Effect of Tween 80's Emulsifier Concentration with Spontaneous Diffusion Method on Stability Solution Tegeran's Nanoemulsion Natural Dyes

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Abstract:
Batik is an integral heritage of the Indonesian culture. With a variety of beauty, style and quality, natural colors and patterns of interest makes batik a very popular traditional cloth. Indonesia as one of the tropical country has great potential to produce healthy batik and friendly to the environment by utilizing various resources that available in nature. Efforts to produce environmentally-friendly batik can be done using colors derived from extracts of plant that contain flavonoide with attractive colors. Natural dyes for textile materials are generally obtained from the extracts of various parts of plant one of its is wood. Formulation of colouring material in an emulsion system that is ready to use is an attractive approach to encourage the use of natural colourant. In this research, colouring material was extracted using an organic solvent and emulsified in water with the aid of a surfactant. This research aimed to identify the characteristics of emulsion of tegeran wood extract. The concentration of mixture of tegeran wood extract and surfactant was varied (5, 10, 15 and 20% and the characterization included microscopic structures, colour intensity and viscosity. The research showed that viscosity increased with the increase in concentration of tegeran wood extract (ranged from 17,33 – 28,67 cp). Tegeran dyes emulsion with 3% tawas addition, analysis its size used to particle size analyzer for 5, 10, 15, 20, 25, and 30% concentration resulted 1,55, 2,2, 2,05, 2,6, 2,4 and 2,65 nano meter. Stability tend to decrease with the decrease in extract concentration, while intensity increase with the rise in extract concentration.

Key words: *batik, tegeran, spontaneous diffusion, natural dyes, nanoemulsion*

1. Introduction

Batik is an integral heritage of the Indonesian culture. With a variety of beauty, style and quality, natural colors and patterns of interest makes batik a very popular traditional cloth. Indonesia as one of the tropical country have great potential to produce healthy batik and friendly to the environment by utilizing various resources that available in nature. Efforts to produce environmentally-friendly batik can be done using colors derived from extracts of plant that contain flavonoide with attractive colors. 1856 [1] resulted that syntetic dyes in textil, both of native and syntetic fiber.

Syntetic dyes has disadvantage which caused of environment polution such as sulfuric acid, chloride acid, chrom, cupprum, zinc and costic. This compound as sourced of several disease such cancer and the others [2]. One ways to solve it throughout process production natural dyes. Natural dyes still has barrier to be advanced develop it such as not practice (needed deep it more than 10 replicates) and the colour not bright like syntetic dyes. This research objected to explore tegeran wood (*Maclura cochinchinensis*) as natural dyes for batik cloth.

To minimize the complicated process production of colouring use of natural dyes which would be implemented in advanced batik cloth, formulation needed to use it. Nano emulsion is one of great chance to solve it. Nano technology is defined as plan, process, and also implement its structure or amterials in nanometer scale. Nano emulsion is one of its products hopefully could be advanced implement it in the natural dyes from tegeran to the batik cloth.

2. Materials and Method

Tools which were used in this reseach included hammer mill, primisima cloth, whatmen paper, rotary evaporator, balanced, refractometer, glassware, pippette, magnetic stirrer, viscometer, chromameter, microscope, and PSA (Particle Size Analyzer). The raw materials included tegeran wood, glycerol, tween 80, tawas, methanol and aquades.

This research was divided into two steps pre and main research. In the pre research: tegeran woods mashed into 20 mesh, added methanol 1:4 composition, dept it during 24 hour, filetered with primisima cloth and whatman paper, and evaporated used to 50C during 2 hour. Steps of natural colourant extraction process as follow in the figure 1 below.

