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COLOR IMPROVEMENT OF PRETREATED GMELINA WOOD BY IMPREGNATION OF NATURAL DYES

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ABSTRACT

The purpose of this research was to improve the appearance of pretreated gmelina wood (*Gmelina arborea*) by coloring with a natural dye. The dyes used in this research were obtained from sappan (*Caesalpinia sappan*) and tegeran (*Cudrania javanensis*) wood waste with a size of 20-40 mesh. The anatomical characteristic that determined the permeability of the gmelina wood was investigated referring to International Anatomist Wood Association (IAWA), while the characteristic of the dye was analyzed using X-ray diffraction (XRD). The wood was colored by immersing in dye mixture (ratio dye and water of 1:5 wt/wt) at room temperature during 72 h. The results showed that the lumen diameter of vessel, fiber, and pit of gmelina observed were 159 μm, 23 μm, and 6 μm, respectively. The XRD analysis showed that the structure of sappan was more amorphous than tegeran, which led to penetrate deeper into the wood. The pretreated wood provided more dyes penetration compared to the untreated wood due to the removal extractives from the wood. The pretreatment on gmelina wood would facilitate the natural dye to be impregnated into the wood cell resulting in more attractive color of the wood.

KEYWORDS: Anatomical characteristic, coloring, *Gmelina arborea*, sappan, tegeran.

INTRODUCTION

Utilization of fast growing species from community forests has became one of alternative solution to minimize the impact of the imbalance between the demand and the supply of raw

materials. One of fast growing species that used in this research was gmelina wood (*Gmelina arborea*). However, the wood provides low quality due to low in density and low in natural durability (Shmulsky and Jones 2011). Moreover, it provides a white, which was considered to be unattractive. Those drawbacks that discussed above caused limitation in utilization. For instance, it could be used for handicraft purposes. However, for handicraft purposes, the wood should provide an aesthetic appearance which is indicated by the attractive color, fiber direction, and image or pattern. Unfortunately, these criteria could not provide by the wood from community forest. The easier effort to improve the appearance of the wood is modify the color by application of dye.

Natural dyes have been widely used by Indonesian people since ancient times because they are non-toxic, renewable, easily degraded and environmentally friendly (Yernisa et al. 2013). Natural dyes are generally obtained from extracts of various parts of plants such as roots, wood, leaves, seeds or flowers (Kwartiningsih et al. 2009). Natural dyes have been widely used for coloring batik such as kesumba rivet (Pujilestari 2014), turmeric (Wijana et al. 2016), sappan (Pujilestari 2017), indigofera (Ariyanti and Asbur 2017), tegeran wood (Atika and Salma 2017), and tea plants (Alamsyah 2018). Avocado peel has been studied for its coloring properties as a textile dye (Kusumawati 2018). Extractive substances consisting of flavonoids, saponins, tannins and anthocyanins are a class of wood dyes. Flavonoids cause red, yellow, brown or blue wood, while tannins give a yellow color (Rosyida and Zulfiya 2013). Natural dyes have also begun to be developed as wood dyes with the aim of increasing the artistic value of woods with pale colors such as sengon wood, jabon wood and albasia wood. Previous studies mixed areca nut, betel leaf and gambier seeds for coloring Albasia wood (Bogoriani 2010), jabon wood was stained using tannin bark extract (Muflihati et al. 2014), kesumba rivet seeds were used as a dye on sengon wood (Putri et al. 2016).

Several factors influence the effectiveness of wood coloring, including type of dye, type of wood, thickness of wood, and the properties of the wood especially the anatomical structure such as the size and frequency of cells. In wood, the dye could be deposited in cavities or micropores in the wood cells and are thought to be able to make hydrogen bond with hydroxyl groups in the chemical components of wood cell walls (Sunarto 2008). Welly et al. (2016) reported that natural dyes have the disadvantage that not all dyes can color the wood fibers directly. In general, natural dyes that are adhesive require auxiliary substances, both acidic, alkaline, and salt to color the wood fiber.

