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Production of *Brachiaria mutica* as natural dyes powder for textile application: characterisation study

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Abstract. Innovation of synthetic dyes successfully eliminated the utilisation of natural materials as textile dyes. Simplicity as well as variety of colours strongly increased the application of synthetic dyes. Nevertheless, environmental concern has lately forced the use of natural dyes. To simplify the usage of natural dyes, production of natural dyes powder is inevitable. Investigation was carried out to examine the effects of solvent and filler types on the yield, chromaticity, flavonoid content, and water content of natural dyes powder. In this work, *Brachiaria mutica* was used as a sample source. It was crushed and grinded before extracted using mixture of methanol and water as solvents at various methanol concentration of 25%, 50%, 75%, and 100%. Extraction was carried out at a ratio of raw materials to solvent of 1:10. The filler of maltodextrine or gum arabic was added into dye extract before then converted into powder form by the aid of spray dryer. It was found that among the provided solvents, system using 50% methanol was considered to be optimal in resulting high yield as well as flavonoid content, while maltodextrine is chosen as providing better result than that of gum Arabic. Moreover, methanol concentration of 50% and 5% maltodextrine filler generated greyish yellow colour with the lowest water content of 0.9%.

1. Introduction

Natural dyes found as a result of the perseverance and the intelligence of our ancestors, can last for centuries. These dyes can be obtained from plant roots, parasitic insects, and even secretions of sea slugs. Natural dyes can be used for dyeing and painting. Dyes and pigments can be distinguished based on their solubility. Generally, dyes are organic compounds that can dissolve in solvents while pigments that are often used to paint are generally inorganic compounds or minerals that cannot dissolve in painting media (oil, water, etc.) and dispersed in the matrix.

To produce bright colours of red, blue, purple or yellow, high skills and experiences are required. Colour is obtained by applying chemical compound called chromophore or chromogen, which is something raising colour. As textile dye, chromophores must be absorbable by fibres thus provide fastness to washing characteristic. Colour can bind to the surface of the fibre. Mordanting agent contains metal ion to bind with textile fibres plays an important role in determining the obtained final colour. Alum, as a source of aluminium ions, is the most widely used mordant in the past. Other



mordant that are also widely used in the past are iron, copper and tin. *Indigofera tinctoria* is a kind of vessel dyes that is able to bind to fibres through oxidation reduction reactions with application of no mordanting agent.

Dyes and colouring processes have been known for a long time, as long as the finding of textiles. Nature is rich of plants that can produce various colours for dyeing process. Almost all organic matter can produce colour when it is boiled in to be dyeing solution, but only certain plants are able to produce colours that meet the requirements as colorants. Natural dyes are commonly taken from leaves, stems, fruits, flower petals, bark, and roots of the plants. The widely used natural blue colour substance is indigo, obtained from the leaves of *Indigofera tinctoria* plant. Natural dyes have complex chemical structures. Unlike synthetic dyes, natural dyes are not a single structure but a mixture of related chemical compounds. Based on the chemicals constituents, natural dyes are classified into [1] indigoid, anthraquinone, napthaquinone and benzoquinone, flavonoid, carotenoid, and tannin-based dyes.

However, due to the complex extraction process, the applications of natural dyes have been replaced by synthetic dyes that firstly found in 1856. The latter provides brighter colour and better fastness thus improve the efficiency of dyeing process. Rapid development of synthetic dyes increasingly discourages the utilisation of natural dyes. In Indonesia, synthetic dyes are among the major components required by batik home industries. The continue growing of batik export values shown by the achievement of export values of batik of about 58.46 million dollars in 2017. The number of batik industries in Indonesia was reported to be 136 thousand industries, in which 91.6% of them are located in Pekalongan.

Despite the economic contribution of batik, the generated wastewater threatened the surrounding environment. Utilisation of various dyes and chemical agents in the production process is a source of environmental pollution. The discharge of dyeing process wastewater contains high BOD, COD, and TSS. Due to high capita and operational costs of wastewater treatment, only about 0.6% of batik industries in Pekalongan developed their own installation of wastewater treatment [2]. It led to the direct disposal of harmful wastewater contained high concentration of BOD, COD, and TSS, beyond the allowable threshold [3, 4]. It is known that those high parameters are responsible in the harmful effects to water quality and organism.