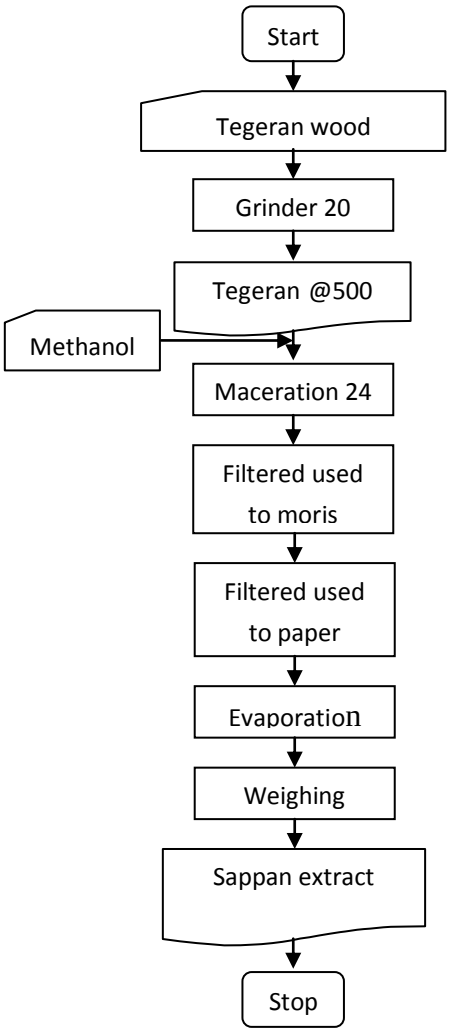

Figure 1. Tegeran Wood Extraction and Emulsion Steps Process Production

The sappan extraction adjust in the 20 brix and then arrange in four the treatment of the research 5, 10, 15 and 20% based concentration as shown in Table 1 below.

Table 1. Factorial Design of Natural Colourant Emulsion

Concentration (c)	Based (ml)	Mixture (ml)	Water Based Mixture (ml)

0,05	50	2,5	47,5
0,1	50	5	45
0,15	50	7,5	42,5
0,2	50	10	40

For advanced natural emulsion processed used to (1, 3 and 5 %) mordant addition as shown in the figure 2.

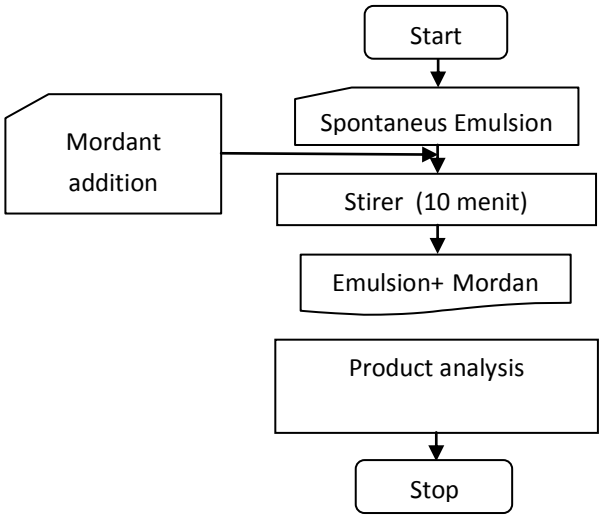

Figure 2. Tegeran Colourant Emulsion With Tawas Addition Steps Process Production

In the main research held into steps: spontaneous emulsion processed production used to magnetic stirrer with 33,3% and 66,6% concentration. In the emulsion solution added the tawas with concentration 1, 3 and 5%. The best result analysed included: colour, viscosity, and particle size used to PSA.

3. Result and Discussion

3.1 Tegeran Wood Extraction Processed

Natural dyes used to natural wood as raw materials has critical parameter reach optimum process production, which one of it yield. Based on the research, yield of tegeran dyes extraction with 20 brix concentration resulted as shown in Table 2.

Tabel 2. The yield of tegeran extraction

Steps Extraction	Yield of tegeran extraction (%)
1	8.78
2	7.25
Average	8.02

Extraction defined as separation process between solute and solution. Solution used to extraxtion process without another materials component [3]. In the extraction steps used hammer mill tool. To produce teregan wood with the same saphe used to hammer mill tool. The best solution which could be solute the extract component [4]. In this research used to methanol to extract the tegeran wood.

Based on [5], the best solute for tegeran extraction was methanol. In this research also used to methanol as solute to extract the tegeran wood. Methanol had polarity the same as tegeran wood materials which was identified as flavonoid component.

3.2 Main Research

A. Direct Analysis to The Tereran Wood Dyes

Suryani [6] identified emulsion system as stabilized system caused of each particle had characteristic koagulation and aggrgation process production so that made emulsion break. The streghten of layer between surface is important thing characteristics in stabilization emulsion. Direct parameter used to analysis the emulsion stabilisation as shown in the Figure 3.

