COMPARISON OF MICROWAVE AND CONVENTIONAL EXTRACTION METHODS FOR NATURAL DYES IN WOOD WASTE OF MAHOGANY (SWIETENIA MAHAGONI)

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COMPARISON OF MICROWAVE AND CONVENTIONAL EXTRACTION METHODS FOR NATURAL DYES IN WOOD WASTE OF MAHOGANY (SWIETENIA MAHAGONI)

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Natural dyes from mahogany are usually obtained by conventional extraction. This extraction process requires a large solvent, a long duration of the process so that the energy requirements are also higher. Therefore, the use of "green techniques" to extract natural dyes with a minimum of energy and solvent should be considered. One extraction method that has been developed is the microwave-assisted extraction (MAE) method. In this work, the effects of microwave power, material to solvent ratio, and the heating time on the extraction results and the pigment components of the extract were examined. A comparison of the time required for reflux extraction and Soxhlet was also made. In microwave extraction, the highest yield was obtained at optimum extraction conditions such as microwave power of 600 W, the ratio of material to solvent of 0.02 g/mL, extraction time of 30 min. While for reflux and Soxhlet extraction, the extraction time needed to obtain optimum yield was 120 min and 720 min, respectively. Identification of compound components by the phytochemical test. Descriptions of the effects of microwave and conventional extraction are shown by damage to the surface structure of solid materials using Scanning Electron Microscopy (SEM).

Key words: natural dyes, microwave-assisted extraction, soxhlet extraction, heat reflux extraction, swietenia mahagoni

INTRODUCTION

Nowadays, textile production is increasing rapidly which causes an increase in the need for textile dyes as well. Synthetic dyes are widely used because of it easier to obtain, more practical and have a variety of colors. The use in small amounts is enough to give the desired color but causes environmental pollution. Synthetic dyes which are disposed of without being treated first can pollute the environment, especially the marine environment [1].

The exploration of the source of natural dyes become widespread with the increasing awareness of the negative impact of synthetic dyes on the environment and the wearer. Natural dyes derived from plants come from roots, bark, leaves, flowers, and seeds. It can be applied to dye fibrous materials and especially as a food coloring [2]. The advantages of natural dyes are non-toxic, non-carcinogenic, the source can be updated (renewable resources), there are no elimination problems (respectful with the environment), it reduces pollution and does not produce allergic reactions on the skin. Some natural sources produce really beautiful and economical dyes at price compared to chemical dyes [3–5]. In addition, natural dyes (pigments) derived from plants act as antimicrobials because they contain tannins that are antimicrobial [6]. In addition, normally natural plant dyes possess antioxidant and other health-promoting properties [7].

In this work, natural dyes derived from mahogany wood can be obtained by extraction. Mahogany wood is widely used for the construction materials and furniture industry. The processing of wood in building materials and furniture will produce wood waste in the form of powder. Generally, residues are not used optimally, while mahogany contains several types of pigments such as flav-noids, tannins, and quinones [2]. The dyes derived from mahogany produce a yellowish brown, this is due to the content of tannins and flavonoids in the mahogany extract [8].

Until now, natural dyes are still used in some textile industries obtained by boiling raw materials with water as a solvent (traditional method). For laboratory scale, it is usually done by conventional methods such as reflux and soxhlet. This method takes a long time and the amount of solvent is relatively large with low yield [5, 9–12]. This is what drives the technological innovation of the extraction process by using microwaves as energy sources with more optimal results in a relatively short time [13, 14]. This method also includes "green techniques" in the process of extracting natural dye.

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Basically, the work of the microwave method or common-

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ly called microwave-assisted extraction (MAE) occurs when the microwaves heated materials and solvents directly resulting in a localized heating that propelled the splintering of the walls of the plant cells and triggered the release of more dye in the solvent [3, 15]. Therefore, MAE is a potential alternative method especially when the material extracted is a plant [15–17].

