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Potential and Application of Vegetable Tanning Materials from Industrial Forest Plantation in Indonesia

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***Abstract**: Vegetable tanning materials are sourced from plants that contain tannin compounds. Tannins are obtained through the extraction method from the roots, stems, bark, or fruit of the original plant. There are several forest managements companies in Indonesia that produce wood from plant species Acacia auriculiformis and Acacia mangium. Both species are known as Acacia. Acacia is an important source of tannin for material tanner vegetables. This wood is produced as a raw material for the pulp and paper industry. However, the bark contains tannin and has not been used optimally. Therefore, the study potential and possible applications as vegetable tanning agents have been carried out. The research was started by investigating the availability of bark, extracting tannins to obtain extracts containing tannins. Furthermore, the extract obtained was applied as a vegetable tanning agent for the goatskin tanning process. The results showed that the bark of the Plant Industry Forest has the potential to be developed as a source of tannins for material vegetable tanning agents. The application of acacia bark extract in goatskin tanning has obtained tanned leather that meets the Indonesian National Standard.*
***Keywords:** Industrial plantation forest; tanning agents; leather*

1. Introduction

The potential of the leather processing industry in Indonesia is very large. The importance of the leather tanning industry in supporting the economy in a country as in Italy and other countries. According to the General Directory of Livestock and Animal Health of the Republic of Indonesia (2011), the leather tanning industry is only able to produce 350,000,000 square feet (sqft)/year, while the demand for the footwear and leather goods industry is 673,000,000 sqft/year, so every year it is still experienced a shortage of 323 million sqft [1]. When viewed in terms of price, according to the Association of Leather for Us Company, goatskin that has been tanned (grey smooth goatskin) measuring 66.5cm x 50 cm is valued at 28.00 Euros equivalent to Rp. 446.000, -. The Indonesian Tanners Association (APKI) consists of 180 large and medium-sized tanneries operating with a production of 87 million feet/year with exports of 5,500 tons. Leather tanning is a series of processes to change the state of raw leather that is not resistant to fungi, bacteria, microorganisms, chemicals, and heat, to become more stable and resistant to a few conditions that have the potential to cause damage [1]. The common tannery material used in the

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industry today is mineral tanner in the form of chrome sulfate which has the advantage of being easy to process and superior to the resulting tanned leather [2]. However, these tanning materials cause environmental pollution [3–6]. Currently, the waste from the leather tanning industry that uses chromium is classified as toxic and hazardous waste which poses a hazard to the health of living beings and the environment. In the tanning process, chromium reacts with the skin between 54-57% and the rest will be wasted with waste [7].

Vegetable tanning agents can be another alternative to the use of chromium. Vegetable tanner is an environmentally friendly product. In the tanning industry, currently, the vegetable tanning agents used are Mimosa and Quebracho. In Indonesia, both types of tannery materials are imported products. The world's total production of tannins is 200,000 tons/year, of which 90% of the tannins are condensed tannins obtained from plant extracts [8] . The price of powdered tannins in the world market according to search results on several online sites (alibaba.com), for example, the trade name of mimosa is Rp. 1,447,500, - in packs of 25 kg or Rp.57,900, -/kg.

Mimosa is an extract of the *Acacia mearnsii* plant and Quebracho is a tanning agent extracted from the plants *Schinopsis balansae* and *Schinopsis lorentzii* [9]. Several types of Acacia plants as vegetable tanning agents have also been reported, including [8,10–13]. Based on this study, researchers see the opportunity for Indonesia to have Acacia-producing forests between *Acacia mangium* and *Acacia auriculiformis* sourced from Industrial Plantation Forests. Industrial Plantation Forests is a forest developed to increase the potential and quality of production forest by applying intensive silviculture to meet the needs of raw materials for the pulp and paper industry as well as the production of processed wood. This wood production leaves the bark that has not been used optimally. In a previous study in one HTI company, the bark was left in the forest area as a natural fertilizer for the land before being replanted. This condition provides an opportunity that the Acacia bark can be used as a source of tannins for vegetable tanning agents.

