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Unveiling the Potential of Secang (*Caesalpinia sappan L.*) as a Novel Tanning Agent: A Promising Alternative for the Leather Industry

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Article

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ABSTRACT

The exploration of vegetable tanning's natural resources is fascinating as an alternative to basic chromium sulfate for its natural abundance and considerable environmental impact. In this work, an attempt has been made to extract vegetable tannin from Caesalpinia sappan L. bark using water as a solvent with different levels of temperatures and incubation times. The highest yield efficiency was observed at 80 °C for 4 hours. The presence of tannin content and polyphenolic compounds were screened by the phytochemical analysis using Iron (III) chloride ($FeCl_3$), sodium hydroxide (NaOH), and ensured by the thin layer chromatography (TLC), and the Fourier transform infrared (FTIR). The extracts were then determined by percentage of extract yield, phytochemical, tannin type, moisture, total solids, and tannin characteristics. The findings indicate that the extract of Secang contains 4.98±0.28% total moisture, 95.02 ± 0.28% total solids, and 72.12 ± 2.81% total soluble solids, 52.28 ± 1.79% tannins, and 19.84 ± 1.41% non-tannin. Moreover, Fourier transform infrared (FTIR) analysis revealed a clear functional group within Caesalpinia sappan L., as evidenced by the type of tannin that is condensed. The TLC assay exhibited a considerable intensity under UV lights. It included several shades of light blue and blue, which could indicate the presence of secondary metabolites and tannin. Compared to mimosa, as the commercial standard for tanning agents in the leather industry, Caesalpinia sappan L. presents promise as an eco-friendly substitute for basic chromium sulfate and a new alternative source of vegetable tannin for the leather industry.

KEYWORDS

secang, Caesalpinia sappan L., vegetable tanning, tannin extraction, leather industry

INTRODUCTION

The leather industry holds significant importance globally due to its economic, cultural, and historical significance. Leather is a versatile material widely used in fashion, footwear, furniture, automotive, and luxury goods. It is highly prized for its robustness, visual attractiveness, and superior qualities [1]. Commonly, tanning agents like chromium salts have been widely employed due to their effectiveness in transforming raw animal hides into durable and supple leather. However, using these agents has raised severe environmental and health issues. Chromium salts are highly toxic, and their disposal contributes to water pollution and soil contamination. Moreover, the tanning process consumes large amounts of water and energy, further exacerbating its environmental impact. Tanning is a process that has two stages which occur simultaneously. The first stage involves the tanning agents diffusing into the hide through capillaries between the fibres and fibre bundles and into the spaces between the individual fibrils that make up the fibres. The second stage involves the combination of the tanning agents with collagen, the protein that makes up most of the hide fibres [2]. In addition, the pollution load of chrome as a tanning agent generates a high amount of biological oxygen demand (BOD) and chemical oxygen demand (COD), total suspended solids (TSS), total dissolved solids (TDS), and chromium (Cr). The major physiochemical characteristics of leather industrial effluent contain BOD (350 to 1248 mg/L), COD (850 - 400 mg/L), TSS (500 - 700 mg/L), TDS (532 - 14.000 mg/L), and Cr (10.2 - 725 mg/L) [3]. These values are much higher than the tolerance limits for industrial effluent discharged into land surfaces or public sewers.

Given the growing awareness of sustainability and the need to reduce the ecological footprint of industries, there is an essential demand for alternative tanning agents that are both effective and environmentally friendly. The continued survival of the leather industry is contingent on adopting sustainable tanning techniques. Progress in this area is paramount to ensure we can continue enjoying high-quality leather goods without compromising the environment [4].

Mimosa, sumac, tara, quebracho, divi-divi, valonia, chestnut, and oak are among the conventional vegetable tanning agents that have been used introduced extensively for producing eco-friendly leather since generating less pollution than chrome tanning and are considered “green tanning” method [5-7]. The vegetable tanning process involves a polyphenols group of vegetable tanning, which reacts with the collagen of leather through the hydrophobic bond, producing an interaction between protein-polyphenol complexation [8]. Further, only mimosa and quebracho are outstanding for vegetable tanning and suitable for industrial applications, although the hydrothermal stability of collagen is low compared to chrome tanning agents. In addition, mimosa requires a purification process of extract, which contains a mixture of phenolic compounds, simple sugars, organic acids, and hydrochlorides [9]. The harvesting time of both plants also needs a long period of 7 - 10 years for generating a suitable vegetable tannin extraction. Another challenge decreases the inclination of mimosa and quebracho import [10]. So, it has become imperative to investigate alternative local sources for vegetable tannin-producing plants widely distributed throughout Indonesia.