Considering the harmful effects of synthetic dyes along with the chemical agents, many researchers have paid their attention in studies of natural dyes extraction [5-8]. Commercially, natural dyes have been utilised by batik home industries. Many customers interested with soft and unique colour generated by natural dyes. Nevertheless, the colour repeatability is still a major problem in natural dyes utilisation. This is due to there is no exact formula for commercial extraction of natural dyes. Natural dyes extraction needs relatively long time and complicated process thus minimising the overall process efficiency. Moreover, natural dyes solution has to be prepared right before utilisation. Distribution of natural dyes solution is almost impossible. Thereby, investigation of natural dyes powder for textile application is inevitable. Previous studies on production of natural dyes powder have been done [7, 9-11]. However, any report in overall characterisation of natural dyes powder is not available. This article reported rendement, chromaticity, and water content of natural dyes powder as the effects of solvent and filler types.

Few researches in obtaining high quality natural dyes by optimising extraction methods, fabric surface modification, as well as dyeing methods have been done [10, 12, 13]. In general, despite the high extraction efficiency and the high colour fastness, the colour reproductivity was relatively low. This is due to natural dyes in form of liquid and gel are relatively unstable. The development of novel dyeing methods and standardisation of natural dyes products offer effective and environmental friendly natural dyes. Conversion of liquid of gel natural dyes extract to be powder is believed to be able to solve colour consistency.

2. Methods

Spray drying is the most common method used to convert dye extracts into powder form in a simple and inexpensive way. The extract is sprayed into the storage tank through a fine spray nozzle. Contact that occurs between the fine grains and hot air that blows into the tank will evaporate the solvent so that dry particles are obtained at the bottom of the tank. This method can be applied to dyestuff molecules which are stable to heat because the dry powder of the dyes will be directly related to hot air [1, 7, 14-16].

2.1. Materials

Brachiaria mutica was used as sample of natural dyes. Deionised water was used for all of the solutions preparation. Methanol was purchased from Merck. Commercially available maltodextrine and gum Arabic were used as fillers.

2.2. Procedure

Schematic diagram of natural dyes powder production was given in Kusumastuti et al. [9]. The surface colour and surface darkness/ lightness of natural dyes was measured using chromameter. Data output is in the form of L*, a*, and b* values. L* refers to levels of darkness/lightness between black and white, while a* and b* are chromaticity coordinate. The coordinate a* shows the balance between red/green, and b* between yellow/blue. Colour difference was measured using Chroma meter Konika Minolta CR-400.

2.2.1. Extraction of natural dyes. Prior to extraction process, raw materials were crushed using chopper and then dried to reduce water content. A grinder was used for further reduction of raw materials particle size. Investigation was carried out to examine the effects of solvent types (water and methanol), in which effects of methanol was studied at methanol concentration of 25%, 50%, 75%, and 100%. In each experiment, 400 g of *Brachiaria mutica* was extracted in the solvent in ratio of raw materials to solvent of 1:10.

2.2.2. Production of natural dyes powder. Dyes and solvents were separated and concentrated by heating in a storage tank to reach 1/3 of initial volume. After that, filler was added into the concentrated dyes in order to investigate its effect to the moisture content at the condition of without filler, with 5% maltodextrine filler, and with 5% gum Arabic filler. The dyes were then pushed by a compressor at a pressure of 4 bar to be dried in spray dryer. This process was carried out at a drying air temperature of 175oC and a drying air flow velocity of 3 m/s.

The water content and yield of the obtained natural dyes powder were determined to define process efficiency as well as physical characteristics of the natural dyes powder. Proximate analysis was carried out to test the water content, phytochemical analysis was done to examine flavonoid content, while colour retention was determined by ratio of sample absorbance before and after treatments.

3. Results and Discussion

3.1. Yield of natural dyes powder of *Brachiaria mutica*

Yield is a parameter showing the amount of dyestuff resulted from a certain amount of raw materials. The obtained pure extract was weighed to determine extract yield. The yield is indicated in %, the higher yield indicates more extract was produced. Previous studies showed that total yield of natural dyes powder resulted by ethanol as solvent was not significantly different than that of rendement of natural dyes powder generated by methanol as solvent, but it was significantly higher compared to that of water as solvent [9]. The selection of solvent is done by considering economic factors, taking into account that methanol price is cheaper than that of ethanol, that could minimise production costs. It is

therefore, methanol was chosen as the solvent for further research. The yield and flavonoid content of *Brachiaria mutica* dyes powder as solvent variations are shown in Table 1.