Vegetable tanning materials are sourced from plants that can be used directly or extracted first [14]. Several studies on extracts of tannins from plants as tanning agents have been carried out including [8,11,15–18]. The tanning material consists of tannins, non-tannins, and other water-insoluble compounds [19]. The effectiveness of vegetable tanning agents can be done through the extraction of tannins. Extraction of tannins from various plants as tanning agents has been carried out in previous studies [20]. Tannin compounds are polar in the form of glycosides [6]. Thus, tannins can be extracted using water as a solvent. [19] Tannins found in acacia bark can be obtained through the extraction process. Various tannin extraction methods have also been reported including the water bath method, with autoclave, reflux, and Microwave-assisted extraction (MAE) with the aim of short extraction time, use of a small amount of solvent, higher extract yield, and lower costs [8, 12,13,21,22].

Trials of vegetable tanning agents from bark extracts of *Acacia mangium* and *Acacia auriculiformis* from Industrial Plantation Forests have been carried out [20,23]. The skin raw material used is goatskin. The opportunity to use goatskin as raw material for tanning is quite widely used in the leather industry [24]. For comparison, the leather produced using extracts of the bark of *Acacia mangium* and *Acacia auriculiformis* from Industrial Plantation Forests was compared with the leathers that were leather with commercial tanning agents mimosa and quebracho.

2. Methods

2.1. Study of bark potential from industrial forest plantations

A study on the potential availability of *Acacia* bark was carried out by collecting data from industrial forest plantation companies and the Indonesian Central Statistics Agency. The collected data is then converted using the proportion of bark that has been previously determined by [25], which is 15% of the total tree trunk.

2.2. Extraction of tannins from the bark of *A. mangium*

The bark of *A. auriculiformis* and *A. mangium* were cut into pieces with a size of 1 -2 cm and dried to a moisture content of ±15%. Furthermore, the dry skin was ground with a disc mill powder machine and filtered to obtain material that passed a 60-mesh sieve. Extraction of tannins was carried out using water as a solvent. Comparison of materials with solvents 1:20. Process extraction was also carried out by heating at 100 ^{0}C for 30 minutes. Then the mixture was filtered using filter paper to obtain the filtrate. The filtrate was concentrated using a vacuum rotary evaporator to obtain the extract. Extraction results will be obtained from tannin extract to calculate the yield with the formula 1 :

$$\text{Yield (\%)} = \frac{\text{weight of extract (g)}}{\text{weight of take (g)}} \text{ x } 100\% \qquad \text{....(1)}$$

2.2. Determination of tannin extract content

Determination of tannin content by the skin flour method [26]. The material is weighed as much as 30g (based on dry weight) in a 1000 ml Erlenmeyer. Then 600 ml of distilled water was added. The mixture was stirred using a magnetic stirrer for 20 minutes. Then the mixture was filtered using filter paper. After the filtrate volume reaches 200 ml, the filtrate is set aside. Furthermore, the percentage of total soluble material is calculated by the formula 2:

$$\text{Total soluble } (\%) = \frac{\text{weight of residue}}{\text{weight of take}} \text{ x } 100\% \qquad \text{....(2)}$$

The non-tannic Determined using leather flour. The previously obtained filtrate was taken as much as 2 x 75 ml and put into 2 Erlenmeyer pieces. Furthermore, the first Erlenmeyer added 6.5 g of skin flour. The mixture was stirred using the shaker for 20 minutes. Furthermore, the filtrate that has been stirred using a shaker is filtered using a suction pump to obtain the filtrate. The used skin flour is put back into the second Erlenmeyer which contains the filtrate. The skin flour is broken down with the help of a stirring rod. The mixture was stirred using a shaker again for 15 minutes. Then filtered using a porcelain funnel with the help of a suction pump to obtain the filtrate. The two filtrates obtained were combined, then filtered through filter paper. The obtained filtrate is made up to 150 ml. then the percentage of non-tannin material is determined by the formula 3:

$$\text{non} - \text{tannin}(\%) = \frac{\text{Weight of residu}}{\text{weight of take}} \text{x } 100\% \qquad \text{....(3)}$$

