THE INTERNATIONAL JOURNAL OF SCIENCE & TECHNOLEDGE

Brazilin Extraction from Secang Wood by Maceration Methods and Application for Leather Dyeing

Entien Darmawati
Lecturer, Polytechnic ATK (Academy of Leather Technology), Yogyakarta, Indonesia
Dr. Ir. Umar Santoso
Professor, Gadjah Mada University, Yogyakarta, Indonesia
Dr. Sudarmadji
Professor, Gadjah Mada University, Yogyakarta, Indonesia

Abstract:
The research aims to get brazilin pigment from secang wood (Caesalpinia sappan L) by extraction maceration methods and dyeing applications on leather (snake crust). The materials used secang wood (dried and fermented treatment), crust snake leather, and aquadest solvent. The research has been done in 3 (three) phase: (1) The extraction by the maceration method using a water solvent with variations ratio (material:solvent) w/v, temperature (22-25^{o}C, 40-60^{o}C and 70-100^{o}C), extraction duration (6,12, 24,30 and 36 hours), On the characteristic of brazilin pigments from color density and yields, (2) The application of brazilin pigment on snake crust leather by dyeing methods and (3) the color fastness test. The results shows that the extracted of brazilin pigmen the optimum yield of 4.72%, the color density 4.81^{o}Be (fermented secang) and optimum yield of 3.89%, the color density 3.78^{o}Be (dried secang), at temperature (70-100^{o}C), durations 30 hours. The results shows dyeing the absorption optimum optimum the color fastness test is 5-4 gray scale means good, the color violed-red (fermented secang), and optimum color fastness test is 4-3 gray scale means fair, the color red-violed (dried secang) at the dyeing formula 15,0% pigmen brazilin concentration, Rpm 12, pH 4,5-5,5. It can be concluded that brazilin pigment from secang wood, can be used as alternative materias dyes in the leather industry.

Keywords: *Extraction, Maceration, Brazilin, dyeing, Snake crust leather*
Category: (Animal Products and Technology)

1. Introduction

The development of leather industry has progressed rapidly both on production and quality. The resulting leather products use more synthetic dyes. Synthetic color is easily obtained from imported materials, but the price is relatively higher, the use of synthetic dyes is very dangerous for the environment, because in it contained carcinogenic properties that allegedly can cause health problems and can pollute the environment one way to overcome the problem is by substitution of synthetic dyes with natural dyes is a substance that is environmentally friendly, can be produced domestically,and relatively less toxic. (Kurniati, N. et al., 2012). The use of natural color has many weaknesses, among others, the manufacturing process requires a long time, not durable tend to fade easily, and the use of relatively large raw materials. However, Many things become doubts when continuing to use synthetic coloring materials because synthetic dye waste endangers human health and indirectly poison and pollute the environment. (Rina, O.et al. 2012).

One of the color-producing plants is Wood secang (*Caesalpinia sappan* L) containing pigment, tannin, brazilline, tannic acid, resin, resorcin,brazillinesappanin, and gallic acid (Pitojo, S, and Zumiati, 2009). Of the most interesting component is the colorsubstance. Wood secang produces a red pigment named brazilin/ brazilein. This pigment has a sharp and brightred color at neutral pH (pH=6-7) and shifts toward purplish red with increasing pH. At low pH (pH = 2-5) brazillin has a yellow color (Adawiyah and Indriati, 2003). According to research conducted by Oktaf Rina (2013), active wood compound secang identified in ethanol extract, brazillin extraction was done by maceration method using 96% ethanol solvent for 3 × 24 hours at room temperature. The concentration process is carried out with rotary evaporator equipment at 80ºC. Based on some previous research, This study aims to determine the optimum conditions of extraction by the maceration method of brazillin / brazilein dyes with variable ratio (material and solvent), extraction temperature, secang type and its aplication on tanned skin (crust reptile). As a substitute for synthetic dyes in leather and leather products industry has never been done.

2. Materials and Methods

2.1. Materials and Equipments

The material used in this research is wood secang from Dlingo, Bantul, Yogyakarta, solvent aquades and crust snake leather. Equipment used include: a set of extraction tools, mixing motors, mercury stirrers, analytical scales, heater, UV-Vis Spectrometer, pH meter, glass tools (glaze buckle, measuring cylinder, test tube, and erlenmeyer), Baumeter tool. Rotary drum with diameter 50 cm and equipped with temperature and rpm setting; Fastness testing using Crochmeter tool, Gray Scale standard, and Staining Scale Standard to assess color staining.