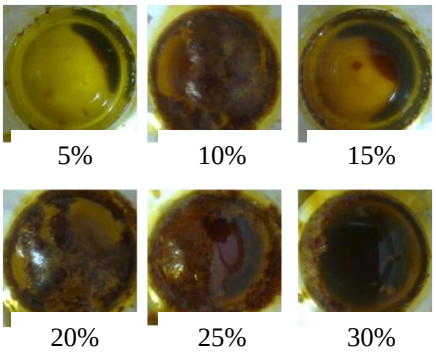

Figure 3. Tegeran emulsion solution with 33,33% Tween emulsifier with concentration 5, 10, 15, 20, 25 and 30%

Based on Figure 1, showed that increasing concentration resulted koagulation tegeran wood dyes. Combination concentration increased 66,7% to optimized its tegeran wood emulsion dyes. Mordant addition used to optimized its tegeran wood dyes to be advanced implemented in the cloth. Tegeran wood emulsion dyes could be shown as Figure 4.

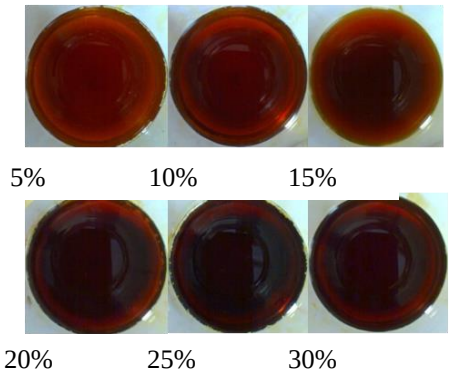

Figure 4. Tegeran emulsion dyes with 66,67% tween emulsifier and 5, 10, 15, 20, 25, and 30 % concentration addition

Tegeran emulsion dyes had brown reddish colour as shown in the Figure 4. The viscosity of the tegeran wood emulsion increased with increasing concentration. Water and oil made good emulsion in this concentration addition. Nguyen 201o said that emulsion influenced by particle sized and bias index between dispersant and indispersant media. In principle, based on direct analysis particle under 0,05 micron resulted transparant colour.

B. Viscosity of Tegeran Wood Emulsion Dyes

The viscositty influenced by its easily to flow as solution. Viscosity has correlation to the emulsion characteristics. The best emulsion for dyes has medium viscosity parameter process analysis [7]. The viscosity of tegeran wood emulsion dyes as shown in the Figure 5 and 6

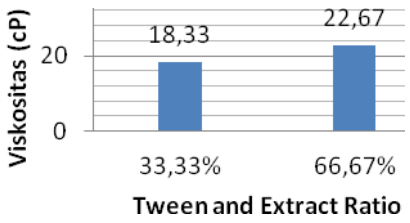

Figure 5. Viscosity analysis 33,33% and 66,67% tween and extract ratio

Viscosity of tegeran wood emulsion with 33,33% tween and extract ratio resulted 18,33 cP and 66,67 % tween and extract ratio resulted 22,67 cP. The increasing of oil/ water ration would be decreased of the dispersant fasa. Decreasing of dispersant fasa would be resulted with increasing its viscosity.

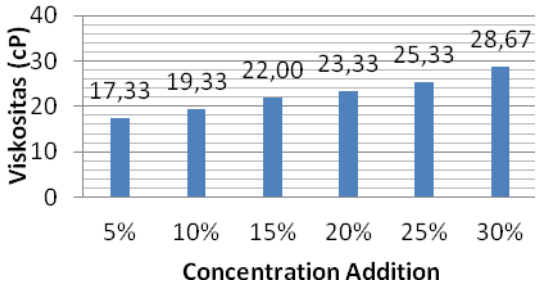

Figure 6. Viscosity value of tegeran wood emulsion in variation concentration addition

Viscosity value increased in 5, 10, 15, 20, 25, and 30% concentration addition resulted 17.33, 19.33, 22.00, 23.33, 25.33, and 28.67 cP. 30% concentration addition resulted the highest viscosity value. Viscosity value analysis also implemented in the tawa as mordant addition with concentration 1, 3 and 5%. Tawas addition resulted viscosity value as shown in the Figure 7. Tawas addition with concentratiion 1, 3, and 5% reseulted viscosity value 20,67, 22,33 and 25,00 cP. Kartika said that one parameter which was influenced to the viscosity value was molecule weight.