Determination of tannin content is calculated using the formula 4:

$$\text{Tannin content } (\%) = \text{total soluble} - \text{non tannin} \qquad \text{....(4)}$$

2.3. Analysis of extractives by GC-MS

Analysis of extractives using the Shimadzu GCMS-QP-2010 plus gas chromatography system (Shimadzu, Kyoto, Japan). A gas chromatographic column was used for matrix separation in samples with DB-5MS and DB-1MS (30 m 0.25 mm 0.25 m) columns from Agilent (Agilent Technologies, Santa Clara, CA, USA). The injector temperature used is 250˚C with an injection volume of 2µL. The carrier gas was helium at a flow of 1 ml/min. The temperature in the injection line was set at 300˚C. The initial temperature of 40˚C was given for 2 minutes, then increased by 10˚C/min until it reached 300˚C and remained constant at that temperature for 3 minutes. Chromatograms were obtained in full scan mode *(*m/z 35-500). Identification of compounds was carried out using the mass spectral libraries NIST47 and the *Wiley Registry TM ^{7th} edition* [27].

2.4. Application of extracts as vegetable tanning agents

Tanning Process methods standard of Center for Leather for Leather, Rubber and Plastics (BBKKP) Yogyakarta, that is: the leather used is goatskin dry durable salt. For tanned skin preparation consisting of soaking, washing, liming, fleshing, deliming, and Pickel. The pickle skin was followed by a tanning process using vegetable tanning agents bark extracts of *Acacia mangium*, and *Acacia auriculiformis*. Furthermore, the leather is overnighted for 24 hours then the leather is washed to remove the remaining tanning agent substances that are still attached, then dried by stretching. The resulting leather is prepared for analysis and testing.

2.4.1. Leather test sampling

The leather that was used as the test sample according to Indonesian National Standard (SNI 0463-1989-A) the leather sample was taken according to the test sample on the back, each (20 x 20) cm^2, located 5 cm from the dorsal line and 12.5 cm from the base of the tail. A test sample was taken from the abdomen area (7.5 x 5) cm2, located in the middle of the abdomen on the boundary line between the back and the neck. More details on taking test samples can be seen in Figure 1.

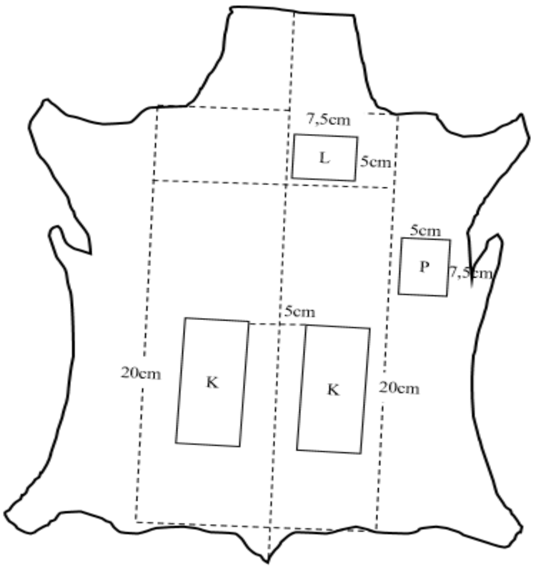


Figure 1 . Sampling pattern for leather analysis and testing where P = Stomach, L = Neck, and K = Krupon

2.4.2. Chemical analysis

Leather material for chemical analysis refers to the method of SNI 0463-1989-A, Chemical analysis includes wrinkle temperature, tannins bound, and degree of tanning. The degree of tanning is the degree of ripeness of the leather, calculated based on the content of the bound tanner divided by the concentration of the raw skin content. The tannins bound and the degree of tanning are calculated by the following formula 5:

$$\text{Tannin bound }(\%) = 100\% - (\text{water content} + \text{fat content} + \text{water soluble content} + \text{ash content} + \text{raw skin content})\%$$

$$\text{Degree of tanning}(\%) = \frac{\text{Tannin bound}}{\text{Raw skin content}} \text{x}100\% \qquad \text{....(5)}$$

2.4.3. **Physical properties**

Physical properties of leather testing, including tensile strength, elongation at break, and thickness. The test sample preparation pattern is in accordance with the ISO 3376:2011 standard. can be seen in Figure 2. The test is carried out using the Universal Testing Machine.