2.2. Research Methods

The experimental research was conducted in the laboratory with 3 (three) phase: (1) Extraction of natural dye of secang wood (*Caesalpinia sappan* L) by maceration method, with variation (a) type of secang (white, yellow and red) by treatmen dried and fermented,Ratio(material:solvent)w/ v, Temperature(22-25^{o}C; 40-70^{o}C; 70-100^{o}C), Duration (6,12,18,24,30 and 36 hours). (2) Application of brazilin dye on snake crust leather, by Through Dyeing and Sandwich Dyeing methods for brazilin dye application on crust snake leather. (3) Color fastness test using Crochmeter to perform wet/dry scouring and gray scale standard to assess color change of color fastness test, And the staining scale standard for assessing the color staining of white fabrics used in color fastness testing. Data were analyzed with 4-way ANOVA.

3. Results and Discussion

3.1. Extraction of Natural Dye of Secang Wood by Maceration Method

This research uses white, yellow and red secang wood, chip size of (1x1) cm, the size of extracted secang materials, the smaller size, the larger surface of the secang materials. So the process will be faster. There are two treatments: (1) Sun drying treatment on white, yellow and red secang wood, (2) Fermented treatment on white, yellow and red secang wood.

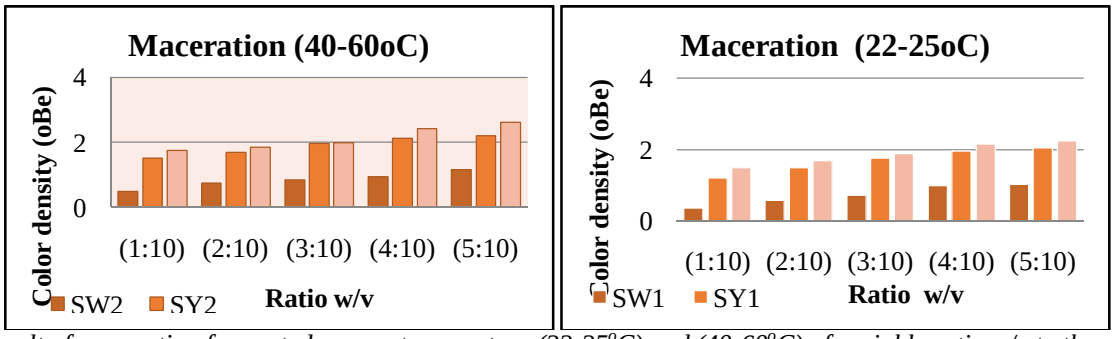

Figure 1: Result of maceration fermented secang temperature (22-25^{o}C) and (40-60^{o}C) of variable ratio w/v to the color density.

Fig. 1, shows that the extraction method of maseration time 24 hours,temperature (22-25^{o}C), color optimum concentration 2,24^{o}Be, ratio (5:20) w / v, pH 2.5-3,0, pigment color (orange-yellow),), in the treatment of red color (SR1), the optimum concentration of density of 2.05, ratio (5:20) w / v, pH 2.0-2,5,color pigment (yellow-orange) on yellow color treatment and the lowest color density obtained 1.02^{o}Be (yellow),pH, ratio (5:20) w / v, pH <2.0, yellow pigment color. The optimum concentration of temperature color (40-60^{o}C) of the 24-hour maseration time ratio (5:20) w / v was 2.62^{o}Be (red-violet), pH 5.0-5.5 on red color (SR2) , and yellow color (SY2) was obtained 2.22^{o}Be (orange-red), pH 3,5- 4,0 and the lowest white color (SW2) density was 1.16^{o}Be (yellow), pH 2.5 -3.0.