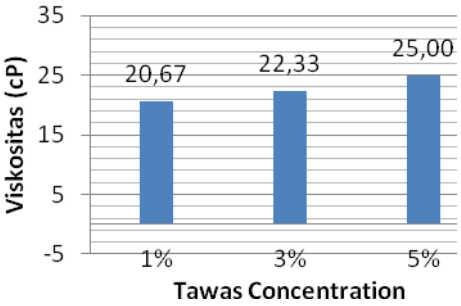

Figure 7. Viscosity value of tegeran wood emulsion with 1,3 and 5% tawas addition

C. L*, a*, b*, dan Hue of Tegeran Wood Emulsion Dyes

The generally to identify product colour used to calorimetry parameter. There are three dimension categhory L*, a* dan b* based on Minolta paremeter. L* difined as bright parmeter in 0 (black) until 100 (white), in vertical scale. While a* as axis horizontal red-pink (positive) and blue-green (negative) value. The secondary axis value represented in b* with yellow colour parameter (positive) and blue in (negative) value. Three dimension

value could be shown in the figure 3. L*, a* and b* value could be conversion in hue colour angle (h^o) and chroma (C*) analogue as intensity colour [8]. Colour analysis of emulsion with 33,33% and 66,67% concentration resulted as shown in the Table 3.

Table 3. L*, a*, b*, and Hue value of the tegeran wood emulsion with 33,33% and 66,67% concentration addition

Tween and Extract Ratio	L*	a*	b*	Hue
33.33%	31.72	9.51	18.32	62.60
66.67%	29.27	12.55	14.23	48.61

L*, a*, b*, and hue value resulted red-yellow colour in 33,33% and red colour in 66,67% identification in the tween –extract ratio concentration. Chroma intensity and Hue colour angle give specific value each treatment. That could be determinate that tegeran addition concentration 5, 10, 15, 20, 25, and 30% specifically contributed in each characteristic colour parameter as shown in the Table 4. Tawas addition prediction contributed to the colour intensity of tegeran colour emulsion in the Table 5.

Table 4. L*, a*, b*, and Hue in different concentration (5, 10, 15, 20, 25 and 30%)

Solution Concentration	L*	a*	b*	Hue
5%	46.77	15.24	41.63	69.93
10%	31.42	15.2	14.69	44.04
15%	29.46	10.23	11.92	49.39
20%	26.28	14.63	17.18	49.61
25%	29.71	11.45	14.07	50.89
30%	26.74	7.24	9.73	53.37
100%	23.10	3.23	4.73	55.70

From the table just could be conclude that difference parameter could be contribute to the specific value.

Table 5. L*, a*, b*, and Hue value with 1, 3 and 5% Tawas concentration addition

Tawas Concentration	L*	a*	b*	Hue
1%	29.11	9.45	9.86	46.24
3%	29.04	16.53	13.47	39.20
5%	27.26	12.37	10.43	40.16

The hue color value range 39,20 until 46,24 still shown in red color range based on Minolta parameter. 5% tawas addition also contributed to the specific hue angel value as shown in the Table 5. Adosko said 2006 said that colour produced by several of native colour combination.

D. Nano Emulsion Size Analysis

To analysis the nano emulsion size parameter, used to particle size analyzer tool. Before used it tool, globula size analysed due to polarized microscope and resulted as shown in the Figure 8.