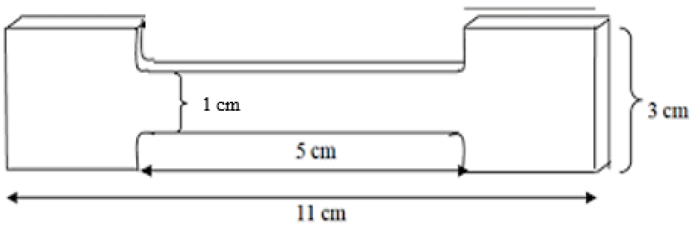


Figure 2 . Sample preparation physical properties

The formula for calculating tensile strength and elongation at break is as follows:

$$\text{Tensile strength} = \frac{\text{Power value}}{\text{Leather lenght x Thickness}} \text{x N/cm}^2 \quad \text{....(6)}$$

$$\text{Kemuluran }(\%) = \frac{\text{(lenght increase)}}{\text{Sample lenght}} \text{x}100\% \qquad \text{....(7)}$$

3. Results and discussion

3.1. Bark potential of industrial plantation forest

An industrial plantation forest is a forest whose development aims to support the growth of the timber industry by providing industrial raw materials. This wood production is produced from natural forests through the activities of Forest Concession Rights, Industrial Plantation Forests, and community forest activities. Forest areas are maintained as areas to produce or produce forest products for the benefit of public consumption, industry, and export. Based on Forestry data in 2020, the total wood production in Indonesia is 61.02 million m^3 which 52.63% are acacia plantations. Timber production in Indonesia is presented in Table 1 [28].

The highest wood production is in Sumatera with Acacia species reaching 69.32%. Furthermore, Java with a production of 12.80% consisting of mixed forest groups, and the Maluku area with a production of 2.60% with Meranti plant species. Acacia wood is a type of wood that is developed in large quantities, especially for the Sumatra region because it has soil suitable for the growth of these plants.

Table 1. Timber production in Indonesian.

Forest areas	Production (million m^3)	Total production (%)	Plant type
Sumatera	41,3	69,32	Acacia
Java	7,76	12,80	Mixed forest group
Borneo	9,71	16.02	Meranti Group
Sulawesi	0,21	0,34	Mixed forest group
Bali dan Nusa Tengara	0,02	0,03	Mixed forest group
Maluku	1,58	2,60	Meranti Group
Total	60,58		

Acacia plants are suitable for planting in peat areas and are also resistant to pests [29]. Regarding the use of Acacia bark as a vegetable tanning agent, Indonesia has great potential. Based on forestry data in 2020, the total amount of wood production in Indonesia is 60.58 million m $^{3.}$ Of the total production, 52.63% were Acacia plants. The production of wood species in Indonesia can be seen in Figure 3.

Figure 3. Timber production in Indonesia in 2020 [28]

It can be seen in Figure 3 that the largest production of logs is Acacia wood as much as 32.114 million m (52.63 percent), mixed forest group wood as much as 20.655 million m (33.85 percent), meranti wood group as much as 4.795 million m (7.86 percent) beautiful wood as much as 0.492 million m (0.81 percent), and other wood as much as 2.961 million m (4.85 percent). The opportunity to use Acacia bark as a source of tannins for vegetable tanning agent can be calculated based on the amount of bark in one tree trunk. If the proportion of bark is about 15% of the tree [25] then the available bark is 4.8 million m ^{3} . An illustration of the separation of the bark after the tree harvesting process can be seen in Figure 4.