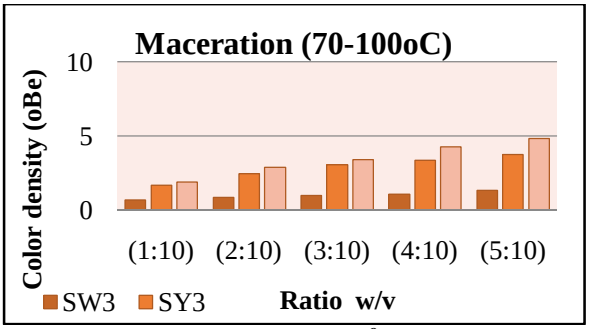

Figure 2: Result of maceration fermented secang (70-100^{o}C) of variable ratiow/v to the color density

Fig 2, the optimum concentration of color density at temperature (70-100^{o}C) of maceration time 24 hours at ratio (5:20) w / v obtained 4.81^{o}Be (violet-red) on red color (SR2), and yellow color (SY2) obtained 3.74^{o}Be (red-violet) and the lowest secang white (SW2) Density is obtained 1,32^{o}Be (yellow). this difference is due to the greater the ratio of solvent to the extracted solid material the greater

the yield, the more extracted the extracted material will contact more frequently with the large amount of solvent than the fewer solvent.

Duration (Hour)	Maceration with variable duration (hour) to the yield								
	SW1	SY1	SR1	SW2	SY2	SR2	SW3	SY3	SR3
6	1.03	1.36	1.55	1.13	1.45	1,93	1.25	1.78	1.92
12	1.24	1.53	1.89	1.35	1.68	2.06	1.59	1.94	2.93
18	1.38	1.69	2.19	1.49	1.75	2.43	1.68	2.73	4.13
24	1.59	1.93	2.35	1.72	2.05	2.87	1.85	3.49	4,55
30	1.70	2.09	2.47	1.82	2.18	3.02	**1.96**	**3.89**	**4,72**
36	1.64	2.12	2.69	1.99	2.34	3.25	1.78	3.63	4.58

Table 1: Maceration fermented secang wood variable duration (hour) to the yield (%)
Description: Maceration temperature (22-25^{o}C), secang wood (SW1=white;SY1=yellow;SR1=Red), temperatur (40-60^{o}C)secang wood SW2;SY2;SR2; and temperatur (70-100^{o}C) secang wood SW3; SY3;SR3)

Table 1, shows that the dyestuff extracted from wood of fermented secang using aquades solvent by maseration method at immersion temperature (70-100^{o}C), ratio (5:10) w/v, 30 hours, optimal yield of 4.72%, Obtained fermented secang wood the color red (SR3), yellow secang wood optimal yield of 3.89%, and white secang wood is not efektif with the yield of 1.96%.

Duration (Hour)	Maceration with variable duration (hour) to the yield								
	SW1	SY1	SR1	SW2	SY2	SR2	SW3	SY3	SR3
6	0.69	1.22	1.42	0.89	1.35	1,84	1.05	1.58	1.82
12	0.84	1.37	1.66	1.08	1.44	2.00	1.13	1.74	2.63
18	1.08	1.45	1.78	1.23	1.59	2.26	1.23	2.53	3.48
24	1.29	1.80	1.93	1.35	1.89	2.56	1.47	3.52	3,69
30	1.40	1.94	2.21	1.57	2.16	2.87	1.56	**3.68**	**3,89**
36	1.52	2.04	2.33	1.72	2.15	3.06	**1.69**	3.64	3.60

Table 2: Maceration dried secang wood variable duration (hour) to the yield (%)

Table 2, shows that the dyestuff extracted from wood of dried secang using aquades solvent by maceration method at immersion temperature (70-100^{o}C), ratio (5:10) w/v, 30 hours, optimum yield of 3.89%, Obtained fermented secang wood the color red (SR3), yellow dried secang wood optimal yield of 3.68%, and white secang wood is not efektif with the yield of 1.69%. The result of maceration fermented and dried secang wood of time variable to the yield, the difference caused by the longer extraction time will give bigger result, because the contact between the solvent and the extracted material is also longer so it will cause the solvent to be enriched by the solute.