This research aimed to improve the appearance of pretreated gmelina wood by coloring with a natural dye. The information regarding the permeability of wood is also needed to analyze the ease of the wood to be colored. Therefore, the identification of the anatomical characteristics of wood would be very useful as a basis for selecting wood and dye as raw material for handicrafts in the coloring process.

MATERIAL AND METHODS

Materials

Gmelina wood (*Gmelina arborea*) was used in this research. Natural dyes were obtained from sappan (*Caesalpinia sappan*) and tegeran (*Cudrania javanensis*) wood. The air dried gmelina wood sample used were size of 2 x 2 x 2 cm for anatomical investigation and 2 x 5 x 20 cm for coloring test, with five replication each. Furthermore, thin sectioning and maceration were performed on this sample to characterize the anatomical structure of gmelina wood. This characterization was based on IAWA (Wheeler 1989).

Wood preparation

The sample preparation using thin sectioning method was carried out by softening the wood sample by boiling until it was easily sliced. Then, the sample was soaked with a mixture of alcohol-glycerin successively with a ratio of 2:1, 1:1, and 1:2 each for a week. The sample was left in the last mixture until it became easy to slice. The axial and radial sections of the sample were sliced using a microtome with thickness of 18-20 μm. The thin section sample was washed in alcohol 30%, alcohol 10%, and distilled water for 2 min each. Next, the sample was colored using safranin for 24 h. After that, the sample was washed in distilled water until clean, and it washed again sequentially with 30%, 50%, 70% and 90% alcohol for 2 min. The last, the sample was arranged on a glass object and covered gently with a glass deck, glued with an adhesive on the glass object, and dried in an oven at 45°C. This thin section sample was used to observed and measured the diameter of the vessels, the number of vessels per mm square, the height and width of the rays, and the number of rays per mm square based on IAWA (Wheeler et al. 1989).

The sample preparation using maceration was carried out based on Sarkhad et al. (2022) method. The wood sample was cut into match-size sticks. These sticks were put into a test tube containing 35% hydrogen peroxide solution and 60% glacial acetic acid with a ratio of 1:1 (v/v). Then, it heated in a water bath at 60^{o}C for 2-3 hours. After that, it shaken until the fibers were separated. It was washed in distilled water until free from acid, then it colored with safranin. The colored fibers were left for 24 hours and then washed with graded alcohol (30%, 50%, 70%, 90%) for 2 min each. The fibers were observed on a glass object using Zeiss binocular microscope. The images of fiber were captured and measured using a software ImageJ. The macerated samples were used to measure the dimension of pits and fibers. Fiber length measurements were carried out at 10x magnification, while fiber and lumen diameter measurements were carried out at 40x magnification. The number of fibers measured for each macerated sample was refers to IAWA (Wheeler et al. 1989).

Dye preparation

The sappan and tegeran wood sample was cut into match-size sticks. These sticks were grinded using hammer mill to produce smaller particle. Then, it was sieved using 20 and 40 mesh sieves. The wood particles that used in this research were those that pass 20 mesh and were netted in 40 mesh (wood particle size of 0.42 to 0.84 mm). The dye solutions of sappan and tegeran were obtained by dissolving the particles in distilled water with the ratio of the weight of

the particles and water of 1:5. The samples were analyzed using Shimadzu, XRD-7000 to investigate the percentage of crystalline and amorphous fraction of the wood. The conditions of the equipment used were transmission symmetry configuration, horizontal goniometer position, using CuK$\propto$ radiation (λ=0.1541 nm) at 40 kV and 30 kV with a diffraction angle (2θ) measured between 10° to 40° at a speed of 2°/min.

Coloring test

Pre-treatment was given to the wood samples to remove the secondary chemical components from the wood, hence the coloring agents can penetrate easier into the wood. The pre-treatment was carried out by immersing the wood in distillate water at 70°C for 1 h. After that, the wood samples were drained and heated in an oven at 60°C until the moisture content was less than 16% (Ma'wa 2019).

Wood coloring was carried out on samples of wood that had been given pre-treatment, as well as on samples of wood without pre-treatment (untreated wood) as a comparison. The untreated and pretreated wood samples were immersed in the dye solution at room temperature for 72 h. The next, the wood samples were drained for 72 hours, and then dried in an oven at 60°C until the moisture content was less than 16% (Ma'wa 2019).

