 9.48 g L⁻¹ oleoresin in 1 and 4 hours respectively. SEM analysis for both of MAE and soxhlet treated samples showed the rupture of the cell and therefore caused the release of cell content. The cell structure in 35 min-MAE treated sample was more opened than the 1 hr-soxhlet treated one. It means that the cell rupture in MAE process was quicker than the soxhlet, and it produced the high yield in shorter time.

1. Introduction

Cinnamon (*Cinnamomum burmannii*) is one of the herb and spice that had been used by the people for long time ago. Its applications were mainly for cosmetic and flavoring. Nowadays, it was revealed that cinnamon also has active compound and therefore its application was broader. Cinnamon may contains flavonoid, antioxidant [1] and antibacterial compound [2] and was considered as a functional food and a remedy for respiratory and digestive problems [3]. Indonesia is one of the world's high producer of cinnamon with total export quantity of cinnamon was the largest in the world [4].

Almost every part of cinnamon plant can be used, but the mostly used is its bark. It can be processed to obtain essential oil or oleoresin. Essential oil is produced by hydrodistillation process, while oleoresin is gained through solvent extraction [1]. Oleoresin is usually more stable than its essential oil since it contains natural antioxidant. Moreover, oleoresin needs less place of storage than its corresponding spice and also have better distribution [5].



There are many extraction method for gaining the oleoresin, they are maceration, soxhlet extraction and percolation. Microwave Assisted Extraction (MAE) was one promising alternatif for oleoresin extraction. MAE was considered as a rapid process, will produce high yield in shorter time and also need less energy and solvent [6-7]. However, there were still little studies about the MAE for cinnamon oleoresin. Therefore, the paper explores the kinetics of cinnamon oleoresin extraction using Microwave-Assisted Extractor.

2. Experimental

2.1. Material

Cinnamon bark as raw material, was gotten from local market in Semarang. The sample was dried in oven for 4 hr and then was grinded to get the 80 mesh powder. The powder was kept in sealed container until being used. Ethanol with concentration of 96% was the solvent for the extraction process, was gotten from PT. Brataco Chemical Indonesia.

2.2. Extraction of cinnamon oleoresin

The extraction process was conducted using modified domestic microwave oven from SHARP Model R-230R(S). One liter flask was used and equipped with condensor on top of it. Ten gram of cinnamon sample powder was added with ethanol, while the solvent to solute ratio was 26 ml/g. The power was 30% from the maximum power of microwave (max power is 399 Watt). Eight extraction cycles were carried out from 5 to 40 minutes. After the extraction, oleoresin was separated from its solvent using rotary evaporator. The yield of oleoresin was counted using equation (1). For the comparison, soxhlet extraction was also done for 1 and 4 hr. The sample weight, kind of solvent and solvent to solute ratio used were same with the microwave process.

$$\text{Yield (\%)} = \frac{\text{mass of oleoresin (g)}}{\text{mass of sample (g)}} \times 100 \% \quad (1)$$

2.3. First Order Extraction Kinetic Model

The differential form of first order kinetic model [8] can be written as in equation (2).

$$\frac{dC_t}{dt} = k_1 (C_s - C_t) \quad (2)$$

Where k_1 is the first order constant rate, t is time, C_t is oleoresin concentration at time t and C_s is oleoresin concentration at saturation. The integration and rearrangement to linear form of equation (2) with the boundary condition $C_t = 0$ at $t=0$ and $C_t = C_t$ at $t=t$ is :

$$\log(C_s - C_t) = \log(C_s) - \frac{k_1}{2.303} t \quad (3)$$

Plot of $\log (C_s - C_t)$ against t will give the k_1 from the slope and concentration at saturation (C_s) from the intercept.

2.4. Second Order Extraction Kinetic Model [8-9]

The differential form of the second order is :

$$\frac{dC_t}{dt} = k_2(C_s - C_t)^2 \quad (4)$$

with k_2 is the second order constant rate. Integration and rearrangement to linear form of equation (4) will result as follows :

$$\frac{t}{C_t} = \frac{t}{C_s} + \frac{1}{k_2 C_s^2} \quad (5)$$

Concentration at saturation and constant rate can be determined from the slope and intercept of the t/C_t versus t curve.