From previous studies, it illustrates the need for the development of natural dyes extraction processes with various plant species that are the source of renewable, safe and nontoxic materials for the environment. Because the extraction process that utilizes microwaves as an alternative source of energy and is environmentally friendly technology, then in this research was explored the source of natural dyes by MAE method through the study on the parameters that affect the product produced. With this new technique used tools and materials that are environmentally friendly, effectively and efficientl .

This study aims to compare the MAE and conventional methods with the effect of extraction time on the yield of extraction. Identification of compound components through a phytochemical test. Changes in the solid structure after extraction can be determined by the use of Scanning Electron Microscopy (SEM).

MATERIAL AND METHODS

Material and chemicals

The material used in this research, mahogany (Swietenia mahagoni) wood waste was obtained from Wagir District, Malang in the form of sawmill powder. The powdered mahogany was passed through 35-60 mesh filte . The analytical grade of ethanol (98% purity) as a solvent for Soxhlet extraction and distilled water as a solvent for MAE and heat reflux extraction, were purchased from domestic distributors in Indonesia. Hydrochloric acid, ferric chloride, sodium chloride and magnesium powders (as chemicals for phytochemical test) also were obtained from domestic distributors in Indonesia. All chemicals were used directly without further purification

Microwave-assisted extraction (MAE)

The extraction of natural dyes from mahogany was carried out using an Electrolux EMM2007X microwave oven with 2.45 GHz magnetron frequency, and the power of 264, 400, and 600 W. Oven dimension with length 46.1 cm, width 28 cm and height 37.3 cm. The extraction process uses a three round neck flask as an extractor, equipped with a condenser that is placed at the top of the oven. The temperature in the extractor flask was measured by a type of thermocouple K 1/16 in (K 0.159 cm) installed in a microwave oven [1].

Raw materials was milled, sifted to a certain size (35-60 mesh). 2 g Mahogany wood powder and 200 mL aquadest solvents was fed into extractor. Water flowed on the cooling system (condenser). After the extraction process in the oven is complete (temperature around 100^{o}C for 10 minutes), the extract then filtered with filter paper (Whatman no.1; 9 cm diameter) using a vacuum filte , the dregs analyzed the surface of the structure with SEM. The obtained extract solution was concentrated and dried at a temperature of 60–80°C to a constant weight.

Heat reflux extraction (HRE)

About 12 g of powdered mahogany and 200 mL of distilled water as a solvent were placed in extractor flask. The temperature of the heater was set up to 100^{o}C. The solvent continues to circulate in the reflux (evaporates, cools and condensation and drips back into the extractor flask containing the material and the solvent extraction is performed with the specifi d time variable (30, 60, 90, 120, 150 and 180 min), and extracts filtered with filter paper (Whatman No. 1; 9 cm diameter), The dregs analyzed the surface of the structure with SEM The extract solution obtained was concentrated and dried at a temperature of 60–80°C to a constant weight.

Soxhlet extraction (SE)

A Soxhlet apparatus consisted of a condenser, extraction chamber, thimble, and a boiling flask was used to conduct extraction. Approximately 24 g of powdered mahogany wood was placed inside the thimble. To this, 400 ml of ethanol as a solvent was added slowly, and extracted until the solvent in the thimble is colorless. This condition can last for 720 minutes.

Determination of extraction yield of mahogany wood waste

The yield of samples produced from MAE, HRE and SE, extractions were calculated by:

$$y = \frac{S}{W} * 100$$

where *y* is extraction yield (%, w/w), *S* is mass of extracted yield (g), and *W* is mass of mahogany wood powder (g).

Qualitative analysis with the phytochemical test

Qualitative analysis was conducted by identification of components with phytochemical test of flavonoid, tannin and quinone compounds [18]. The phytochemical test was carried out on the extracted material with maceration for 2.880 min and MAE for 10 min.