Caesalpinia sappan L., commonly known as Secang or Sappanwood, is a tropical tree species native to Indonesia [11]. The tree is well suited for mountainous regions with moderate temperatures and grows at an elevation of 1000 m above sea level; it has a spiny trunk and branches, is covered in reddish-brown hairs, and the stem is round and brownish-green in colour [12]. It is found in Southeast Asia and the Pacific Islands and is reported to be native to Southeast Asia, especially in countries like Malaysia, India, and Indonesia [13]. Among the majority of plant species found in Indonesia, this tree is traditionally used in the production of natural dyes [14], medicinal purposes [15], and sources of drink [12].

Indonesia is enriched with abundant natural resources of plants with heterogeneous applications like traditional medicine and non-industrial utilization. Up to now, the use of secang in leather processing is seldom known. Therefore, exploring new extract plant materials for vegetable tanning is essential for the sustainability of the Indonesian leather industry. Secang is a potential emerging candidate vegetable tannin material for leather processing. With the promising availability of the secang plant in Indonesia and the high content of tannins, flavonoids, and phenolic compounds, this plant can be processed as an asset in the various application and economic aspects [16]. Hence, the work aims to evaluate the characteristics of extracted tannin from *Caesalpinia sappan L*. bark as a new source of tanning material and their characteristic compared to mimosa.

EXPERIMENTAL

Materials and Methods

Tanning extraction

Approximately 10 g of secang barks were extracted by incubating them in 100 ml of distilled water (1:10 (w/v)) in a beaker glass. For one hour, the incubation was carried out at different temperatures (60, 70, and 80 °C). The solution was filtered, and the filtrate was collected in a beaker. The filtrates were dried in an oven at 60 °C. The best temperature variation results were taken as a reference for the extraction treatment's temperature for the incubation time (1, 2, and 4 hours). The resulting powder was analyzed for extract yield, phytochemical screening, tannin characteristics, and thin-layer chromatography.

Extract yield was determined using the following equation;

$$Extract\ yield = \frac{\text{Extract obtained (g)}}{\text{Amount of moisture} - \text{free barks used (g)}}\ x\ 100$$

Phytochemicals screening

Ten millilitres of distilled water were mixed with one gram of tannin powder. Two drops of ferric chloride were added after the solution was boiled and filtered. A blue-black or greenish solution indicated the presence of tannins in the sample.

Determination of tannin type

Two millilitres of the tannin extract were mixed with a few drops of aqueous potassium hydroxide. Condensed tannins were present, as evidenced by the development of the red colour. The absence of a colour change indicated that there were no hydrolysable tannins present.

Fourier transformation infrared (FTIR) spectroscopic study

The structural identification of the extracted sample was carried out for secang and mimosa by employing the KBr sample disc method using FTIR spectroscopy (IR Affinity-1S, Shimadzu, Japan). The powder of the extracted tannin was mixed with KBr (FTIR grade) and scanned at the range of 4000 - 400 cm^{-1}[17].

Thin layer chromatography (TLC)

Secang and mimosa extracts as much as 0.5 mg were spotted on the plate as an initial marker of the stationary phase. The plates were then immersed in eluent solution (mobile phase) with a mixture of 1-butanol: formic acid: distilled water (4:1:1). The TLC plate was removed until it reached 90% on the plate, then it was observed under UV light.

Determination of moisture and total solids

Five grams of finely ground tanning material was transferred into a tared wide-mouth weighing bottle and weighed accurately. It was dried at about 98.5 - 100 °C in an oven for 3 to 4 hours, cooled in a desiccator for about 20 minutes, and weighed again accurately. The process of drying and weighing was repeated until two weights at an interval of one hour did not differ by more than 2 mg.

$$\text{Moisture content (by weight)} = \frac{(\text{W1} - \text{W2})}{\text{W1}}\, x\, 100$$

$$\text{Total solid content (by weight)} = \frac{\text{W2}}{\text{W1}}\, x\, 100$$

Where: W1= Weight in grams of the material taken for a test; W2= Weight in grams of the residue left after drying