Table 1. Yield and flavonoid content of *Brachiaria mutica* dyes powder in variation of solvents

Treatment	Yield (%)	Flavonoid Content (mg/g)
B1	9.445	1.68
B2	12.508	4.42
B3	12.735	4.54
B4	11.269	2.15
B5	8.57	1.38

B1 = methanol 25%

B2 = methanol 50%

B3 = methanol 75%

B4 = methanol 100%

B5 = water

The average of total yield of *Brachiaria mutica* dyes powder colour in variations of methanol concentration of 25% to 100% was to 9.445% to 12.735%. Table 1 shows that the average total yield of *Brachiaria mutica* dyes powder differed at each methanol concentration. The highest average yield of *Brachiaria mutica* dyes powder was produced at system with methanol concentration of 75%. The amount was not significantly different from *Brachiaria mutica* dyes powder prepared from solvents with methanol concentration of 50%, but significantly different from the treatment of methanol concentration of 25% and 100%. The same condition was also obtained in Delvitasari's study [17] which examined the production of dyes powder from mahogany bark. The yield of *Brachiaria mutica* dyes powder generated by system with methanol concentration of 50% and 75% were higher than that of system with methanol concentration of 25% and 100%. This was because polyphenol compounds in flavonoids found in *Brachiaria mutica* have polar and nonpolar groups. The hydroxyl group on polyphenols has polar properties, while the phenol group is nonpolar [18]. In polar solvents, polar groups will dissolve, i.e. in water contained in organic solvents at low concentrations, while nonpolar groups will be extracted by organic solvents with semi-polar properties.

The average of flavonoids content in *Brachiaria mutica* natural dyes powder as effect of various methanol concentrations were in the interval of 1.68 mg/g to 4.54 mg/g. It was revealed that methanol concentration generated a significant difference of average flavonoid levels of *Brachiaria mutica* natural dyes powder. The highest value of flavonoid content was resulted at methanol concentration of 75%, although it was not significantly different to that of methanol concentration of 50%.

Yield level is a parameter that determines the success of an extraction process. In this study the high yield value was influenced by type of extraction method and type of solvent. Study of Jin et al. [19] confirmed the results, in variation of extraction method, it was found that the comparison of the total yield in the extraction of flavonoids from *Cajanus cajan* leaves, the use of reflux method generated a higher yield than the maceration method. This is because the reflux method involves heating the process, so that the screening of active compounds becomes more optimal.

Plants produce secondary metabolites as products of primary metabolism as well as part of plants defence mechanisms. Secondary metabolites produced by plants, for example are phytochemicals, such as alkaloids, tannins, and flavonoids. Metabolite compounds with various properties do not allow it to dissolve entirely in methanol, therefore, variations in composition between methanol and water are made. Chigayo et al. [20] found that methanol performed the highest extract yield of 52.9% in the extraction of phytochemicals, phytochemical screening and quantitative analysis of total phenols, flavonoids and antioxidant activity of *K. wilmsii* tubers. In addition, Harborne [21] recommended a combination of methanol-water solvent to isolate flavonoids.

Total content of flavonoid in *Brachiaria mutica* natural dyes powder extracted using methanol as solvent at the lowest concentration of 25% was 1.68%. It was because that concentration provided the most polar concentration compared to other solvent concentrations, therefore polar components such as carbohydrates were extracted and leading to the lowest total flavonoid content per sample weight. Proximate analysis revealed that the highest proximate component was carbohydrate, which was 44.37%. Moreover, *Brachiaria mutica* also contains water (17.09%), ash (5.01%), crude fiber (32.28%), each of which is polar with only 1.25% of nonpolar fat. Abarca-Vargas et al. [22] reached the maximum total phenolic content amount and the highest antioxidant potential by applying methanol as solvent. Extraction of *Brachiaria mutica* under methanol concentration of 75% gave the highest total yield and flavonoid content, but they were not significant compared to those resulted by system with methanol concentration of 50%. It is therefore, considering the economic value, further experiment was carried out using methanol concentration of 50%.