Figure 4. Bark after tree harvesting in florests plantation

3.2. Bark Extract

The yield of *A. mangium* and *A. auriculiformis* bark extracts can be seen in Table 2.

Table 2. The bark extract of *A. mangium* and *A. auriculiformis*

Extract	Bark of *A.mangium*	*A.auriculiformis*
Yield (%)	23.53 ±1.12	25.96 ± 0.49
Water soluble (%)	44.62 ±065	46.93 ±0.35
Tannin content (%)	11.04 ±1.12	11.58 ±0.17

Extraction using a temperature of 100 ^{0}C showed that the yield of A. *auriculiformis bark* was higher than that of *A. mangium* bark extract. Thus, also the case with the water-soluble ingredients and levels of tannin extracts. The yield of the tannin extract is influenced by several factors including the solvent, particle size, time, and temperature used [30]. Other studies have reported that different tannin extraction processes will produce different yields. Extraction of tannins using an autoclave took place at a temperature of 121°C with a pressure of 10 lbs (1.1 bar) for 30 minutes to produce a yield of 19.15% [21]. Extraction of tannins using *microwave-assisted extraction* (MAE) using microwaves has an effect that heats the solvent mixture directly and reacts directly with water in the material which causes tissue rupture in plants so that the active compounds dissolve into the solvent [31].

3.3 Analysis of extractives by GC-MS

The results of the identification of extracts from the bark of *A. mangium* and *A. auriculiformis* using GC-MS showed several chemical compounds are presented in Table 3. Each of the ten (10) main chemical compounds of the two bark extracts was presented. The results of the analysis using GC-MS are depicted in the GC spectra of Figure 5. This study has been previously reported [20]. Identification of compounds from the bark of *A.auriculiformis* showed high yields, namely phenyl phenol 33.6%. Based on the retention time, the compound that appeared first in the *A.auriculiformis* variety was 3-methyl-2-heptanone with a retention time of 4,396 minutes, while *A.mangium* was a methyl(6H)dibenzo compound with a retention time of 10,943 minutes. The results of this analysis confirm that *A. mangium* and *A. auriculiformis are* suitable sources of tanning materials. Tanning substances such as tannins are phenolic compounds that can be extracted from plants, as well as these two types of bark. Tannins are a mixture of polyphenols that in plants form glycosides which when hydrolyzed break down into aglycones and glycans [19]. [33] Tannins are polyphenolic macromolecular compounds that can form complexes with metal ions, thus enabling them to be used as corrosion inhibitors on metals.

Table 3. Identification results of extractives from the bark of *A. mangium* and *A. auriculiformis*

No.	***A. mangium***			***A. auriculiformis***		
	Retention time (minute)	Componen	Area (%)	Retention time (minute)	Componen	Area (%)
1	21,649	Palmitic Acid	28,97	13,623	Phenylphenol	33,6
2	24,820	Oleic Acid	21,43	21,594	Palmitic Acid	13,5
3	10,943	Methyl(6H) dibenzo	5,71	10,442	Phenol	8,59
4	13,607	Phenylphenol	5,54	24,777	Oleic Acid	6,61
5	11,185	Geranyl acetat	3,6	28,047	Hexamethyl	6,59
6	13,009	5-hexasiloxane	3,58	29,242	2-ethylhexyl ester	6,59
7	25,236	Stearic Acid	3,42	4,396	3-Methyl-2-heptanone	4,66
8	20,788	Methyl palmitat	3,22	18,333	3-Icosene	4,11
9	14,366	2- Hidroksietil	3,09	22,064	1-Nonadecene	3,12
10	19,113	1-Hexadecene	2,99	14,704	1-Tetradecene	3,04