2.5. SEM Analysis

In order to know the structure of cinnamon powder after extraction, 35 minutes extraction sample was taking in the end of extraction to take SEM analysis, using SEM JEOL type JSM 6510 LA. Sample from 1 hr soxhlet extraction also take the SEM analysis to compare with the microwave process.

3. Result and Discussion

The experimental result of microwave assisted extraction from cinnamon was shown in Figure 1. The figure showed the concentration of oleoresin in different time. From the figure we can see that the concentration of oleoresin was increased rapidly in the beginning of the process then the increasement was slow down with the time. The concentration increase rapidly within 10 minutes, reached around 6 g L⁻¹ of oleoresin. After 10 minutes, the concentration increased slowly, and became almost constant in around 30 minutes. Total concentration of oleoresin got in 40 minutes extraction time was 9.55 g L⁻¹. The same typical curves were also got from MAE of sandalwood oil [8], MAE of polyphenols from *Clinacanthus nutans* [10] and MAE of olive cake oil [11]. The extraction process took place in three steps, they were substrate removal of the outer surface of particles followed by transition phase to diffusion, where convection and diffusion happened and the last step was the escape of solute from matrix and diffused to the solvent [8].

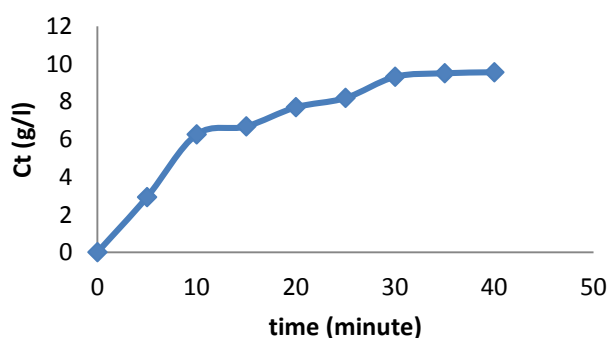


Figure 1. Concentration of oleoresin versus time

This result was quite different with the soxhlet extraction, where the oleoresin concentration was only 3.76 g L^{-1} in 1 hr extraction process. To make it equal with the result of 35 minutes MAE process, soxhlet extraction must be done around 4 hr, with the oleoresin concentration was 9.48 g L^{-1} . These results proved that MAE process was rapid and gave high yield in shorter time. In MAE process, heating efficiency was improved using microwave, where heat was generated through ionic conduction and dipole rotation of sample and polar solvent [12]. The temperature rise will increase the cell rupture and cause the rapid dissolution of the oleoresin from the cell [13]. Moreover, the short contact time of extraction will make the thermolabile components were safe and hence should resulting a better quality of oleoresin [11]

3.1. First Order Kinetic Model

Plot of $\log (C_s - C_t)$ versus t was presented in Figure 2. From the figure it can be determined the constant rate (k_1) and concentration of saturation (C_s), which were shown in Table 1. The figure showed that in the beginning of extraction, experimental result fit with first order kinetic model. But when the time was increased, the experimental result lacked its fitness to the model. The coefficient of determination for this model also low, below 0.90. It implied that the first kinetic model was not comprehensive for the process.

