Waste extracts from forest plants and their application as natural coloring for fabrics

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Abstract. Wood wastes and tree barks are rich in natural coloring-pigments, such as tannin, flavonoids, and anthocyanins. If extracted, those compounds can be beneficial as coloring agents for fabrics. The research aimed to obtain yields of dry extracts from bark wastes of secang (Caesalpinia sp.), bakau merah (Rhizophora apiculata), ketapang (Terminalia sp.), and mangium (Acacia mangium); as well as from wood wastes of secang and ketapang. Wet extracts were obtained through waste extraction using pure water solvent with waste/water ratio 1:4 and 1:6 (w/v), each implementing of two temperatures (60°C and 70°C) and three long immersion treatments before extracting (0/control, 12, and 24 hours). The obtained wet extracts were applied for coloring the fabrics, followed with 3 fixation treatment, i.e. tawas, kapur tohor, and tunjung; and then tested for color-fastness resistance of fabrics against, i.e. washing, ironing, and sun ray referred to the SNI-ISO standards. The results indicated that bakau minyak barks extract brought out the fabric colors to the best result, and the yield dry extract was the highest. The natural coloring extracts of bakau minyak barks produced the best coloring performance of the fabrics against consecutive washing and sun-ray exposure, that is moderate until good (score 3-4), and the ironing results in good until very good (4-5). The yield dry extract of the barks was 28% after implementing 24 hours immersion in water before extraction, weight ratio between the extracted barks and water at 1:4 (w/v), and extraction temperature at 70°C.

1. Introduction

Production of round timber (woods) from the Permit Endeavors in Utilization of Wood Forest Products at Natural Forests (PEUWFP-NF) averaged about 5,865,985.80 m³/year [1]. The amount of wastes generated from activities of producing branch-free tree trunks reached 928,934.81 m³/year [2]. This figure still did not include round timber production from the Permit Endeavors in Utilization of Wood Forest Products at Plantation Forests (PEUWFP-PF), whereby PF’s timber production tended to increase each year. In 2018, production of round timber from plantation forests had already reached 40,945,378.90 m³ [1]. Research results by [3] at Indonesia’s paper mill company (PT Tanjung Enim Lestari Pulp & Paper) that used raw materials of round woods from mangium plantation forests disclosed that the amount of wastes especially in the forms of tree barks, which were generated throughout the pulp manufacture, could reach 216.49 ton/day or about 9% of the weight of round wood. Further, the wastes in the forms of slabs, wood sawdust, twigs, barks, etc. were not yet utilized optimally by wood-processing industries, as they focused only on utilization of wood portions. This
could create serious problems, because the presence of those generated and abandoned wastes became sooner or later accumulated enormously, thereby causing environment concerns in the forests as well as wood factories.

From several various research results [4,5,6], it revealed that wastes from particular tree portions (e.g. woods and barks) could serve as immense resources that were potentially rich in coloring compounds or pigments. Those pigments, when extracted, could be beneficially used as natural coloring-agents/chemicals for fabrics or weaving threads. Coloring chemicals or pigments encountered a lot in plant woods or barks are among others tannins. Tannins typify as inherently polyphenol compounds, which taste bitter in the mouth, soluble readily in water, and could coagulate proteins or other organic compounds such as amino acids and alkaloids [4]. The pigments from plant tannins could in color appear differently, depending of plant species and particular portions of the plants (e.g. woods, barks, twigs, leaves, etc.). For example, tannins from barks of nangka (Artocarpus sp.) trees exhibit red color, while those from nyirih (Xylocarpus granatum) wood trees emerge as brownish red color [7]. Natural coloring pigments could be obtained from their source origins through heating, storage, solvent treatment, or combination of any of those processes partially as well as entirely. The solvents which are used could be inorganic (water) or organic solvents. Organic solvents commonly employed are among others methanol and ethanol [8].

Plant pigments could be produced in liquid extracts or in dry extract forms. The advantage of liquid extracts is that they can be used directly for coloring the fabrics; but their disadvantage is that as organic substances the liquid extracts cannot be stored for a long time, because they are susceptible to deterioration and other degradation processes, getting slimy, or contamination by organisms. Accordingly, for long-term uses or for trades, liquid extracts should be processed into dry extract forms. The needs of natural color solutions for coloring the fabrics, depends on surface area of the fabrics and strength intensity of the desired colors at fabrics.