**Chemical nomenclature of CAS*

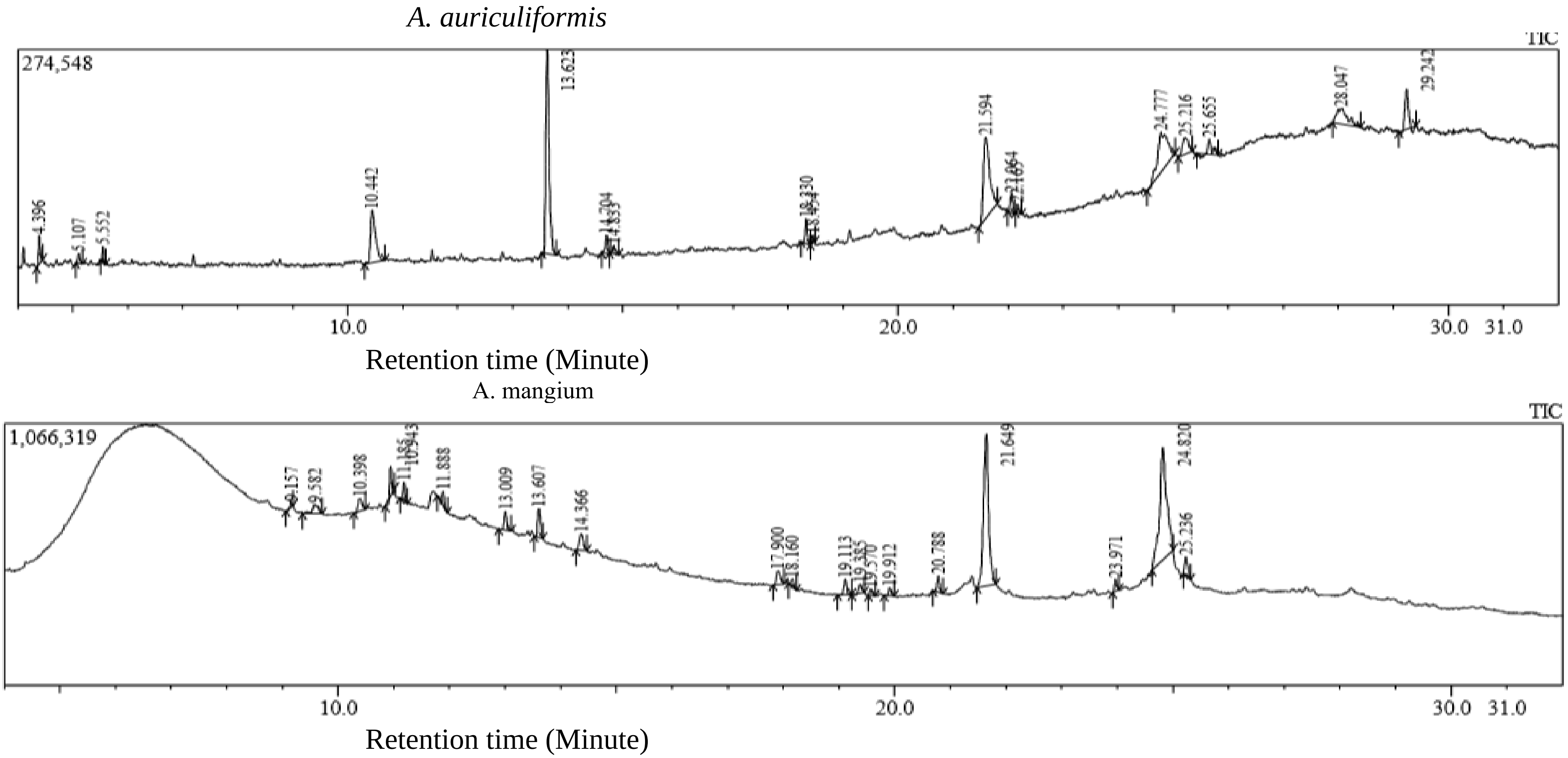


Figure 5. Chromatogram of chemical compounds of A. mangium bark from GC-MS Analysis

3.4. Characteristics of tanned leather

The characteristics of leather include the chemical analysis and physical properties of leather. Table 4 shows the characteristics of leather with *A. mangium, A. auriculiformis* bark extracts tanning agents, and commercial tanning agents such as mimosa and quebracho as well as the Indonesian National Standard of leather.

