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Mass Transfer Coefficient of Oleoresin Extraction from *Peperomia pellucida* L. Using Ultrasonic Cleaner

Siswanti*, Wildan Danny Azra, Tiara Susilawati Ardana

Department of Chemical Engineering, Universitas Pembangunan Nasional "Veteran" Yogyakarta

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Abstract. Indonesia has a variety of wild plants that are beneficial for health, one of which is *Peperomia pellucida* L. Several studies have explored the use of *Peperomia pellucida* for health and cosmetic purposes. This plant can produce oleoresin, a compound obtained by using solvents to extract plants or materials. In this research, extraction was carried out using the Ultrasonic Assisted Extraction (UAE) method. This research aims to investigate the effect of varying the mass of *Peperomia pellucida* and extraction time on the concentration of extracted oleoresin and its mass transfer coefficient. The experiments were conducted using mass and extraction time variations until equilibrium concentration was reached, with a constant solvent volume. The resulting extract was then analyzed to determine the oleoresin concentration in the solvent under various conditions and to calculate the mass transfer coefficient (K_C) by correlating oleoresin concentration with time. Based on the research that has been done with 200 ml of ethanol, variations in sample weight of 1, 2, 3, 4, and 5 grams and extraction time to constant concentration showed that the greater the sample mass and the longer the extraction time, the greater the concentration obtained. Additionally, K_C values increased proportionally with sample mass, with the highest K_C value of 4.4265 x 10^{-5} g ethanol/min.mm² observed at a sample mass of 4 grams.

Keywords: *Peperomia pellucida L., extraction, oleoresin, UAE, mass transfer coefficient*

1. Introduction

Indonesia has many wild plants, including *Peperomia pellucida* L., commonly found in humid areas and near water sources. *Peperomia pellucida* L. is also well-known for its properties as an herbal plant that is inexpensive and effective for treatment, care, and disease prevention because it is easily accessible [1]. Morphologically, *Peperomia pellucida* L. has a unique leaf shape, being heart-shaped and pointed, with short leaf stems [2]. *Peperomia pellucida* L. can act as an antimicrobial, while its flavonoid compounds can be antioxidants [3]. It can also be used as food, flavoring, and medicine. Additionally, there are claims that *Peperomia pellucida* L. can be used in cosmetics [4].

*corresponding author: siswanti@upnyk.ac.id

Oleoresin is the result of extraction, containing key components such as volatile substances (essential oils) and non-volatile substances (resins or gums) [5]. Oleoresin has several advantages, including being economical, stable when heated, free from contamination, and having a long shelf life [5,6]. Oleoresin from *Peperomia pellucida* L. contains active compounds such as dillapiole, caryophyllene oxide, and stigmasterol, contributing to its biological activities [7,8]. The alkaloid content in the extract of *Peperomia pellucida* L. contributes to its antioxidant activity, which may aid in wound healing by increasing antioxidant enzyme levels [9]. Oleoresin from *Peperomia pellucida* L. has been formulated into effervescent tablets as a natural disinfectant, demonstrating its potential applicability in the pharmaceutical and healthcare [10].

Research [11] states that *Peperomia pellucida* L. contains compounds of flavonoids and tannins, which have antibacterial properties. Tannins can stabilize lipid fractions so that they act as antioxidants. And based on research [12], it is stated that the ethanol extract of *Peperomia pellucida* L. can inhibit the development of acne-causing bacteria *Propionibacterium acnes*, with strong antibacterial properties, reaching an inhibition zone of 13.73 mm at a concentration of 15%.