In coloring of fabrics, it involves two consecutive essential stages, which comprise mordanting and fixation. Mordanting intends to expel fat, oil, dirt that stick to the fabric fibers, in order facilitate the penetration of the fabric pores. Meanwhile, fixation aims to strengthen the bonds of colors to the fabrics that has been immersed in the dye solution [9]. In addition, fixation can keep the qualities of color-imparted fabrics from the wear off due to sweat, washing, ironing, and exposure to sun ray. To look into the performance of natural coloring which have been used for coloring the fabrics and then followed with fixative treatment should be tested for color-fading resistance against the washing, ironing, and sun ray exposure. The testing methods should refer to the recognized standards ISO (International Standard Organization) and SNI (Indonesia’s National Standard).

This paper presents the results of the application of dye extracts from several types of forest plant waste on cloth and its color fastness tests on washing, rubbing, and exposure to the sun's heat, as well as the yield of dry extract with several treatments.

2. Materials and Methods

2.1. Materials

The materials which were used in these research comprised wood waste and bark from secang (Caesalpinia sp.) and ketapang (Terminalia sp.) trees, which were obtained from Cilacap Regency (Central Java); bark wastes from bakau merah (Rhizophora apiculata) trees, obtained from Kubu Raya Regency (West Kalimantan); and bark wastes from mangium (Acacia mangium) trees, obtained from Sukabumi Regency (West Java). The materials serving as fixation agents were tawas (Al₂(SO₄)₃), kapur tohor (CaCO₃), and tunjung (FeSO₄). Meanwhile, the material as mordanting agent was tipol chemical. The fabrics that would be imparted with colors were mori or primissima fabrics. The tools which were incorporated consisted of gas stoves, pan for boiling forest plants’ wastes, pails, jerry cans, graduated cylinders, stirrers, weighing scales, cloths for filtering, and oven.
2.2. Methods

2.2.1. Preparing of the wet extracts

Preparing of the wet extracts (in aqueous solution forms), application of wet extracts as natural-coloring at mori (white-colored) fabrics in the Forest Products Research and Development Center (FPRDC), Bogor; and testing on qualities of coloring performance of the extracts at the color-imparted fabrics in the Institute for Quality Testing on Goods, Ministry of Trades, Jakarta, as follows:

2.2.1.1. Extraction of plant wastes

Plants’ wastes were cleaned. Afterwards, the cleaned barks and wood slabs were finely chopped off such that their dimension was reduced to about 1 cm size. The chopped barks and wood wastes were then boiled (extracted) in water for 2 hours, employing two levels of heating temperatures (60°C and 70°C); and concurrently implementing two ratios between the waste materials and water solvent (1:4 and 1:6, w/v). Before boiling, the chopped barks and wood wastes were immersed in water (regarded as pre-extraction), employing three immersion durations, comprising 0 hour (without immersion/control), 12 hours, and 24 hours. After that, the dye solution was filtered. The obtained liquid (wet extracts) was allowed for some time to cool down.

2.2.1.2. Mordanting

Mordanting was performed by immersing the mori fabrics (before fabric coloring with the extracts) in water, to which was already added 1% tipol (w/w), for 2 hours. Afterwards, the fabrics were cleansed by water several times without squeezing until the foams disappeared. Furthermore, the cloth is air-dried until dry.

2.2.1.3. Coloring of fabrics

Coloring was performed by immersing the fabric samples in the wet extracts (natural color) as many as 6-7 times intermittently, while turning over the samples repeatedly in the solution such that the immersed-fabrics became evenly colored. Each time of fabric immersion necessitated about 5-minute duration).

2.2.1.4. Color fixation

Fixation or locking intended to obtain the color-imparted fabrics with the colors suitable as desired by the users; and in order that the fabric colors were not easily faded away. The fixation process could be performed by immersing the color-imparted fabrics for 15 minutes in water, which was already added 2-3% fixation agents (w/v). In this research, three kinds of fixation agents were used separately, which comprised tawas, kapur tohor, and tunjung. Afterwards, the fabrics were cleansed with water, and then aerated.