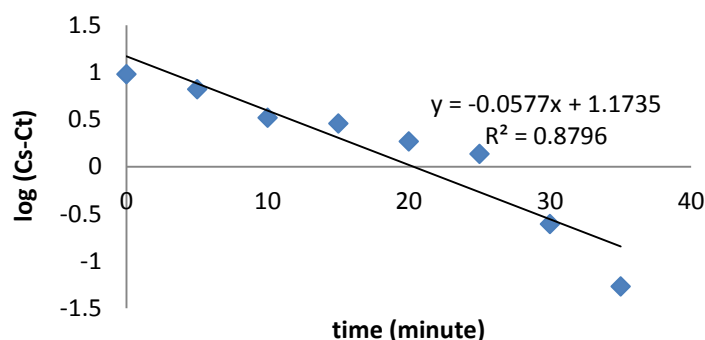


Figure 2. Plot of concentration in first order kinetic of cinnamon oleoresin

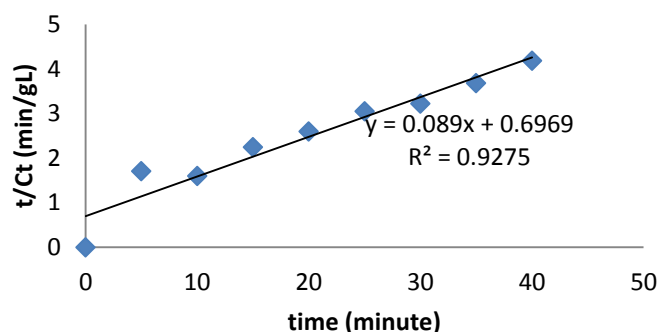


Figure 3. Plot of concentration in second order kinetic of cinnamon oleoresin

3.2. Second Order Kinetic Model

The same data were used to evaluate the second order kinetic model. The plot data were t/C_t versus t (Figure 3), while constant rate and concentration of saturation are presented in Table 1. In contrast to the first order, the experimental data were more prevalent with the second order model after 10 minutes. The deviations from the model were happened slightly. Only in the beginning of the extraction (5 minutes) the deviation was high. This condition probably caused by rapid extraction process due to high dissolution of solute from the particle surface [9]. The coefficient of determination in second order model was also higher than the first order. The R^2 was 0.927, more than 0.90, indicates that the model was fit with the data. From the result above we can conclude that second order model was more suitable for explaining the MAE of cinnamon oleoresin.

Table 1. Parameter of Kinetic Model of MAE of Cinnamon Oleoresin

Model	C_s (g L ⁻¹)	k (L g ⁻¹ min ⁻¹)	R^2
First order	14.894	0.131	0.879
Second order	11.236	0.011	0.927

3.3. SEM Analysis

Scanning Electron Microscope (SEM) was used to examine the cell structure after extraction. Figure 4 (a) showed the MAE cell structure, while soxhlet cell structure was shown in Figure 4 (b). In Figure 4(a), it showed that the cell had opened part, which this opened part dealt with rupturing of cell that made the cell content released. The soxhlet process also had the opened part, confirming that the cell rupture also happened in it. The opening part in MAE process was bigger than in soxhlet process, means that cell rupture in MAE process happened quicker than in soxhlet one. In MAE, rise of temperature will cause the higher heating rate, and it made the mass transfer of constituent enhanced [13]. Moreover, rapid cell rupture was predicted happened due to cell explosion because of thermal stress.

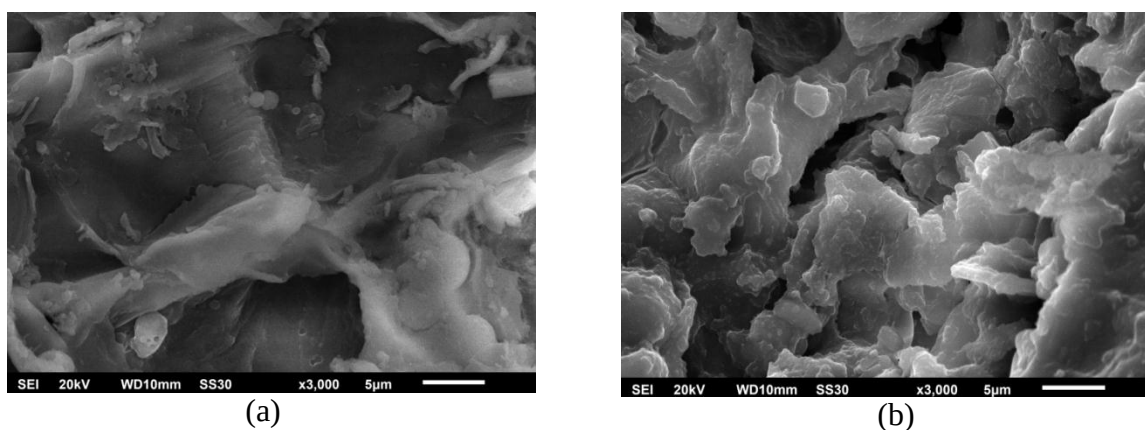


Figure 4. SEM image of (a). 35 minutes-MAE sample, (b). 1 hr-soxhlet sample

4. Conclusion

Microwave Assisted Extraction (MAE) was an alternative extraction method with a rapid process to get higher yield than soxhlet extraction. The kinetic model of MAE extraction was following second order model with the R^2 was 0.92. From SEM analysis it was observed that both MAE and soxhlet process had an opened cell part, confirming that the cell rupture had happened. But the opening part in 35 minutes MAE process was bigger

than in 1 hr soxhlet process, means that cell rupture in MAE process happened quicker than soxhlet.

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