The extraction process is separating a substance from its mixture using several solvents. The extraction methods commonly used in recent years are conventional extraction methods such as maceration, Soxhlet, and hydro distillation, which use large volumes of solvent and require long extraction times. To overcome the weaknesses of these methods, various new extraction techniques have been proposed for the extraction of bioactive compounds from food, one of which is the Ultrasound Assisted Extraction (UAE) method, which uses less solvent volume, shortens extraction time, and is energy efficient. In a literature review, the use of the ultrasound method is increasingly in demand compared to conventional methods, because it shortens extraction time. The ultrasound-assisted extraction method is suitable for heat-sensitive bioactive components with processes at lower temperatures, and the mechanical effect of ultrasound provides greater solvent penetration into cellular materials, thereby increasing mass transfer. At the same time, disruption of biological cell walls facilitates the release of their contents. Therefore, the ultrasonic-assisted extraction method is more effective than conventional methods because it has two main advantages: reducing extraction time and solvent volume usage. Some other benefits of the UAE method are that it can remove extracts from the matrix without damaging the extract structure, use at low temperatures to reduce heat loss, and

prevent the loss or evaporation of compounds with low boiling points. One of the extraction methods that uses ultrasonic waves is Ultrasonic Assisted Extraction [13]. The ultrasonic extraction process is influenced by several factors, including solvent composition, extraction time, sample weight, and water content [14].

For the effect of sample weight, the heavier the material, the greater the extract obtained. This occurs because the increased mass added during the extraction process results in more compounds being extracted optimally [15]. The length of the extraction time can lead to oxidation and changes in the chemical structure of the extract. Therefore, using a short extraction time may result in less optimal extraction of compounds from the material [16].

This research used ethanol as the solvent due to its polar nature, wide availability, selectivity, non-toxicity, good absorption properties, and high solubilizing ability, allowing it to extract non-polar, semi-polar, and polar compounds. A 96% ethanol solution penetrates cell walls more easily than lower concentration ethanol solvents, resulting in a more concentrated extract [17].

Based on this background, this research aims to determine the effect of sample mass and extraction time variations on the concentration of oleoresin extracted and its mass transfer coefficient. To design an efficient ultrasonic extractor or for scale-up purposes, data on the mass transfer coefficient are required, which are still challenging to obtain.

The extraction process can be considered as a mass transfer event that includes:

a) Diffusion of active compounds from inside the material to the surface.

b) Transfer of active compounds from the material's surface to the liquid.

c) Diffusion of compounds within the liquid.

The rate of solid-liquid extraction depends on two main stages: the diffusion of active compounds from the solid into the surface and the transfer of active compounds from the surface of the solid to the liquid. If the solid size is relatively large, the diffusion within the solid to the surface occurs slowly, and thus the mass transfer process is controlled by the diffusion rate. However, as the material size decreases, the surface area of the solid increases, and the mass transfer rate becomes greater. In other words, the distance for diffusion experienced by the solute becomes smaller and is considered negligible [18]. Hence, the extraction rate is controlled by the mass transfer of active compounds from the material's surface to the liquid.

The rate of mass transfer from the solid surface to the liquid can be expressed by the

following equation (1):

$$N_A = K_c \,.\, a.\, (C_A^* - C_A) \quad (1)$$

C_A = Concentration of oleoresin in the liquid phase (g oleoresin/g ethanol)

C_A^* = Equilibrium concentration of oleoresin (g oleoresin/g ethanol)

N_A = Mass transfer rate (g oleoresin/min.mm²)

K_C = Mass transfer coefficient (g ethanol/min.mm²)

a = Surface area of the *Peperomia pellucida* L. powder particles (mm²)

The C_A^* value indicates the concentration of oleoresin in equilibrium. The correlation between the equilibrium concentration in the liquid phase and the concentration of oleoresin in the solid phase on the grain surface is expressed by the Henry equation:

$$C_A^* = H \,.\, X_A \quad (2)$$

H = Henry's equilibrium constant (g sample/g ethanol)

X_A = Concentration of oleoresin in *Peperomia pellucida* L. (g oleoresin/g sample)