2.2.1.5. Testing on qualities of natural-coloring

The testing methods for color-fade resistance of the fabrics consecutively against washing, referred to SNI ISO 105-C06: 2010; against ironing, referred to SNI ISO 105 x 12: 2012; and against sun-ray exposure, referred to SNI ISO 105-BO2: 2014. The stronger the color-fade resistance the greater would be the coloring qualities of the extracts at the fabrics. Qualities of the tested materials (only bark extracts) were categorized into five classes, i.e. class 1 (very poor), class 2 (poor), class 3 (moderate), class 4 (good), and class 5 (very good/best coloring performance).

2.2.2. Dry coloring extracts

In this method, 20 ml of wet extract solution derived from various forest plant wastes was taken, then put into porcelain cups, and further heated in the oven (100°C) until it produces a dry extract. The yields of dry coloring extract were determined using the formula:

\[ \text{Y} = \left( \frac{\text{W}_i - \text{W}_f}{\text{W}_i} \right) \times 100\% \]  \hspace{1cm} (1)

where, \( \text{Y} \): Yield of the dry coloring extract (%)
\( \text{W}_i \): Weight of coloring extract solution + weight of porcelain cup (g)
\( \text{W}_f \): Weight of dry coloring extract + weight of porcelain cup (g)
2.3 Data analysis
The obtained yield data of dry coloring extracts was analyzed and assessed using a completely randomized design (CRD) with factorial pattern. The factors as incorporated were 6 kinds of forest plants’ wastes (L), 3 immersing durations (S), 2 heating temperatures for extraction (H), and 2 weight ratios between the waste materials and water solvent (P). Each of the treatment combinations (LSHP) was replicated five times (R). When the effects of individual factors (L, S, H, P) or interaction (L x S x H x P) were significant on the yields, then the assessment continued with the Tukey tests. The data processing that implemented factorial CRD and Tukey tests was assisted by the SAS program of version 9.0.

3. Results and Discussion
3.1. Application of natural coloring extracts at fabrics and quality testing
Results regarding the application of natural coloring extracts, from six kinds of forest plants’ wastes, at mori fabrics using pure water as solvent, before and after fixative treatments, were presented in Table 1.

Mori fabrics which were immersed in the aqueous solution of natural coloring extracts exhibited colors that differed from each other. Apparently this occurrence depended on the waste’s species of forest plants as well as the waste’s plant portion (woods or barks in the same species). In this research, extraction of secang woods using water solvent (pH≈7.0) brought out the wood extracts at mori fabrics with yellowish brown color. Meanwhile, extraction of secang barks using also water solvent resulted extracts with reddish brown color. The specific compound present in secang barks that imparted extracts with reddish color was the so-called brazilin or C_{16}H_{14}O_{5}[10,11,12]. Conversely, the color of brazilin compound would change and become violet red under the base condition (pH>7.0). Likewise, brazilin compound in secang barks if extracted with organic solvent (e.g. methanol, ethanol), the color of obtained extracts would even become darker, i.e. strong brown. This occurred, because organic solvent could extract out almost all the compounds in secang barks, such as protein, carbohydrate, fat, besides extracting coloring pigments (i.e. brazilin and tannin). Meanwhile, the extracting ability of water solvent was limited only to the coloring-pigments and starches [13].

Further, extracts from ketapang woods exhibited grey color at mori fabrics, whereas correspondingly the color of extracts from ketapang barks was brown (Table 1). This visual appearance related to high content of tannin in tree barks, as inside the barks were present parenchyma tissues, which signified one that makes up tree bark tissues. The parenchyma tissues function to store reserve food and other compounds, including the tannins [14]. The thicker the tree barks, then the greater would be the tannin content inside. Barks of bakau and mangium trees contained a great amount of the condensed tannins or concentrated tannins, which was inherently the so-called flavonoid polymers [15,16]. Bark extracts from bakau minyak and mangium species brought out the tannin with brown color in the fabrics (Table 1).