Effect of filler to yield of natural dyes powder of *Brachiaria mutica* was investigated at concentration of maltodextrine of 5%, gum arabic 5%, and without filler. Table 2 presents results of yield and content of flavonoid in variation of filler. Dubey [23] stated that extract could be encapsulated by active agent, in this research were maltodextrine and gum arabic. These active agents protect the core material from environmental influences. Encapsulation technique is very helpful in minimising rate of pigment degradation as well as increase shelf life [24]. Mechanically, maltodextrine covers the core material by forming a smooth surface ball [25]. At this stage maltodextrine covers flavonoid pigment, controlled by stirring. Moreover, maltodextrine react with water to form a colloidal liquid, upon heating, it has the ability as adhesive, clear colour, smelly, and non toxic.

Table 2. Yield and flavonoid content of *Brachiaria mutica* dyes powder in variation of filler

Treatment	Yield (%)	Flavonoid Content (mg/g)
D1	12.538	4.56
D2	7.375	3.14
D3	4.365	8.27

D1 = maltodextrine of 5%

D2 = gum arabic of 5%

D3 = without filler

Yield is a parameter shows the amount of dyes powder produced from a certain amount of *Brachiaria mutica*. Table 2 shows that there were yield increment of about 68-187% by using gum arabic and maltodextrine as fillers, respectively, compared to that of extraction without filler. The filler was added to increase the yield of final product. Moreover, the addition of filler material is very helpful in minimising quantity of dyes powder attached to the drying wall. The results showed that variations of filler type resulted in significant different of average value of *Brachiaria mutica* extract. There were significant different of extract yield by the application of various fillers, compared to control system, i.e. extraction without the addition of filler. Extraction system employed maltodextrine as filler generated the highest natural dyes powder. Utilisation of filler was intended to increase the total solids of dried materials. The branched maltodextrine molecules simplify the binding of hydroxyl groups in matodextrine to hydroxyl groups in the solvent. Very fine and hygroscopic dyes powder was generated by spray dryer. The hygroscopic natural dyes powder tends to easily absorb water from high air humidity environment, lead to the agglomeration of natural dyes powder. The increase of agglomerated natural dyes resulted in the reduction of final product yield. It was observed that *Brachiaria mutica* extract filled with gum arabic tends to produce wet end product. The incomplete dissolved core materials as well as the clumped gum arabic lumped in the spray dryer hose. In the end, the products tend to be wet, charred and attached to the tube wall. Similar condition was found in previous research [17, 26-29].

Flavonoid content is mainly determined by the addition of filler. Table 2 describes significant reduction of flavonoid content by the utilisation of filler. *Brachiaria mutica* extract powder produced without the application of filler contained flavonoid of about 8.27 mg/g. The addition of 5% of maltodextrine decreased flavonoid content to be 4.56 mg/g. The occurrence of clumping due to the use of gum arabic even decreased flavonoid content to be 3.14 mg/g. Degradation of flavonoid content by utilisation of filler could be minimised by addition of stabiliser, for example MgCO₃, as observed by some researchers [30, 31].

3.2. Characteristic of *Brachiaria mutica* dyes powder

Analysys of characterisation of *Brachiaria mutica* dyes powder is important to design form of product dosage and to be considered in process selection of production. Production of *Brachiaria mutica* dyes powder was done through extraction and drying processes under spray dryer. During the drying process, type of filler was varied using maltodextrine of 5%, gum arabic of 5%, and without filler. Utilisation of filler was aimed to produce non-sticky filtrate in the drying process. The process was done under inlet temperature of 120-130°C and outlet temperature of 75-80°C. The condition was determined by considering the boiling point of methanol in about 64.7°C thus expected that methanol was completely evaporated. Higher temperature resulted in darker natural dyes powder.