The mass balance equation for *Peperomia pellucida* L. extract (A) in the solvent in the extractor is expressed in the following equation:

$$R_{in}\, A - R_{out}\, A = R_{acc}\, A \quad (3)$$

$$K_c \,.\, a.\, (C_A^* - C_A) - 0 = \frac{d\,(W.\, C_A)}{dt} \quad (4)$$

Assuming that the particle size is uniform and spherical with a radius r, the number of particles n_b , and the sample mass M, equation (4) becomes:

$$K_c \,.\, n_b \,.\, 4\pi \,.\, r^2 \,.\, (C_A^* - C_A) = W \frac{dC_A}{dt} \quad (5)$$

$$M = n_b \,.\rho_s \,.\, \frac{4}{3} \,.\, 4\pi.\, r^3 \quad (6)$$

M = Mass of Peperomia pellucida (g)

n_b = Number of Peperomia pellucida particles

ρ_s = Density of Peperomia pellucida powder (g/mm³)

r = Radius of Peperomia pellucida powder particles (mm)

t = Extraction time (min)

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W = Weight of solvent (g)

Thus, the number of particles is:

$$n_b = \frac{M}{\rho s \cdot \frac{4}{3}.\pi\, r^3} \quad (7)$$

Substituting equations (5) and (7)

$$K_c \cdot \frac{M}{\rho s \cdot \frac{4}{3}.\pi\, r^3} \cdot 4\pi.\, R^2 \cdot (C_A^* - C_A) = W \frac{dC_A}{dt} \quad (8)$$

$$K_c \cdot \frac{3M}{\rho s \cdot r} (C_A^* - C_A) = W \frac{dC_A}{dt} \quad (9)$$

$$K_c \cdot \frac{3M}{\rho s \cdot r \cdot W} (C_A^* - C_A) = \frac{dC_A}{dt} \quad (10)$$

The mass transfer of oleoresin from *Peperomia pellucida* L. powder to the solvent is approximated using the mathematical model derived from equation (10) and expressed in equation (11):

$$\frac{DC_A}{dt} = \frac{3M \cdot Kc}{\rho s \cdot r \cdot W} (C_A^* - C_A) \quad (11)$$

The equilibrium concentration C_A^* can be determined using $C_A^* = C_A$

The concentration of the active compound in the solid phase (sample) in a batch system is obtained from the total mass balance equation of the active compound, expressed in equation (12):

$$M \cdot X_{A0} = M \cdot X_A + W \cdot C_A \quad (12)$$

X_{A0} = Initial concentration of oleoresin in Peperomia pellucida (g oleoresin/g sample)

2. Research Method

2.1 Materials

The primary material used is *Peperomia pellucida* L., which was harvested from Karangduren, Kebonarum, and South Klaten. Other materials used for extraction include 96% ethanol, which was purchased from Progo Mulyo, Jl. Selokan Mataram CT III No.1, Caturtunggal, Depok, Sleman, Yogyakarta.

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2.2 Equipment

The equipment used includes a Soxhlet apparatus, a distillation apparatus, a Biobase Ultrasonic Cleaner (frequency 40 kHz, power AC 220V 50Hz, capacity 10 L), and a UV-Vis Spectrophotometer (Spectral bandwidth: 1.8 nm, Light source: Xenon flash lamp, Detectors: Dual silicon photodiodes, Wavelength range: 190 – 1100 nm, Wavelength accuracy: 1.0 nm).

2.3 Methods

2.3.1 Preparation of *Peperomia pellucida* L.

The *Peperomia pellucida* L. used in this research is fresh. The plant is washed and dried by air drying at room temperature. Afterward, it is ground into smaller pieces using a blender and screened using a ≥30>100 mesh sieve.

2.3.2 Determination of Oleoresin Content in *Peperomia pellucida* L.

An amount of 50 g of *Peperomia pellucida* L. is wrapped in filter paper and then placed in a Soxhlet to be extracted with 500 mL of ethanol. The resulting extract is separated by distillation, evaporated, and dried in an oven until a constant weight is achieved.