After the colored wood has been left at room temperature for a week, it was then cut 2 cm in width in longitudinal direction. The area of colored wood was observed and captured using a stereo microscope with a digital video microscope. The area and linear length of colored wood on the captured image of the wood was measured using a software ImageJ. The illustration of measurement was depicted in Fig. 1. The results of the depth calculation on the test samples are then averaged. The depth of the dye is calculated by following formula:

$$P = \frac{a}{A} \tag{1}$$

where: P refers to depth of dye penetration, a refers to the colored area in μm^2, and A refers to the linear length of measured colored area in μm.

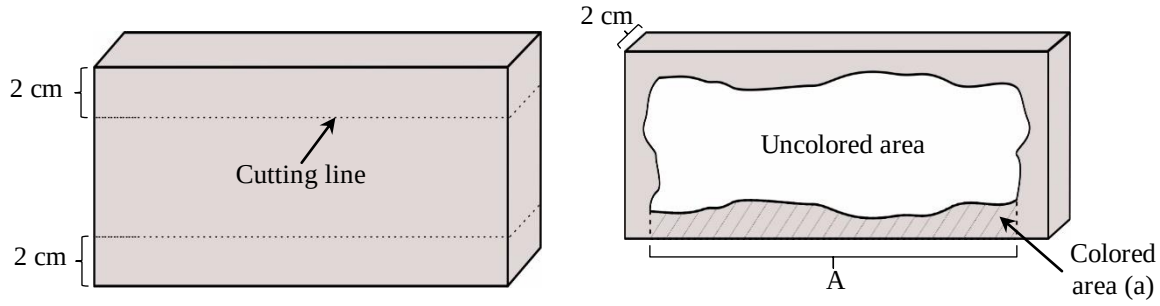

Fig. 1: Illustration of colored gmelina wood sample (left) and measurement of the dye penetration (right).

RESULTS AND DISCUSSION

Anatomical characteristics of gmelina

The ease of gases and liquids movement within the wood is known as permeability properties. A wood with good permeability is indicated by high porosity due to the large pores (Plotze and Niemz 2011). It means that wood with large cell cavities and high cell frequency would produces good permeability. Damayanti et al. (2020) reported that there are variations in the permeability properties of wood, depending on the wood constituent components, the shape and content of the cell walls, facilitating the penetration of fluids through diffusion.

As shown in Tab. 1, although gmelina provided a large vessel lumen, however it was short. Moreover, dimension of fiber, ray, and pit were found to be classified as small to medium. It was also found that the frequency of vessel and ray cell per mm were low. Therefore, it could be concluded that gmelina wood provided relatively medium-sized cells with low frequencies. It would affect the drying, preservation, pulping, and coloring process of the wood. It reported by Wardyani et al. (2017) that with large open vessels, it tends to be easier for dyes or preservatives to penetrate into the wood and drying process.

Tab. 1: Anatomical characteristic of gmelina.

Anatomical parameter	Mean Value	Classification*
Vessel lumen diameter (μm)	159	Large
Vessel per mm^2	5	Few
Vessel length (μm)	266	Short
Fiber wall thickness (μm)	4	Thin to thick
Fiber length (μm)	1019	Medium
Fiber diameter (μm)	31	-
Fiber lumen diameter (μm)	23	-
Rays per mm	3	Wide
Ray width (series)	4	Medium
Ray width (μm)	132	-
Ray height (μm)	556	-
Pit diameter (μm)	6.3	Small

Note: Classification according to IAWA (Wheeler et al. 1989).

Characteristics of the dye

The color produced by natural wood dyes is obtained from extractives stored in the wood cells. The extractives are not only stored in the cell lumen, however some of them can also be stored in the cell wall. Therefore, the structure of the cell wall will affect the amount of extractives that can be extracted from the wood. The results in Fig. 2 shows that sappan provided higher amorphous (84.79%) and lower crystalline (15.21%) fraction compared to the tegeran. This is considered to affect the amount of extractives that can be extracted from the wood. The higher amorphous fraction causes the extractive to exit the cell wall more easily, which in turn will increase the amount of the dye in the solution.