Determination of non-tannin

One hundred millilitres of unfiltered secang bark extract (V1) were mixed with 6.25 g of chrome hide powder (W1). The solution was homogenized by rotary stirring at 100 RPM for 20 minutes. The filter paper was used to filter the solution, and a beaker was used to collect the filtrate. A porcelain cup was filled with 50 millilitres (V2) and then heated to 105 °C to dry it out. After heating the sample, the starting weight of the porcelain cup is subtracted from the sample's weight to determine the final weight (W2). The following formula can be used to determine non-tannic content [18]:

$$\%\ \text{Non tannin} = \frac{\text{W2}}{\text{W1}}\ x\ \frac{\text{V1}}{\text{V2}}\ x\ 100\%$$

Determination of tannins, tanning strength, purity of tannin

The determination of the tannin content, tanning strength, and purity of tannin was carried out by the Bureau of Indian Standards (BIS). The following formula can calculate tannin content:

$$\%\ \text{Tannin} = \%\ \text{Total soluble solids} - \%\ \text{Soluble non tannins})$$

$$\%\ \text{Tannin strength} = \frac{\%\ \text{tannin}}{\%\ \text{non} - \text{tannin}}$$

$$\%\ \text{Purify of tannin} = \frac{\%\ \text{tannin}}{\%\ \text{total soluble solid}}\ x\ 100$$

Measurement of pH

The pH of extracted secang bark tannins was noticed by using a pH meter according to the official method Bureau of Indian Standards [18]. Sample pH was measured after calibration of the pH meter in buffer solutions with pH 4 and 7.

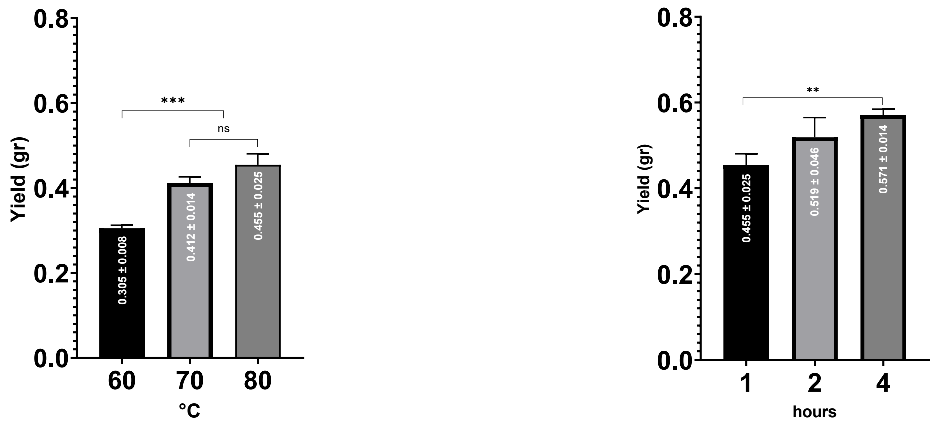
Data analysis

Data analysis for differences in temperature and incubation time used One Way ANOVA, followed by Tukey's multiple comparisons tests, with GraphPad Prism 8 software. Quantitative chemical composition (tannin analysis) between extracted secang and mimosa was analyzed using quantitative descriptions.

RESULTS AND DISCUSSION

Tannin extraction

In the previous study, secang was extracted using 60% ethanol as a solvent with the help of ultrasound-assisted extraction (40 kHz; 60 °C) [19]. In this work, secang wood was extracted using water as a solvent. The extraction was carried out by exposure to a heating process with various extraction temperatures of 60, 70, and 80 °C for 1 hour. Based on Figure 1a., the secang wood extraction produced the highest extraction yield at a temperature of 80 °C. However, the temperature between 70 °C and 80 °C was not significantly different ($P > 0.05$), but a significant difference ($P < 0.05$) was the temperature of 60 °C. Previous studies reported that the highest extraction yield using water as a solvent is 80 ˚C [20]. The temperature at which secang extraction is performed is a crucial factor that affects the per cent yield. Increasing the temperature causes a drop in the surface tension and viscosity of the solvent, reducing the cavitation threshold value. This lower value facilitates the formation of cavitation bubbles that can damage the cell walls of the plant. However, this damage also causes the solvent to diffuse into the plant matrix, which makes it easier to extract the compounds and improve the yield of the extract.



a) b)

Figure 1. The extract yield on different temperatures (a) and extraction time (b)

Different incubation times during Secang wood extraction at 80 ˚C resulted in varying yields ($P < 0.05$). According to Figure 1b, the 4-hour incubation time produced the highest yield, while the 1-hour incubation time produced the lowest yield. These findings suggest that the extraction yield increases with longer extraction times. This is consistent with conventional extraction methods such as maceration or reflux, which require more than 3 hours of extraction time [21]. In addition, Unango et al. reveal that temperature is the key factor that affects the per cent yield during *Hagenia abyssinica* tannin extraction [20]. In this study, a high yield of extract was obtained by extracting at 80 °C for 4 hours. During the extraction process of secang wood, the percentage of yield increases in proportion to the length of time the wood is exposed to heat. This increase in yield percentage is due to the swelling and hydration of the solid wood material caused by the cavitation effect. This effect creates microjets on the material's surface, which can cause damage and make it easier for the solvent to penetrate the wood matrix. As a result, the solvent's ability to extract the secang wood increases, leading to a higher extract yield.