2.3.3 Determination of Wavelength for Maximum Absorbance

The oleoresin obtained from Soxhlet extraction is dissolved in a solvent at a specific ratio, and its absorbance is measured using a spectrophotometer at a particular wavelength. The process is repeated with a wavelength range of 340 – 450 nm until the wavelength at maximum absorbance is obtained.

2.3.4 Standard Curve Preparation

A certain amount of oleoresin is diluted with ethanol in a 20 mL volumetric flask, and its absorbance is measured using a spectrophotometer. The experiment is repeated for varying concentrations of oleoresin to establish a correlation between absorbance and concentration.

2.3.5 Ultrasonic Extraction Process with Varying Sample Mass

Extraction is done by adding 200 mL of 96% ethanol into a 500 mL beaker and 1 g of *Peperomia pellucida* L. This is then placed in an ultrasonic device filled with water. Every 3 minutes, the absorbance is measured to monitor the concentration. The experiment is repeated for varying weights of *Peperomia pellucida* L. powder (2, 3, 4, and 5 g).

2.3.6 Experiment to Determine the Mass Transfer Coefficient (Kc)

To determine the value of the mass transfer coefficient, data obtained from experiment 2.3.e, which shows the correlation between concentration and extraction time, is used. This can be expressed using equation (11). The equation is solved using the Runge-Kutta-4 method with the Scilab application. Using the Golden Section optimization method, the mass transfer coefficient value is chosen based on the minimum SSE (sum of squared errors).

3. Result and Discussion

3.1 Determination of Henry's Equilibrium Constant (H)

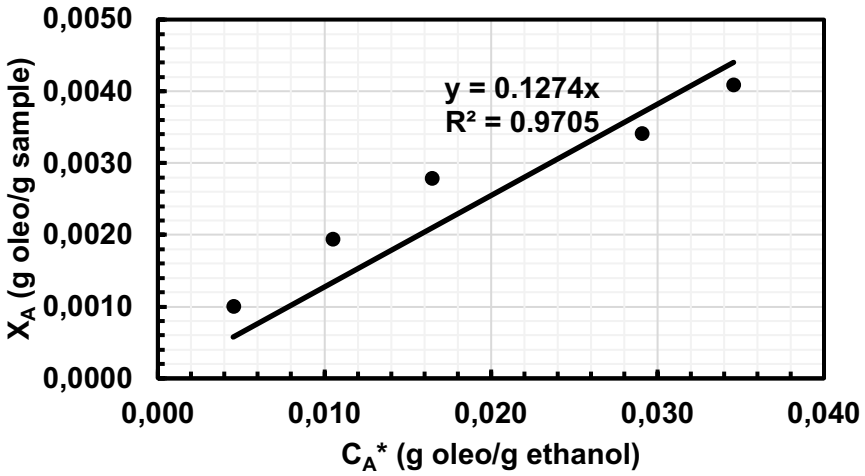

Figure 1. Correlation between C_A* and X_A at equilibrium

The correlation between C_A* and X_A at equilibrium is obtained from equation (2). The equilibrium constant is determined based on its slope. The data of the correlation between C_A* and X_A at equilibrium is shown in Figure 1, and the slope obtained is 0.1274.