RESULT AND DISCUSSION

Effect of microwave power

Figure 1 shows the effect of power on yield, where at 264, 400 and 600 W, power are obtained respectively 3.40%, 3.51%, and 3.78%. The results are shown in Figure 1 when the microwave power increased from 264 to 600 W with an extraction time of 10 min, the yield of a woodwaste of mahogany increased. The power param in breaking down plant cell tissue and will control the amount of energy that will be received by the material to be converted into heat energy. It is this heat energy that helps the diffusion process or the extraction of the dyes extract from the material into the solvent [19]. The greater the power, then the polar molecules in the material when exposed to microwave radiation will experience an increasingly rapid rotation (oscillation and collision motion) to produce heat energy detected by the increase in temperature, so that the greater the power, the greater the energy produced [20, 21].

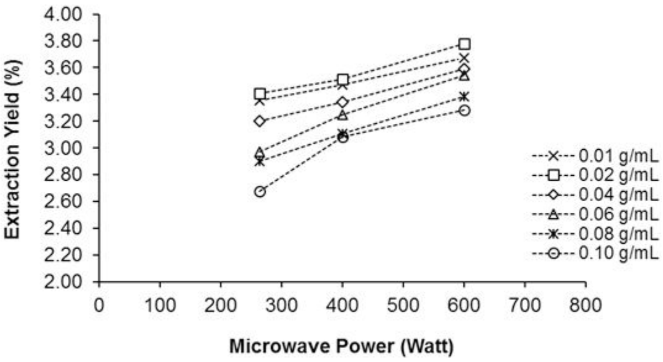


Figure 1: Effect of microwave power on the yield of mahogany wood dust extraction on a variety of material to solvent ratio for 10 min

Effect of feed to solvent ratio (F/S)

Figure 2 shows the effect of the ratio of the material to the solvent to produce the yield of the extract. In this study a water solvent was used based on the nature of polarity and has a high dielectric coefficient value that is approximately 80.4 [22], so it was very good at absorbing microwave. Figure 2 shows that the increase in the ratio of the material to the solvent causes a decrease in extracted yield. In general, the ratio of the material to the solvent with smaller molecules in the extraction system causes more solvent to be in contact with the material than the larger ratio [23]. When large volumes of solvent lead to swelling of the material, it is easier to break down the cell wall and release the dye in the solvent. The results shows that the ratio of the material to the optimum solvent was obtained at 0.02 g/mL, the power of 600 W with an optimum yield of 3.78%.

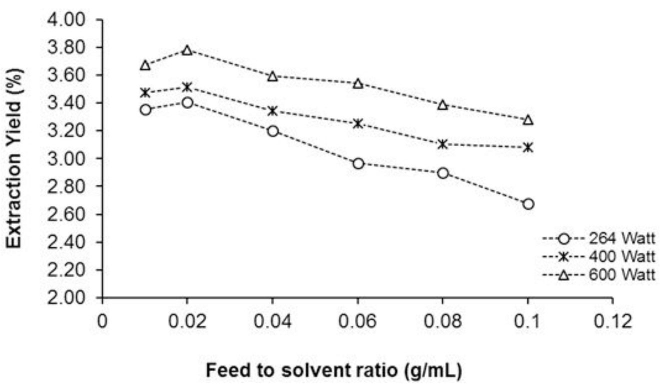
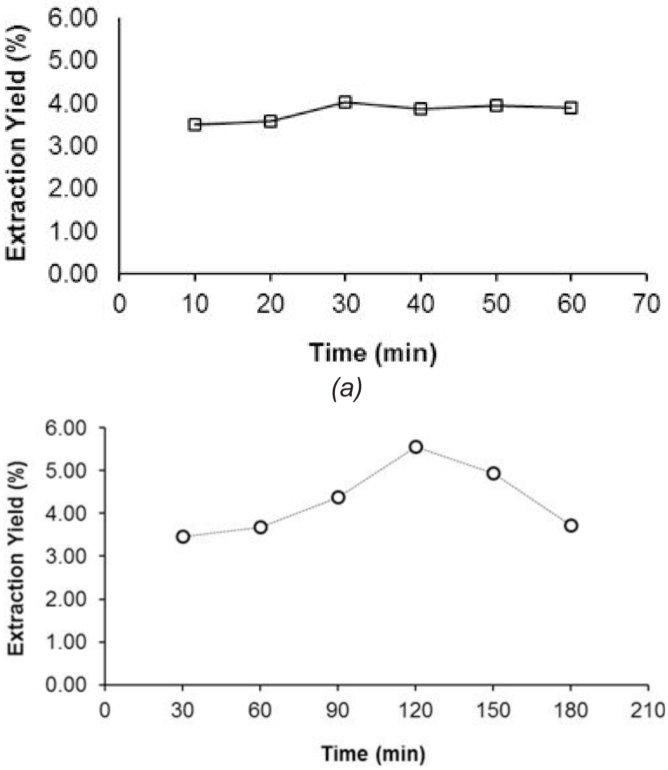