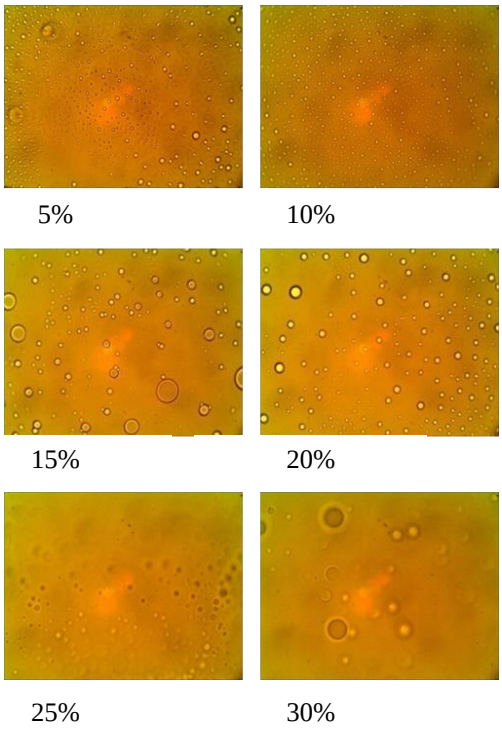

Figure 8. Globular tegeran wood emulsion with 5, 10, 15, 20, 25 and 30% concentration addition (100x size scale)

Side shape, size and distribution of tegeran wood emulsion also analysed related with its emulsion stabilision. The droplet known has circle with shape and size uniform distribution. Advanced analized related with its droplet size analysis, used to particle size analysis resulted as shown in the Table 6 below.

Table 6. Droplet size analysis with 3% tawas addition

Concentration (%)	Acerage Particle Size (nm)
5	1.55
10	2.2
15	2.05
20	2.6
25	2.4
30	2.65

In the Table 6 showed that average size with 5, 10, 15, 20, 25, 30 % concentration addition resulted size globuler 1.55, 2.2, 2.05, 2.6, 2.4, and 2.65 nm. The emulsion stability depend on its size particle analyzer and distribution. Droplet globular analysis due to used Particle Size Analyzer. The average measured by sauter mean diameter (SMD: d32). Sauter mean diameter defined as ball diameter analysis with volume / area surface ratio analysis.

4. Conclusion

The yield of tegeran dyes extraction with 20 brix was 8,02%. Tegeran dyes emulsion with tween 80 concentration 66,67% has characteristic more stable than 33,33% concentration. For advanced research, tween 80 concentration 66,67% formulation has added 1, 3, and 5% tawas. The viscosity value of 66,67% tween and 5, 10, 15, 20, 25, 30% solution resulted 17,33, 19,33, 22,00, 23,33, 25,33 28,67 cP. The tawas addition with concentration 1, 3, 5% had viscosity 20,67, 22,33 and 25,00 cP. Increasing solution concentration and tawas addition had positive correlation with viscosity value. Based on colour analysis, tegeran dyes solution had colour range between yellow until reddish

yellow. Tegeran dyes emulsion with 3% tawas addition, analysis its size used to particle size analyzer for 5, 10, 15, 20, 25, and 30% concentration resulted 1,55, 2,2, 2,05, 2,6, 2,4 and 2,65 nano meter.

References

[1] Djufri, R, A. Koesnarno, A. Salihima, A. Lunis. 1996. *Teknologi Pengelantangan, Pencelupan dan Pengecapan.* Institut Teknologi Testil, Bandung.
[2] Cahyadi, Wisnu. 2006. *Bahan Pengawet Makanan*. Bumi Aksara, Jakarta.
[3] Pasto, D., Johnson, C., Miller, M. 1992. *Experiments and Techniquest in Organic Chemistry.* New Jersey : Prentice Hall Inc.
[4] Kurnia, Rizky. 2010. Ekstraksi dengan Pelarut.http://lordbroken.wordpress.com. [20 Juni 2011].
[5] Hernani, D. Sumangat, Risfaheri, S. Yuliani, T. Hidayat, H. Herawati, dan F. Kurniawan. 2010. Ekstraksi dan Formulasi Bahan Pewarna Alami untuk Batik dengan Stabilitas Mendekati Pewarna Sintetis. Laporan Akhir Balai Besar Litbang Pasca Panen, Litbang Deptan, Bogor.
[6] Suryani A, Sailah I, Hambali E. 2000. *Teknologi Emulsi*. Bogor: Jurusan Teknologi Industri Pertanian, Fakultas Teknologi Pertanian, IPB.
[7] Riawan , Indra., E. Hartoyo., S. Rukmini. 2006. *Panduan Tekstil dan Evaluasi Tekstil*. Museum Tekstil DKI Jakarta.
[8] McGuire, R. G. 1992. Reporting of objective color measurements. Hortscience. 27 (12):1254-1255.