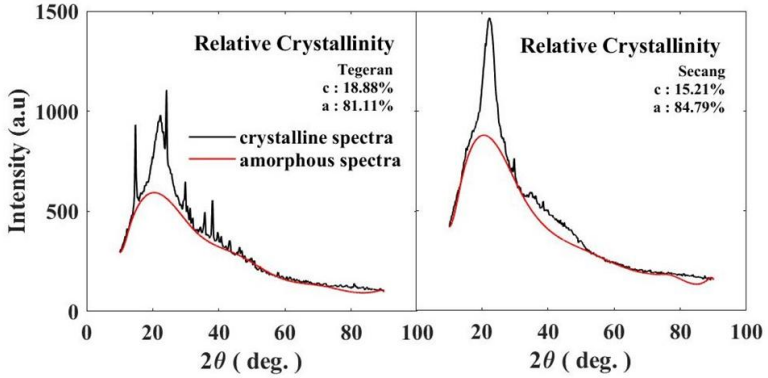

Fig. 2: Crystallinity of sappan (right) and tegeran (left), under *XRD analysis*

Coloring on gmelina wood

The results of coloring test on gmelina (Fig. 3) shows that penetration of sappan into the gmelina was twice higher than the penetration of tegeran. This could be affected by the higher amorphous fraction of sappan than tegeran which led to produce a higher amount of the dye in the solution as discussed above. The higher amount of the dye in the solution is considered to cause the higher amount of dye penetrated into the wood. In addition, the Segal equation is used to determine the level of amorphousness and crystallinity of the material to make a determination (Nam et al. 2016). The dependence of Segal amorphous intensity on crystal size, cellulose polymorph, and the degree of polymorphic conversion led us to simulate the diffraction patterns of wood in order to more thoroughly evaluate the relationship between these factors.

The result also shows that the penetration of both of sappan and tegeran into pretreated wood was higher than untreated wood. This phenomenon indicated that wood pre-treatment by immersion in high temperature would remove the wood extractive from the wood and replace them with the dyes. As reported by Muflihati et al. (2014) in previous research that difference in retention of the dyes in the wood is depend on the dimensions and arrangement of the wood cells. This finding also confirmed the study reported by Welly et al. (2016) that apart from the anatomy of the wood, the method of coloring also affects the absorption of materials that entering the wood. In addition, it was reported that permeability increased due to structural modification and chemical changes in cell wall components at temperatures above 75^{o}C (Taghiyari and Malek 2014).

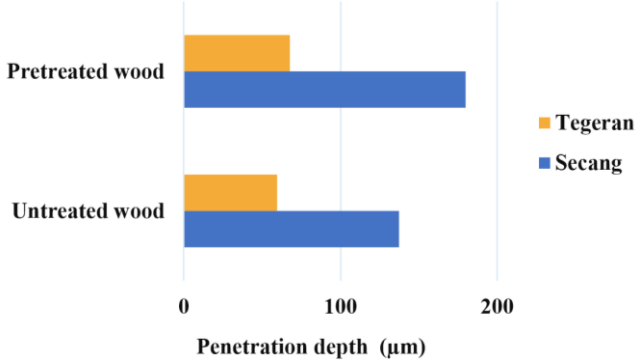

Fig. 3: Penetration depth of sappan and tegeran into pretreated and untreated gmelina wood.

Fig. 4 shows the appearance of the gmelina wood before and after coloring. As depicted in Fig. 4, uncolored gmelina wood was off-white to yellowish. Coloring with sappan generated reddish gmelina wood, whereas tegeran generated yellowish gmelina wood. The result of visual observation found that there was no notable difference in visual brightness between untreated and pretreated gmelina both in coloring with sappan and tegeran. It also seems that the color was not evenly distributed throughout both of treated and untreated wood surfaces. It might be caused by the wood extractives that were not removed evenly from around the wood surfaces, leading to difficult for the dyes to bond with the wood.