3.2 Effect of Powder Mass on Oleoresin Concentration

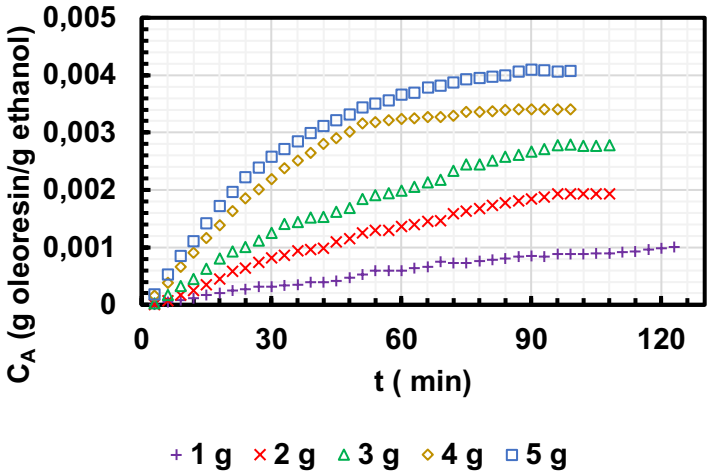

Figure 2. Correlation between Oleoresin Concentration and Extraction Time at Powder Weights of 1, 2, 3, 4, and 5 g

The data showing the relationship between varying masses of *Peperomia pellucida* L. powder and oleoresin concentration, observed at the wavelength of maximum absorption, which was found to be 414 nm, is presented in Figure 2.

In Figure 2, it can be observed that as the extraction time increases, the concentration of oleoresin extracted becomes larger. This happens because the contact time between the solvent and the powder increases, allowing the solvent to extract more oleoresin. Increased extraction time enables better solvent penetration, disrupting cellular structures and releasing oleoresin [19]. Over time, the concentration difference relative to time changes less because the solvent's ability to extract oleoresin diminishes as it approaches the saturation point [20].

The more mass that is used, the larger the resulting concentration. This occurs because of the principle behind ultrasonic waves, which create cavitation bubbles. When the bubbles burst near the cell walls of the material, shock waves are generated, causing the cell walls to break. The breaking of the cell walls allows the components inside to be released and mix with the solvent, so the more material there is, the more is extracted. Increasing the sample in the medium leads to more frequent collapse of cavitation bubbles near the solid surface, triggering the release of active compounds more effectively [21].

Furthermore, as more sample mass is used, the time to reach the saturation point is faster because, in the initial phase of extraction, there is a high concentration difference between the substance in the material and the solvent. The more samples used, the greater this concentration gradient, which causes the compounds to transfer to the solvent more quickly until equilibrium

is reached. Additionally, more solid particles are exposed to ultrasonic waves, increasing the contact points between the waves and the sample, and speeding up the mass transfer process [22]. As the amount of material increases without a proportional increase in solvent volume, the solvent reaches its solubility limit more quickly. Thus, the saturation point is reached sooner.

3.3 Simulation of Oleoresin Mass Transfer Coefficient Calculation (K_C)

The result of the K_C and SSE simulations in Scilab can be seen in the table below:

Table 1. K_C and SSE Values Obtained from Simulation Modeling

Powder Weight (g)	K_C x 10^{-5} (g ethanol/min.mm^2)	SSE x 10^{-7}
1	2.2889	1.948
2	2.7933	5.779
3	2.9125	8.857
4	4.4265	4.790
5	3.8710	1.233

The increase in powder weight used in extraction directly contributes to the rise in the mass transfer coefficient. This is due to the increased surface area of contact between the solid phase (powder) and the surrounding solvent, which is a key factor in the mass transfer process. More powder increases the total active surface area available for molecular interactions, thus raising the K_C value and accelerating the mass transfer rate. However, beyond a certain threshold, increasing the powder mass can lead to a decline in the mass transfer coefficient. This is primarily attributed to particle aggregation and compaction, which reduce the effective surface area and create diffusional resistance within the solid matrix. Excessive solid loading may also cause poor solvent penetration and limited fluid movement, especially in static or batch systems, reducing mass transfer efficiency [23,24].

4. Conclusions

Based on the research conducted, it can be concluded that the greater the powder weight used, the more oleoresin is extracted. Additionally, the longer the extraction time, the higher the oleoresin concentration obtained. The mass transfer coefficient (K_C) generally increased as

the powder weight used for extraction increased. The most significant mass transfer coefficient value was obtained in the experiment with a 4 g sample weight, yielding a value of 4.4265 x 10^{-5} g ethanol/min.mm^2.

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