Phytochemicals screening

The qualitative analysis is an initial determination of the presence and type of tannin. The presence of tannin was observed upon the addition of a few drops of ferric chloride into the sample solution. The dark blue colour indicates that the sample contains tannin which is suitable for the tanning of leather. At the same time, the type of tannin (condensed or hydrolysable) was carried out by the addition of a few drops of saturated NaOH into the sample solutions. The precipitation of red or reddish clearly shows the presence of condensed tannins like mimosa and quebracho.

The phytochemical result of secang wood is given in Table 1. The result shows the extract of secang wood is present in tannin and corresponds to condensed tannin. Along these lines, the extract of secang wood is suitable for the tanning process due to the condensed tannin has large molecular size, the availability of additional phenolic groups for bonding, and is more reactive than hydrolysable tannin [22].

Table 1. Phytochemical results of the tannin test and test for condensed hydrolysable tannins

Phytochemical	Test	Colour	Secang	Mimosa
Tannins	$FeCl_3$	blue dark	+	+
Hydrolysable tannins	NaOH	no changing	-	-
Condensed tannins	NaOH	reddish	+	+

(+) the presence of the phytochemical; (-) the absence of the phytochemical

FTIR analysis

FTIR analysis was used to observe functional groups in the extracted materials with a specific spectra range of 4000 - 400 cm^{-1}, and the spectrum was presented in Figure 2. The FTIR examination of standard tannin includes the primary peaks that need to be taken into consideration: 1704 cm^{-1}, 1707 cm^{-1}, and 1325 cm^{-1} [17]. According to the result obtained in this study, the IR spectral bands have confirmed that the extract of *Caesalpinia sappan L*. sample contains condensed tannin. This was proven by the indication of absorption bands in the range of 1704 - 700 cm^{-1} region. These bands are confirmed to be an asymmetric stretching of ester (C-O); the band at 1612 cm^{-1} belongs to the stretching of C=C bonds of the aromatic ring. The absorption above 3000 cm^{-1} region is assigned to (CH_2 or CH_3) and OH bond stretching vibration in the phenolic and aliphatic structures, which are a band at 2932 cm^{-1} and 3380 cm^{-1} [23]. This FTIR spectrum established that *Caesalpinia sappan L*. extract is condensed tannin.

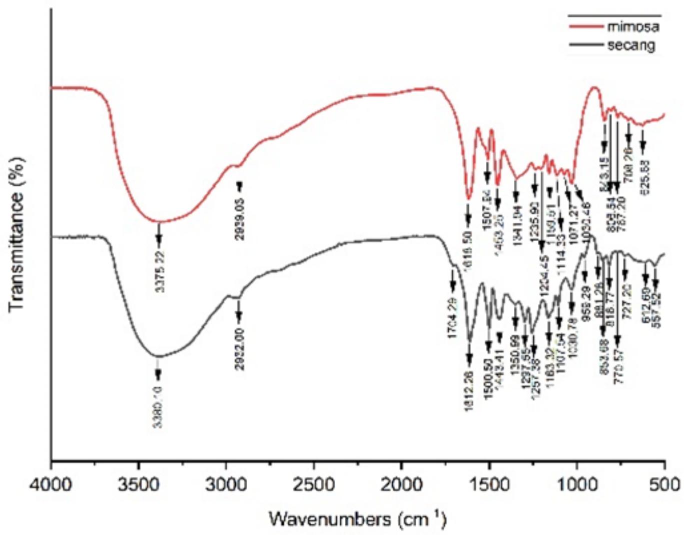


Figure 2. Fourier transformation infrared (FTIR) spectrum of secang and mimosa

Thin Layer Chromatography (TLC)

The TLC profile of the extracts was analyzed, and the Rf values of each spot were presented in Table 2, the result proved the existence of tannin in the extract. Clear zones were observed at Rf of 0.894, 0.788, and 0.729. Among these spots, 0.894 and 0.729 show strong intensity in UV lamps and contain different colours of light blue and blue, which may correspond to the presence of tannin and secondary metabolites [23].