Fig. 4: The appearance of uncolored and colored gmelina wood using sappan and tegeran. Sample size of 20 cm in length, 5 cm in width, and 2 cm in thick.

The anatomical structure of sappan dyed gmelina wood is shown in Fig. 5. The result shows that the lumen of vessels was filled with a clear deposit both on the dyed-untreated and -pretreated gmelina wood. On the other hand, the ray cell was observed to be fully dyed. This was confirmed by the appearance of elliptical spots on the tangential section and wide elliptical to rectangular spots on the radial section which indicated the coloring ray. Different from the rays, the vessels generated clear shine in the lumen both on the surface of radial, tangential, and axial sections. As a result, the dye was difficult to penetrate either by diffusion or through the pit channels into the vessel cell walls as well as shown in the Fig. 5 that the vessel cell walls and lumens seemed to be undyed. Almost the same phenomenon was reported by Roque et al. (2020) that impregnation on gmelina wood absorbs only a small amount of solution. However, it was more commonly found in untreated gmelina wood (Fig. 5 up) than in pretreated gmelina wood (Fig. 5 down). This phenomenon was also considered as one of the reasons for more dyes penetrating into the pretreated wood compared to untreated wood (Fig. 3).

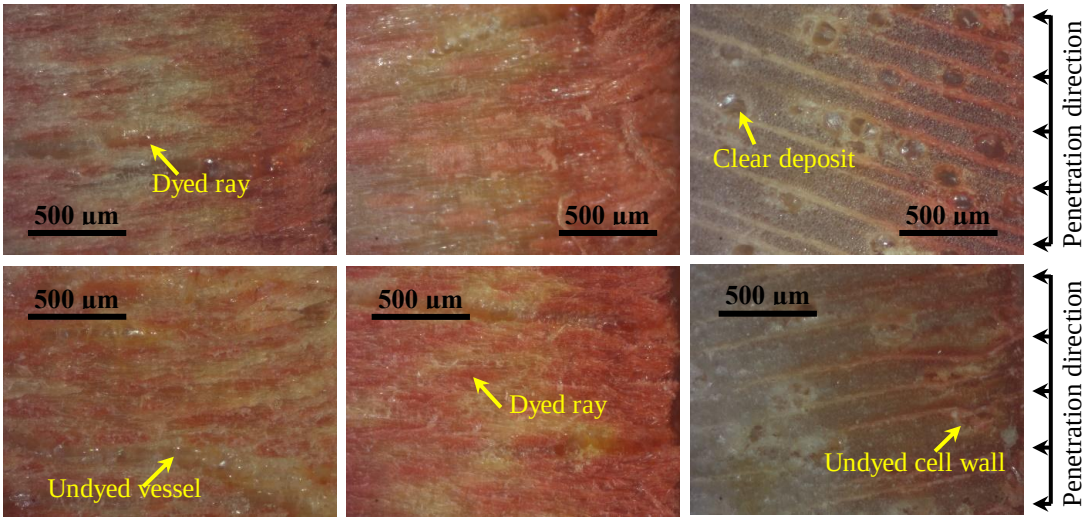

Fig. 5: The appearance of the sappan dyed-untreated (up) and -pretreated (down) gmelina wood in axial (right), radial (middle), and tangential (left) sections.

CONCLUSIONS

It was concluded from this study that gmelina wood provides relatively medium-sized cells with low frequencies. Gmelina wood provides size of vessel lumen, fiber lumen, and pith diameter of 159, 23, and 6.3 µm, respectively. Meanwhile, the frequency of vessel and rays are 5 and 3 per mm^2, respectively. The crystallinity of sappan and tegeran wood will indirectly contribute the penetration ability of these dyes into the wood. The coloring test on gmelina wood shows that although the dye is difficult to penetrate into the gmelina wood through the vessels, it is able to penetrate well into the wood through the rays. Pretreated wood provides more dyes penetration compared to the untreated wood due to the removal of wood extractive by immersion at high temperature. The depth of penetration of sappan in gmelina wood is more than double that of tegeran penetration. Better penetration of sappan on gmelina wood would be promising to be developed as an attractive natural dye to improve the appearance of wood for aesthetic purposes.

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