Figure 2: Effect ratio of mahogany wood powder with solvent to extracted yield(time of 10 min, material size of 35-60 mesh)

Effect of extraction time

The extraction time is one of the important factors that affect the acquisition of the extraction. The extraction time is closely related to the duration of contact of the solvent with the material. In general, the extraction time will increase its yield advantage.



(b)

Figure 3: The yield of extract produced with (a) Microwave-assisted extraction of power 400 W, Feed to Solvent ratio of 0.02 g/mL; (b) Heat reflux extraction (Feed to solvent ratio of 0.02 g/mL)

Figure 3 shows the effect of extraction time on yield with MAE and HRE. The extraction parameter with MAE is 400 W, the ratio of the material to the solvent is 0.02 g/mL, the temperature is uncontrolled. The time required to obtain optimum results with MAE is 30 min with the optimum yield of 4.01%.On the HRE and SE (feed to solvent ratio of 0.02 g/mL), the optimum yield obtained was 4.93% for 120 min and 4.85% for 720 min, respectively. In Figure 3a it is shown that for 10 min to 30 min it is in the diffusion phase, where the yield increase is insignificant. At 30 min onwards it appears to enter equilibrium conditions. Whereas for HRE (Figure 3b) it is seen that the yield increase lasts quite a long time for 30 to 120 min. After 120 min there was a decrease in yield due to the heating of the material has lasted so long that it degrades the thermolabile pigment component [19, 24]. The comparison of yield extraction with MAE, HRE, and SE is presented in Table 1.

Table 1: Comparison of yield with MAE, HRE and SE

Technique of extraction	Solvent	Time required (min)	Yield (%, w/w)
MAE	Water	30	4.01
HRE	Water	120	4.93
SE	Ethanol	720	4.85

Table 1 shows that the MAE method obtains extraction yield faster than conventional methods (HRE and SE). Heating with MAE is selective and volumetric. The heating is selective, meaning that microwave radiation can directly penetrate extractors that are transparent (continuing microwaves) so that the radiation can be directly absorbed by the materials and the solvent. The volumetric heating means that there is a direct heating in the overall volume of material, so that the heating can be uniform (evenly distributed) and last faster. In contrast to HRE method, the performance increases for a long time because the heat flows slowly from the mantle heater to the extraction mixture with a mechanism of conduction and convection. This is what causes faster extraction yield obtained when extraction is done with MAE compared to conventional methods [25].