Table 4. Characteristics of leather with vegetable tanning agent from bark extracts of *A. mangium* and *A. auriculiformis* as well as a commercial vegetable tanning agent

Characteristics of leather	Vegetable tanning agents from Industrial Plantation Forest		Commercial vegetable tanning agents		The Indonesia National Standard
	A.mangium	*A.auriculiformis*	Mimosa	Quebraco	
Chemical analysis					
Tannins bound (%)	54.24	53.07	45.58	45,80	-
Degree of tanning (%)	188.62	184.55	167.37	162.26	Min. 25
Wrinkle temperature (C^0)	74	80	82	82	Min 70
Tanned leather physics					
Tensile Strength (N/cm^2)	2103.57	2430.58	2396.76	1728,36	Min. 1000
Elongation (%)	38.26	39.60	26.02	28.92	Max. 70
Thickness(mm)	1.08	1.16	1.18	1.18	-

Levels of tannins bound goatskin tanned using tanning materials bark extract *of A. mangium* and *A. auriculiformis* shows the results are not much different view Figure 6. The concentration of the same tanning agents, tannin levels are bound to both bark extracts showed a higher yield compared to commercial tanning agents. This is due to differences in the chemical composition of the tanners used. Vegetable tanning agents have a chemical composition called astringency so that they can affect the quality of the tanned leather produced [34]. Astringency is the ratio between tannins and non-tannins where astringency is the main characteristic that determines the tanning properties of various types of tanning extracts [10]. The mechanism of tannins bound to the skin is the formation of a number of hydrogen bonds between the phenolic hydroxy groups of tannins with carbonyl groups and amine groups in skin collagen [17].

The degree of tanning is the percentage ratio between the tannins bound to the skin structure and the raw substance leather. According to the Indonesian National Standard (SNI 06-0994-1989-A), a high number of degrees of tanning shows that perfect leather including her physical properties. When compared with standard tanned leather, the degree of tanning of leather with bark extract met the standard.

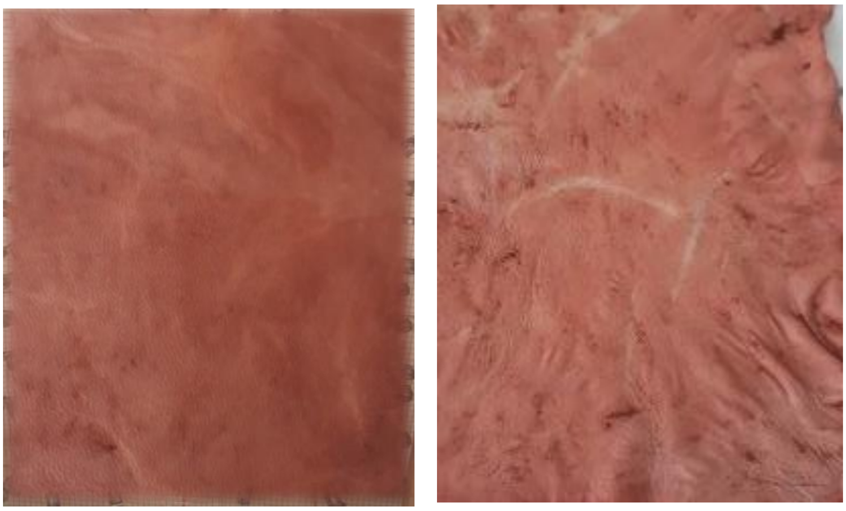
Wrinkle temperature is a temperature that indicates that the skin has been tanned where the molecular structure of collagen has stabilized [35]. The wrinkle temperature of leather using A.auriculiformis bark extract showed a higher value than the wrinkle temperature of *A. mangium* extract. The resulting wrinkle temperature shows a lower value than that of vegetable tanners such as mimosa and quebracho. However, when compared with the SNI standard, the value already meets the standard [36]. In vegetable tanning agents, the tannin molecules form several hydrogen bonds with collagen and create a tanning matrix thereby providing thermal stability to the tanned leather [6]. The water content in the collagen molecule affects the high and low wrinkle temperature, the high-water content in leather causes a low wrinkle temperature and vice versa.