At the color-imparted mori fabrics, except the extracts color from ketapang woods, the original color of the other extracts changes more immensely after the fixation or locking process. These color changes depended on kinds of fixation agents (Table 1). From those three kinds of fixation agents which were used, fixation using tunjung/ferro sulphate (FeSO_{4}) brought out the fabrics with aptly darker/black colors, compared to the fabric colors using two other fixation agents (kapur tohor/CaCO_{3} and tawas/Al_{2}(SO_{4})_{3}). Fixation using tawas brought out the fabric colors which were almost similar to or slightly weaker than the original colors of extracts at the fabrics (without fixatives); whereas, fixation using kapur tohor resulted in the fabric colors stronger than such extract’s original colors at the fabrics. Meanwhile, fixation using tunjung exhibited fabric colors which contrasted with such also original extract colors at the fabrics (Table 1). Fixation using tunjung resulted in fabric colors which were darker (stronger black) compared with the original colors before fixation, because tannins in the color extracts reacted with Fe^{2+} ions in tunjung, forming complex salts and thereby causing black colors of the fabrics [17]. From all the extract materials, the extracts from bakau minyak barks brought
out the fabric colors which contrasted. The colors of mori fabrics after being immersed in bakau minyak extract solution became brown, but after kapur tohor fixation the fabric colors changed to marun red; whereas the treated with tunjung fixative became blackish brown (Table 1).

Table 1. Application of extracts from 6 kinds of forest plant wastes used as natural coloring pigments for mori fabrics, then treated with 3 kinds of fixation agents

| Kinds of wastes (as extract origins) | Colors of extract-immersed mori fabrics |
|-------------------------------------|----------------------------------------|
|                                     | Without fixation agents (control)      |
|                                     | Kapur tohor (CaCO₃)                    |
|                                     | Tawas (Al₂[SO₄]₃)                      |
|                                     | Tunjung (FeSO₄)                        |
| Secang barks                        |                                        |
| Secang woods                        |                                        |
| Ketapang barks                      |                                        |
| Ketapang woods                      |                                        |
| Bakau minyak barks                  |                                        |
| Mangium barks                       |                                        |

Qualities of bark extracts from wastes of forest plant, as natural-coloring pigments applied at mori fabrics (followed with kapur tohor fixative) were indicated by the color-fade resistance of washing, ironing, and sun-ray exposure. Relevantly, from the four kinds of bark extracts (Table 2), natural coloring extracts from bakau minyak barks afforded the highest qualities (best coloring performance of the fabrics) against consecutively the washing (moderate until good), the ironing (good until very good), and sun-ray exposure (moderate until good). Meanwhile at fabric dyed with natural dyes extracts from ketapang and secang barks, the qualities (except against the washing) were equally comparable to the coloring qualities of extracts from bakau minyak barks. Compared to the other three kinds of bark extracts (secang, ketapang, and bakau minyak) the qualities (coloring performance) of extracts from mangium barks at the fabrics were the lowest. The low color-fade resistance of mangium bark extracts at the fabrics in this research was presumably attributed to the mangium barks which were used in fact not the fresh ones. As such, the mangium barks had been stored for more than 3 months, before being extracted.

Table 2. The color-fade resistance of the bark extracts* from four forest plant species

| No. | Color-leaching resistance against | Secang | Ketapang | Bakau minyak | Mangium |
|-----|----------------------------------|--------|----------|--------------|---------|
| 1.  | Washing                          | 2      | 2 - 3    | 3 - 4        | 2       |
| 2.  | Rubbing/ironing                  | 4 - 5  | 4 - 5    | 4 - 5        | 3 - 4   |
| 3.  | Xenon ray (could representatively substitute for sun-ray exposure) | 3 - 4  | 3 - 4    | 3 - 4        | 2 - 3   |

Remarks: * Mori fabric that has been dyed and fixed with kapur tohor; class 1 (very poor); class 2 (poor); class 3 (moderate); class 4 (good); and class 5 (very good/best coloring-performance)
3.2. Yields of dry extracts

Natural coloring chemicals or pigments commonly used in industries are in the form of dry extracts, which are preferable to wet extract forms. This is because wet extracts cannot be stored for a long time. Wet extracts are usually used by the related crafters and home industries, whereby their locations are usually close to raw material (wet extracts) sources or places. Dry extracts, before being used, are added to the colorless materials, which could function as color fixing.