There are two widely used colour measurement methods, i.e. objective and subjective colour measurement methods. Colour can be analysed objectively with organoleptic physical instruments or subjectively with human senses. In this study, colour testing was carried out objectively with colour chromameters and quantified into L * a * b * notations, also known as CIELAB. In the CIELAB colour unit, L * indicates lightness, while a * and b * are chromaticity coordinates. The chromatic a* axis extends from green (-a*) to red (+a*), and the chromatic b* axis extends from blue (-b*) to yellow (+b*). While the lightness dimension, notated as L*, ranges from 0 (pure black) to 100 (diffuse white). In the intersection of a* and b* axes cross, at the L* value of 50, is pure, balanced, shows neutral gray.

Tabel 3. Chromaticity of *Brachiaria mutica* dyes powder in variation of filler

Treatment	L*	a*	b*	°Hue	Colour
D1	34.16	-11.54	36.27	52.58	Yellow
D2	35.52	-9.03	33.65	50.41	Grayish Yellow
D3	35.34	-10.12	34.88	51.78	Yellow

D1 = without filler

D2 = maltodextrine

D3 = gum arabic

Chromaticity of *Brachiaria mutica* dyes powder in variation of filler is given in Table 3. Higher lightness level L* indicates lighter colour of natural dyes. Value of 0 indicates black colour while value of 100 indicates white colour. The lightness level of *Brachiaria mutica* dyes powder was in the range of 34.16 to 35.52. The lightness level was significantly affected by types of filler. Addition of maltodextrine resulted in the lightest colour of *Brachiaria mutica* dyes powder, followed by natural dyes powder processed by the aid of gum arabic, while system without the utilisation of filler generated the darkest colour of natural dyes powder. The results of this study were in line with those obtained by Delvitasari [17] in the process of extraction and production of natural powder dyes from mahogany. It was found that the addition of fillers gave a significant effect on the lightness of mahogany bark dyes powder. Lighter natural dyes powder was generated by the system with addition of fillers.

Notation of a* and b* are chromaticity coordinates, in which +a* indicates red direction, while -a* indicates green direction. In average, natural dyes powder obtained in this research resulted in a* of -

9.03 to -11.54. Notation of b^* shows chromaticity of blue and yellow. Value of positive b^* ($+b^*$) from 0 to 60 and negative b^* ($-b^*$) from 0 to -60. In this research, average value of b^* were in the range of 33.65 to 36.27. In the colour coordinate, *Brachiaria mutica* natural dyes powder using maltodextrine as filler resulted in a^* of -9.03 and b^* of 33.65, resulted in grayish yellow colour. *Brachiaria mutica* natural dyes powder gave yellow colour at a^* of -10.12 and b^* of 34.88. Average value of a^* and b^* resulted by *Brachiaria mutica* natural dyes powder produced without filler had a^* of -11.54 and b^* of 36.27. Based on values of L^* , a^* and b^* , $^{\circ}$ Hue of *Brachiaria mutica* natural dyes powder could be determined. The $^{\circ}$ Hue in the range of 51° to 60° implied that at all treatments, *Brachiaria mutica* natural dyes powder generated yellow colour.

Another study on natural dyes characterisation was done based on solvent type. Investigation was carried out at methanol concentration of 25%, 50%, 75%, 100%, and water as solvent. Table 4 describes experiment results on the given variation.

Table 4. Chromaticity of *Brachiaria mutica* dyes powder in variation of solvent

Treatment	L^*	a^*	b^*	$^{\circ}$ Hue	Colour
B1	38.16	-7.54	31.27	51.58	Yellow
B2	35.57	-9.07	33.59	50.43	Grayish Yellow
B3	35.69	-9.12	33.88	50.68	Grayish Yellow
B4	36.01	-9.21	33.86	50.56	Yellow
B5	37.96	-7.21	31.67	51.72	Yellow

B1 = methanol of 25%

B2 = methanol of 50%

B3 = methanol of 75%

B4 = methanol of 100%

B5 = water

In the variation of the solvent, the brightness level of *Brachiaria mutica* natural dyes powder, expressed by L^* was in the range of 35.57 to 38.16. Table 4 revealed that variation of solvent determined colour direction and brightness of the dyes powder. Utilisation of water as a solvent raised a bright yellow colour of *Brachiaria mutica* dyes powder, while the addition of methanol to 25% gave no significant effect. Increasing concentration of methanol to 50% generated a grayish yellow colour. The optimal solvent for the extraction of *Brachiaria mutica* was 50% methanol, proven by the insignificant changes of natural dyes powder by the increase of methanol concentration to 75%. Further increase in the concentration of methanol to 100% actually resulted in brighter colour.