Table 2. Rf values of secang extract

Plant specimen	Test	Distance travelled by spots (cm)	Distance moved solvent (cm)	Rf values of the spots
Caesalpinia sappan L. extract	Light Brown	3.80	4.25	0.894
	Light Purple	3.35	4.25	0.788
	Yellow	3.10	4.25	0.729

Physical characteristics

The physical characteristics of tannin extract are shown in Table 3. The results show that the extract of secang wood contains total moisture, total solids, and total soluble solids in the extract of secang wood as 4.98 ± 0.28%, 95.02±0.28%, and 72.12 ± 2.81%, respectively. The extract also contains tannins (52.28 ± 1.79%) and non-tannin components (19.84 ± 1.41%), while the level of tannin strength and purity ratio was found to be 2.642 ± 0.166% and 0.550 ± 0.018%, respectively. The pH of the extract was 4.667 ± 0.021, which is within the required range (pH 4-6) for the vegetable tanning process. Thus, secang wood is a potential alternative vegetable tannin material for leather industries. However, the analysis of standard commercial vegetable material had greater amount of moisture (12.070 ±

0.069%), total soluble solids (86.627 ± 3.606%), tannin (63.047 ± 5.162%), non-tanning (23.580 ± 1.961%), and lower total solid (87.930 ± 0.069), comparable on tanning strength (2.698 ± 0.449%), purity (0.717 ± 0.059%), and pH (4.537 ± 0.031). In recent studies in Table 4, secang was found to have high total solids content as a vegetable tanning agent. Characteristics of plants, such as species, age, and source, as well as soil composition, pH, bark dimensions, and extraction methods, along with the types of solvents used, can all contribute to variations [22].

Table 3. Quantitative chemical composition (tannin analysis) of secang and mimosa

Properties	Secang	Mimosa (standard)
Total solids (%)	95.022 ± 0.283	87.930 ± 0.069
Moisture (%)	4.978 ± 0.283	12.070 ± 0.069
Total soluble solids (TSS, %)	72.117 ± 2.811	86.627 ± 3.606
Non tanin (NT, %)	19.838 ± 1.411	23.580 ± 1.961
Tannin (% T = TSS - NT)	52.279 ± 1.793	63.047 ± 5.162
Tanning strength (% TS = T/NT)	2.642 ± 0.166	2.698 ± 0.449
Purity ratio (% T/TS)	0.550 ± 0.018	0.717 ± 0.059
pH	4.667 ± 0.021	4.537 ± 0.031

Table 4. A recent study of the discovery of organic tanning material

Plants	Tannin (%)	Moisture (%)	Non-Tanin (%)	Total solids (%)	Total soluble solids (%)	pH	Ref.
Mimosa (standard)	22-48	8.8	-	-	-	4.8	[2,24]
Caesalpinia sappan L. (Secang)	52.27	4.97	19.83	95.02	72.12	4.68	This study
Pontederia crassipes	22-25	-	-	-	-	4.35	[24]
Xylocarpus granatum	47.8	-	-	-	-	-	[25]
Solanum incanum	12.13	7.59	9,32	94.01	21.45	5.2	[26]
Tessmannia burttii	61.2	-	-	-	-	-	[27]
Acacia mearnsii	84.3	-	-	-	-	-	[27]
Acacia xanthophloea	23.8	8.90	7,20	91.10	31.00	4.5	[28]
Acacia nilotica	16.80	7.10	11.20	92.90	28.00	5.62	[28]
Hagenia abyssinica	11.73	5.90	5.60	94.10	17.33	4.96	[28]
Lawsonia inermis L.	10.2	8.97			25-33		[29]

CONCLUSION

Secang wood extraction at 80 ˚C for 4 hours was found to have the highest yield. The phytochemicals screening and FTIR chromatogram confirmed that the extract of secang wood contains tannins of the condensed type. The physical characteristics of secang wood and mimosa were found to be representative of differentiating the effect of leather tanning. The tannin content of secang wood is slightly lower compared to mimosa. However, the tanning strength of both samples was above the required minimum (1.5) for tanning materials. Therefore, secang wood should be considered for commercial tanning of leather that can promote eco-friendly tanning practices.

Author Contributions

Conceptualization - Abidin MZ; Methodology - Abidin MZ, Yuliatmo R; Investigation and Resources - Kurnianto AS, Wicaksana AW; Writing original draft presentation - Kurnianto AS, Wicaksana AW, Yuliatmo R, Putra MP, and Abidin MZ. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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