Data about yields of dry extracts, were presented in Table 3; and strengthened through statistical analysis (Appendix 1), which revealed that the acquirement of dry-extract yields varied significantly due to different kinds of forest plant origins (for the waste extracts), immersing durations in water, heating temperatures (during the waste extraction), and ratios between the waste materials and water.

Extraction of barks and woods waste, without undergoing water immersion (control) brought out the lowest yields of dry extracts (Table 1). Longer immersion durations (to 12 and 24 hours) tended to increase the yields of dry extracts (as confirmed through the Tukey test; Appendix 1). This is because during the immersion the diffusion of water into wood/bark tissues and cells aptly intensified with longer immersion duration, thereby softening the tissues, weakening the bondings between cell walls and coloring pigments, and hence causing them to easily move out during the further extraction stage. In this research, it turned out that 24 hours immersion imparted greater dry-extract yields significantly, compared to the yields with 12 hours immersion.

Weight ratios between the waste and water solvent, during the waste extraction significantly affected the dry-extract yields (Table 3, Appendix 1), whereby the greater the ratio (1:4) the higher would be the yields of dry extracts; and conversely the lower yields for the lower ratio (1:6). Apparently with such 1:6 lower ratio, the water might also intensify the hydrolysis on the removable matters forming more fragmented particles or simpler compounds with lower molecular weights in the filtrates (after passing through the cloths), which presumably prone to volatilization during the oven drying, thereby lowering the dry extract yields.

Table 3. Average yields of dry extracts from waste materials (woods and barks) of 4 forest plant species

| Immersion durations * (before waste cooking), Hours | Weight ratio between waste materials and water | Cooking/Extraction temperature (°C) | Forest plant species/Waste materials/Yields of dry extracts (%) |
|-----------------------------------------------------|-----------------------------------------------|-----------------------------------|---------------------------------------------------------------|
|                                                     |                                               | Secang woods                      | Secang barks | Ketapang woods | Ketapang barks | Bakau minyak barks | Mangium barks |
| 0 (control)                                         | 1 : 4                                         | 60                                | 13.51       | 14.30         | 15.27         | 13.72             | 17.72         | 11.83         |
|                                                     |                                               | 70                                | 14.14       | 15.10         | 16.27         | 16.42             | 19.83         | 15.43         |
|                                                     | 1 : 6                                         | 60                                | 11.91       | 10.81         | 11.81         | 11.52             | 14.90         | 9.93          |
|                                                     |                                               | 70                                | 11.47       | 12.32         | 14.18         | 14.31             | 13.49         | 12.53         |
| 12                                                 | 1 : 4                                         | 60                                | 23.61       | 24.02         | 25.51         | 19.00             | 22.47         | 17.14         |
|                                                     |                                               | 70                                | 19.71       | 23.03         | 24.31         | 21.86             | 25.24         | 19.68         |
|                                                     | 1 : 6                                         | 60                                | 19.07       | 19.11         | 20.35         | 16.59             | 20.01         | 14.93         |
|                                                     |                                               | 70                                | 18.02       | 19.11         | 20.35         | 19.66             | 19.16         | 17.71         |
| 24                                                 | 1 : 4                                         | 60                                | 20.60       | 21.51         | 22.67         | 20.83             | 24.96         | 18.75         |
|                                                     |                                               | 70                                | 21.35       | 22.50         | 23.84         | 24.92             | 28.04         | 22.87         |
|                                                     | 1 : 6                                         | 60                                | 18.72       | 17.50         | 18.63         | 18.23             | 22.24         | 16.41         |
|                                                     |                                               | 70                                | 18.20       | 19.22         | 20.44         | 21.62             | 21.05         | 19.44         |