3.3. Water Content of *Brachiaria mutica* Dyes Powder

Measurement of water content of the dyes powder was carried out to determine the water content of the dyes powder available after the drying process. Water content is one of the important indicators that determine the quality of the product of natural dyes powder because it will affect the shelf life of the product. The effect of addition of filler type to the moisture content of *Brachiaria mutica* dyes powder was investigated under extraction system of *Brachiaria mutica* without fillers, with maltodextrine fillers, and with gum Arabic fillers. The results of the study can be seen in Table 5.

Table 5. Water content of *Brachiaria mutica* dyes powder in variation of filler

Treatment	Water Content (%)
D1	0.938
D2	1.375
D3	2.365

D1 = maltodextrine 5%

D2 = gum Arabic 5%

D3 = without filler

Table 5 shows that addition of maltodextrine as filler in the extraction of *Brachiaria mutica* could reduce the water content of dyes powder. Higher water content was found in the dyes powder processed in the system without addition of filler. This condition is in accordance with the finding of Fabra et al. [32] in which equilibrium water content of noni samples containing maltodextrine at a given water activity was lower than that of noni samples without maltodextrine. The water sorption capacity of the dyes decreased by the incorporation of maltodextrine because in nature, it becomes less hygroscopic. The addition of maltodextrine leads to the increase of total solids in the dried material, in which application of more filler gave further decrease of water content [33]. This is due to the ability of maltodextrine to bond free water thus more maltodextrine succeeded in decreasing water content as also found by Caliskan and Dirim [34]. The increase of maltodextrine gave significant effect on the water activity of the sumac extract powders. However, maltodextrine application induces the degradation of colour strength. Basically, maltodextrine is white in colour [35]. This affects the basic color of the *Brachiaria mutica* powder, in which increasing the lightness. Moreover, the ability of maltodextrine to form layers around the colour pigment significantly increased the lightness of the powder [36]. Increment of maltodextrine concentration resulted in a decrease in water content. Therefore, the addition of solid particles such as maltodextrine can accelerate equilibrium time of water content. The simple molecular structure of maltodextrine can easily remove bound and free water in the drying process. On the other hand, the addition of maltodextrine can reduce the water absorption capacity of the dyes powder because it is less hygroscopic.

Maltodextrine could increase the total solids of dried material thus decrease the amount of evaporated water. It is therefore, increasing maltodextrine concentration will reduce water content of the powder. This is also supported by the theory of Masters [37] in which higher dried total solids accelerate the evaporation resulted in lower water content of material. In addition, maltodextrine will form hydrogen bonds with water molecules in the extract. Water will be removed by drying process, ignites crystallisation. The higher maltodextrine concentration accelerates crystallisation and water evaporation, so that lowering water content of material.

Besides being influenced by the drying process, water content of the dyes powder is also greatly determined by the storage conditions. The high water content in the *Brachiaria mutica* natural dyes powder was due to the characteristics of dyes powder which is very easy to absorb water. This is due to the balance between the product humidity with the environment. Inappropriate storage process of the dyes powder could increase the product water content. The addition of gum Arabic produced dyes powder with higher water content due to the nature of the gum which can form a gel thus inhibits drying process. This is because the water trapped in the gel is difficult to evaporate that it formed a semi-wet powder [17, 27].

4. Conclusion

A research on the characterisation study of *Brachiaria mutica* dyes powder has been done. Under the variation of solvent, it was found that the highest yield of 12.735% and flavonoid content of 4.54 mg/g were resulted by system with solvent of methanol 75%. However, due to the insignificant achievement of the yield and flavonoid content of those of system with methanol 50%, further experiments were carried out using methanol 50%. In variation of filler, *Brachiaria mutica* dyes powder produced by the addition of maltodextrine generated the highest yield of 12.538% and flavonoid content of 4.56%. In term of colour, *Brachiaria mutica* dyes powder has yellow to grayish yellow. Addition of maltodextrine was able to minimise water content of *Brachiaria mutica* dyes powder to be 0.938%.

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