3.2. The Application Brazilin Dyes on Snake Crust Leather with Dyeing Methods

The goal of staining on the tanned skin is: (1) beautify the appearance of tanned skin by reinforcing the color, polishing, smoothing the appearance of the tattoo and covering the defects / colors of the uneven base paint, (2) Protect the skin rajah from damage due to friction, heat, rain and rays sun.In this study using natural dyes to get the color looks original like the animal skin is still alive. The result of application of brazillant dye on snake crust with method of Through Dyeing and Sandwich Dyeing methods with the formula that is:(1) brazilant dye concentration 10-15%, dyeing temperature 70oC, moderate ideal pH 4.0-4.5, aquadest 200% from wet skin's wet skin weight, 1-1.5 hour dyeing time, and variation of rotary drum velocity / Rpm (4; 8; 12; 16 and 20).The staining process with brazillar dye is firstly used as a paste, and is slowly inserted in experimental drums that have crusted snake skin and warm water, stirring and anchoring on the experimental drum for 60-90 minutes. The drum rotation is observed until the liquid becomes clear, the final pH is 3.8.

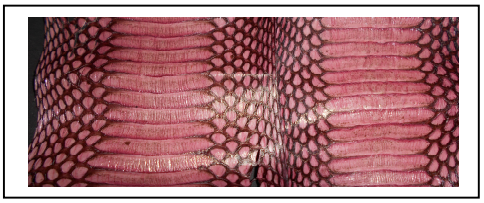

Figure 3: Brazilin application results on the snake crust leather (Fermented secang wood)

.

Fig 3, the result of the application of acidic / acid dyes brazillin (fermented treatmen red secang wood) having anionic group will bindor combining its ionic charge with cationic amino acid groups of skin and providing a violet-red color to the skin of snake crust leather.

R p m	Variable kecepatan drum (Rpm) terhadap % serapan								
	SW1	SY1	SR1	SW2	SY2	SR2	SW3	SY3	SR3
4	1.9	22,9	49.4	6.0	33.5	67,8	10.2	39.8	78.5
8	3.8	31.5	56.2	8.3	38.4	76.0	13.8	49.4	82.3
12	4.0	38.4	60.8	9.6	59.5	81.6	18.4	65.3	**85.7**
16	3.9	48,4	59.3	14.5	65.9	80.1	22.4	**78.2**	83.8
20	3.8	51.9	58,9	19.7	70.6	78.8	**24,0**	75.6	82.6

Table 3: Result of application of dye (fermented secang wood) variation of Rpm to % uptake of color on snake crust leather

Table 3 shows that brizilin on crust leather of snake fermentation variation obtained optimum% uptake 85,7%, rpm 12, dyeing time 1 hour, red-violet skin color, from red secang type wood. And obtained optimum% uptake of 78.2%, Rpm 16, 1.5 hours dyeing time, red-violet snake skin color, from yellow secang wood, and secang from white wood % uptake 24,0.

Figure 4: Brazilin application results on the snake crust leather

Fig 4, the result of brazilin dye application (dried treatmen red secang wood),which is acidic / Acid dyes having anionic groups will bind or combine their ionic chargewith cationic amino acid groups which give a red color.violet on the leather of snake crust leather.

R p m	Variable speed drum (Rpm) to% uptake								
	SW1	SY1	SR1	SW2	SY2	SR2	SW3	SY3	SR3
4	1.4	20,0	33.3	4.5	26.3	53,8	8.8	29.4	74.2
8	2.3	28.3	44.1	6.3	33.2	65.3	10.2	39.4	79.1
12	2.9	34.2	49.5	8.5	49.3	71.2	13.3	55.3	**81.5**
16	3.3	43,3	53.9	12.5	52.5	78.3	19.7	**69.4**	80.3
20	3.5	49.0	62,2	16.3	57.8	80.2	**22,2**	67.6	78.2

Table 4: Result of dried secang wood application variation Rpm to % uptake of color on snake crust leather

Table 4 shows that briziline on crust skin of snake fermentation variation obtained optimum% uptake 85,7%, rpm 12, dyeing time 1 hour, red-violet skin color, from red secang type wood. And obtained optimum% uptake of 78.2%, Rpm 16, 1.5 hours dyeing time, red-violet snake skin color, from yellow secang wood, and secang from white woods.

3.3. Fastness Test

The results of color fastness testing using Crochmeter to perform wet-dry and gray scale scrubbing to assess color change in color fastness tests, and Staining scale standard to assess color staining on white fabric used in color fastness testing. Grays scale 5-4; 4 (Good) for fermented secang type red secang wood and gray scale 4-3 (good enough) for dried secang type yellow secang wood.