Remarks: Yield data average of five replications; *Regarded as pre-extraction.
Further, waste-cooking temperature (during the waste extraction) also significantly affected the dry-extract yields (Table 3, Appendix 1), but this depended on the species/portions of plant origins. For bark extracts (except for secang bark), highest yield was achieved at 70°C extraction temperature. This occurrence was commensurate with the research results by [15], whereby they performed the extraction on bakau (Rhizophora sp.) tree barks at also 70°C extraction temperature. It seemed that at 70°C temperature there occurred an increase in kinetic energy of the extractives as well as of the water solvent, thereby intensifying the movement of water molecules, enhancing the collisions between extractive molecules and water molecules which brought out the bark extracts in more fragmented shapes (smaller particle sizes) that could pass through the cloths even more easily into the filtrate, hence removing more intensively of bark extractives, and accordingly increasing the yields of dry extracts, compared to the yields at lower temperature (60°C). At 70°C extraction temperature, the yield of dry extracts (indicatively predominated by tannins) from bakau minyak barks was the highest, which achieved 28%, while at 60°C temperature the yield was about 25% (Table 3).

4. Conclusion
The research results of the 6 types of plant waste as a natural coloring agent showed:
1. The qualities (coloring performance) of bakau minyak barks extract that was applied for coloring mori fabrics (followed with kapur tohor fixation treatment), associated with color-fade/leaching resistance of the fabrics, were the best on rubbing/ironing process (good until very good quality), while the washing and sun-ray exposure shows moderate to good quality.
2. The highest dry extract yield was 28.04%. It is achieved by implementing 24 hours immersion of the extracts from bakau minyak (Rhizophora apiculata) barks in water (as pre-extraction or before extraction), followed with the extraction that employed weight ratio between the extracted barks and water at 1:4 and extraction temperature at 70°C.

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Appendix 1. Analysis of variance on yields of dry extracts, regarding the effect of kinds of forest plants’ waste origins, waste-immersion durations, waste-boiling/extraction temperatures, and weight ratios between waste materials and water (w/v)

| Sources of variance | df | SS          | MS          | F-calculated       | Pr > F |
|---------------------|----|-------------|-------------|--------------------|--------|
| L (Kinds of waste origins) | 5  | 709.67073   | 141.93416   | 341.51**            | 0.0001 |
| S (Immersion duration/treatment in water) | 2  | 3.743.05239 | 1.871.52619 | 4.503.13**          | 0.0001 |
| H (Cooking/Extraction temperature) | 1  | 143.56521   | 143.56521   | 345.44**            | 0.0001 |
| P (Weight ratio between wastes and water solvent) | 1  | 931.61104   | 931.61104   | 2.241.58**          | 0.0001 |

Interactions:
| L*S          | 10 | 176.66832   | 17.66683    | 42.51**             | 0.0001 |
| L*H          | 5  | 160.00539   | 32.00108    | 7.00 **             | 0.0001 |
| S*H          | 2  | 25.47130    | 12.73565    | 30.64**             | 0.0001 |
| L*S*H        | 10 | 23.92751    | 2.39275     | 5.76**              | 0.0001 |
| L*S*P        | 5  | 49.18544    | 9.83709     | 23.67**             | 0.0001 |
| S*P          | 2  | 8.67834     | 4.33917     | 10.44**             | 0.0001 |
| L*S*P*       | 10 | 10.81330    | 1.08133     | 2.60**              | 0.0049 |
| H*P          | 1  | 4.54276     | 4.54276     | 10.93**             | 0.0011 |
| L*H*P        | 5  | 62.61786    | 12.52357    | 30.13**             | 0.0001 |
| S*H*P        | 2  | 7.00585     | 3.50293     | 8.43**              | 0.0003 |
| L*S*H*P      | 10 | 9.96060     | 0.99606     | 2.40**              | 0.0096 |

Remarks: df = degrees of freedom, SS = sums of squares, MS = mean squares; Pr = probability; ** = very significant effect (Pr < 0.01); CV (coeff of variation) of yield data = 3.502%; the Tukey tests consecutively for L factor, D0.05 (minimum significant difference) = 0.3777; for S factor, D0.05 = 0.1961; for H factor, D0.05 = 0.1338; for P factor, D0.05 = 0.1961; for highest level of interaction (L*S*H*P), D0.05 = 0.7164.