The chemical composition of raw skin will have an influence on physical strength, including chemical bonds and the content of collagen protein content of the goatskin Globular protein content affects the stiffness properties, both on dry and fat content for leather. Water and fat content affect the level of skin elasticity that has been tanned. The physical strength of the skin is determined by the structure of the tissue and the chemical composition that composes the skin, including fibril diameter, fiber diameter, thickness and thinness of the fiber bundle, the angle of braiding, and thickness or thinness of the skin [37]. The results of the physical strength test of the leather aim to evaluate the quality of tanned leather products. [38] The physical properties of leather are defined in two versions, namely physical strength, and tissue structure. These two traits have a very close relationship. In general, physical properties are defined as the leather resistance to external influences, including mechanical influences, humidity, and external temperature [39]. Another factor affecting the tensile strength of the leather is its thickness.

Measurement of the physical properties of leather consists of tensile strength and elongation at break referring to ISO 3376: 2011 concerning the measurement of physical properties of leather. The tensile strength of the leather is the maximum stress that the leather can withstand when stretched or pulled before the leather breaks. The results of the tensile strength measurement showed results that were not much different. when compared with Indonesian National Standard, the quality of leather has met the standard. The tensile strength of leather is influenced by the structure of the collagen network that makes up the leather [39]. The structure of the leather forming the woven determines the level of tensile strength, at a webbing angle of less than 45°, the tanned goatskin has high tensile strength and vice versa if the angle formed by the collagen fibers is more than 45°, the resulting tensile strength is lower [40]. Tensile strength is very important in the leather product industry, tensile strength that does not meet the standard causes leather to break or crack easily.

Elongation at break is the increase in length of the leather when it is pulled until it breaks. The elongation value of tanned leather produced using *A. auriculiformis* and *A. mangium* tanning agent has met the SNI standard. The resulting leather is rather stiff [41]. Vegetable tanners cause the leather to become stiff, dense, and plump. Tanned leather can be softened as in chrome tanning by using oil. The fatliquoring process in the tanning process is carried out so that the tanned skin becomes weak [19]. The degree of tanning is an indicator of the amount of tannin that enters or bounds to the skin so that it becomes limp or flexible even when dry.

Thickness tanned leather produced largely influenced by skin raw materials crude that is used [42]. The thickness of goatskin ranges from 0.85-1.57 mm. The thickness of the tanned skin produced is also related to the amount of tannin that reacts with the skin causing the thickness of the skin to also increase [43]. The use of vegetable tanners can increase the tensile strength and increase the thickness of the leather due to the chemical bonding of the tanning agent's substance, resulting in thicker and fuller leather. Figure 6 shows a picture of tanned leather with vegetable tanning agents extracts of bark *A. auriculiformis* and *A. mangium*.



a b

Figure 6. The leather whit vegetable tanning agents bark extract: a. *A. mangium* ; b. *A. auriculiformis*

4. Conclusion

Forest production from industrial forest plantations in Indonesia in 2020 was 60.58 million m^3. This wood production tends to be stable to meet industrial needs. Of the total production, 52.63% were acacia plants. Where the bark as a byproduct produced as many as 4.8 million m^3 of wood utilization. The bark of *A. mangium* and *A. auriculiformis* that have not been used optimally can be used as a source of tannins for vegetable tanning agents to replace imported commercial tanning agents. The yield of extraction using water from the bark of *A. mangium* and *A. auriculiformis* was 23.53% and 25.96%, respectively. The results of the extractive analysis showed that this Acacia plant contains tannins. The leather produced using *A. mangium* and *A. auriculiformis* bark extracts produced leather that met Indonesian national standards.

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