4. Conclusion and Recommendation

4.1. Conclusion

4.1.1. Extraction of Brazillin Dyes

The result of Brazilin natural dye extraction from secang wood more efficiently is done by maceration methods, extract of brazilin wood pigment secang at temperature (70-100^{o}C), time 30 hours, obtained optimum yield 4.72% color density 4,81^{o}Be for fermented secang wood of the color red (SR3) and optimum yield 3,89%, color density 3,78^{o}Be, for dried secang wood of the color yellow, The result of brazilin identification of the presence of an OH-functional group and a double bond characteristic of the brazillin compound, pigmen dyes and white secang wood is not efektif with the yield and color density.

4.1.2. Application of Brazillin Dyes on Snake Crust Leather

The results of brazilin application on snake crust leather at Rpm 12, pH 4.5-5.5 with optimum condition of 10.0% concentration dyes secang, duration time dyeing 90 minutes, optimum % absorbsen dyes 82,6% fermented secang wood the color red, the direction of violet-red color, and optimum condition concentration of 15.0%, 90 minutes, the direction of violet-red color, and translucent color throughout the snake crust leather.

4.1.3. Absorption Test to Power Color Fastness

Color fastness test results. Grays scale 5-4 (Good) for fermented secang wood the color red on snake crust leather and gray scale 4-3 (good enough) for dried secang wood the color yellow on snake crust leather.

4.2. Recommendation

Utilizing environmentally friendly dyes from various color-producing plants for dyeing and finishing development in leather and leather products industries.

5. References

i. Adawiyah D.R dan Indriati, 2003. Color Stability of Natural pigmen from Secang woods (Caesalpinia sappan L) Proceeding of the 8th Asean food Conference, Hanoi 8-11 October 2003.
ii. Abrahart, EN, 2005. Dyes and Their intermediates, Secon Edition, Published Edward Arnold, London, pp. 145-144.
iii. Covington, D, Anthony, 2009. Tanning Chemistry, The Science of leather, second edition, Published The Royal Society of Chemistry, Cambridge. Pp. 269.370-383.
iv. Ding Zhiwen, 2008. Reuse Technology of Leather Wastes, Second Edition, China Leather & Footwear industry Research Institute, China. pp. 45-63
v. Darmawati,E,Triatmodjo,S and Roos,D. 2015. The Application of secang natural dye on sheep leather crust suede using ikat jumputan Method, Proceedings ISTAP, Faculty of Animal Science, Universitas GadjahMada, ISBN: 978-979-1215-26-8, pp 778- 784.
vi. Darmawati,E, Sudarmadji, Santoso,U.2016.The Application of Affal Parchment Leather a Souvenir Materials Using Secang Bark Dye Color Fastness, Proceedings ICTIS, ITP PRESS DOI 10.21063/ICTIS.2016.1015, pp 88-93.
vii. Darmawati, E, Santoso, U, Sudarmadji. 2017. The Application of natural dye from secang wood (Caesalpinia sappan L) on crust cattle leather by dyeing methods using emboss techniques. Proceedings ICBSH, Gadjah Mada University Press, ISBN: 978-602-386-235-1, pp 31-39
viii. Kurniati, N. dkk. 2012. Ekstraksi dan Uji Stabilitas Zat Warna Brazilein.Indonesian Journal of Chemical Science.
ix. Lemmens, RHMJ and N.Wulijarni, S, 2009. The Southeast Asian Vegetable Resources, Plant-plant pengahsil Dyesand Tannin, cetakan1, Bogor, PT. Balai Pustaka (Persero), Jakarta. P. 23-36
x. Mastuti, E, Kim, E.V, and Chistanti, M.E, 2012. Ekstrasi Senyawa Brazilin dari Kayu Secang (Caesalpinia sappan Linn) sebagai bahan baku alternatif untuk zat warna alami, Ekuilibrium, Vol. XI, (1); 1-5.
xi. Pitojo, Setojo, and Zumiati, 2009. Vegetable Food dyes, mold-5, Publisher Canisius. Yogyakarta, hal.10-16
xii. Rina, O. dkk. 2012. Efektivitas Ekstrak Kayu Secang (Caesalpinia sappan L) Sebagai Bahan Pengawet
xiii. Daging. Jurnal Penelitian Pertanian Terapan Universitas Bandar Lampung