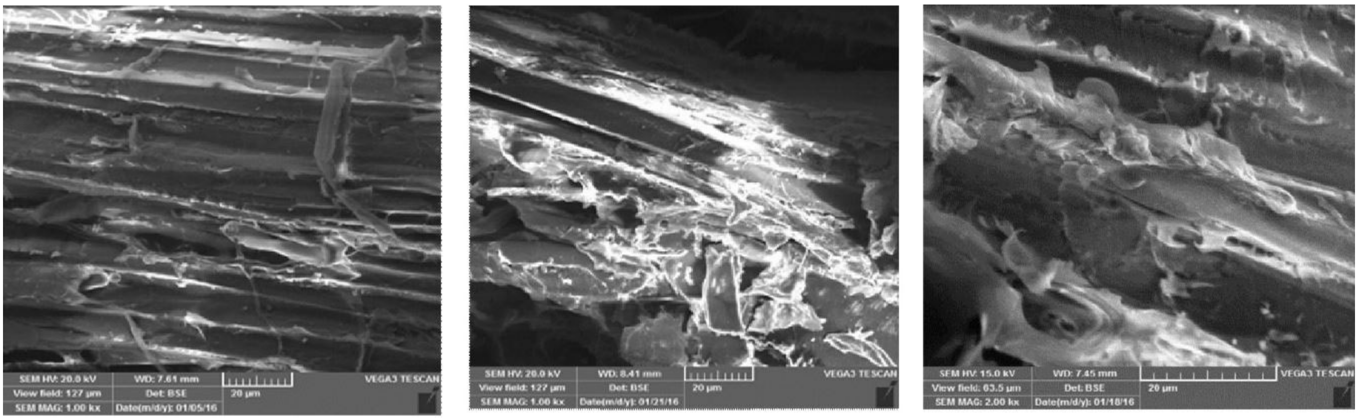
Structure surface morphology analysis by SEM

The description of the effect of the use of MAE and HRE on the morphology of the mahogany wood powder surface was carried out by SEM at 1000 magnifications.Fi - ure 4 it can be seen that there is surface damage to the particles of the material, which before it is extracted (Figure 4a) shows a picture of particle structure damage that is reduced in size by mechanical treatment. The particle structure is shown cut in the horizontal direction. The surface structure of the particle after extraction with MAE is shown in Figure 4b. The surface structure looks hollow (hollow) and the surface layer looks broken/cracked and peel off. While the picture after extracted by HRE method (Figure 4c), visible surface structure also peel off as if the surface is thinner and more flat

Phytochemical test

Phytochemical test performed on extracts produced by the method of cold maceration (extraction without the provision of heat) and the MAE (the temperature in the extractor around 100°C). The result of phytochemical test identified that flavonoids, tannins, and quinones (natural pigment) are found in woodwaste of mahogany as shown in Table 2.

The materials extracted with MAE and maceration methods differ slightly in terms of color density. In general, the color of the extract with MAE is more concentrated than extract with maceration. This also shows that quantitatively allowing microwave extraction (10 min) has a higher dye concentration than the maceration method (soaking for 2 days).



(a) (b) (c)

Figure 4: SEM image of mahogany powder size 20 μm with 1000x magnification on: (a) Powder before extraction; (b) Powder after extraction with MAE (Power of 264 W, time of 10 min); (c) Powder after extracted with HRE at time of 120 min, 2000 x magnification

Table 2: Pigment identification of extracted dyes

Type of material	Types of pigment	Reagents	Observation	Test results	
				Maceration	MAE
Mahogany wood	Flavonoid	Mg powder + HCl	Orange	+	++
	Tannin	NaCl + $FeCl_3$	Blackish green	+	++
	Quinone	NaOH	Red	+	+
Note: + = Strong;++ = Stronger					

CONCLUSION

In this study, extraction of natural dyes from a wood waste of mahogany powder was successfully extracted by using microwave-assisted extraction method. To obtain maximum results, the impact of several parameters was investigated such as microwave power, the ratio of material to the solvent and the duration of extraction. The results showed that the best condition required to achieve maximum yield (3.78 %) was the power of 600 W with material to solvent ratio (F/S) of 0.02 g/mL, time of 30 min. The extraction results obtained with MAE were compared with HRE and SE that the MAE gave the same extraction results (not very different), but the solvent consumption was lower and the extraction time was shorter than the HRE and SE. Therefore, it can be concluded that the extraction of natural dyes from mahogany waste with MAE is an efficient and efficient extraction method, especially in terms of energy consumption and solvents compared to conventional methods. In the mahogany wood, flavonoid, tannin and quinone (phytochemical tests) were detected. The SEM analysis showed that damage to the material structure formed a hole, cracked and peeled off after extraction with MAE for 10 min.

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