Production of natural dyes powder based on chemo-physical technology for textile application

by Dewi Selvia Fardhyanti

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Abstract. The application of natural dyes has been inseverable part of human life. Initially, natural dyes were the only colorants used for textile application. However, the invention of synthetic colorants at the end of the 19th century triggered a significant decrease in the wide and exclusive application of natural colorants. Research was conducted on various solvent type, temperature, and extraction time. Extraction temperature was varied at 70, 80, 90, 100, and 110oC, while extraction time was investigated at 60, 90, 120, 150, and 180 min. Volume ratio of dye material to solvent was 1:20. The dyestuff and solvent were separated by heating. Then dried using a spray dryer and the yield of the solid was weighed using balance. Investigation of effect of solvent type to total rendement found that methanol was the most suitable solvent for *Brachiaria mutica* extraction. The best condition for *Brachiaria mutica* extraction were temperature and time of 90°C and 180 minutes, respectively. Investigation on solution pH revealed the optimum pH of 12.

1. Introduction

Indonesian economy is highly supported by textile and textile product industry. Both domestic and international demands supplied by the sector contribute to large employment. In 2013, this sector employed about 1.55 million labours. Capacity of textile and product textile increased from about 1.77×10^6 tonne in 2000 to be 1.87×10^6 tonne in 2006 [1].

Many industries applied dyes and pigments in their processing. Annual production of dyes and pigments to supply textile, pharmaceutical, food, cosmetics, plastics, paint, ink, photographic and paper industries is estimated to be over 7×10^5 tons with over 10,000 types [2].

The application of synthetic textile dyes is indicated to be excessive. This is due to the purpose of meeting the required coloration of global consumption of textiles due to cheaper prices, wider ranges of bright shades, and considerably improved fastness properties in comparison to natural dyes. However, the production of synthetic dyes is dependent on petrochemical source and some of these dyes contain carcinogenic amines. The application of such dyes causes serious health hazards and

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influences negatively the eco-balance of nature. ~~A~~ higher demand is put towards the greener alternatives or agricultural residues. As a result, natural dyes are among the promising options for developing a greener textile dyeing process and such interest is reflected to the increased number of recent publications. Plant leaves are potential sources of natural dyes because of their easy availability and abundant nature.

Natural dyes are considered to be eco-friendly as these are obtained from renewable resources as compared to synthetic dyes which are derived from non-renewable petroleum resources. These are biodegradable and the residual vegetal matter left after extraction of dyes can be easily composted and used as fertilizer. They produce soft colours soothing to the eye which are in harmony with nature.

Over the past two decades, the use of natural dyes has increased again. Along with the increase of consumer awareness about environmentally friendly textile materials and the importance of environmental safeguards, natural dyes have been reused in colouring textile materials. Issues that synthetic dyes are carcinogens improve the demand of natural dyes. However, there are limitations to natural dyes, including the long and time-consuming process. In addition, the resulting colour is difficult to repeat, so that in large-scale colouring it is relatively difficult to obtain uniform colours.

A number of aspects have to be considered to re-introduce natural colorants into technical dyeing. There are some important aspects which also define experimental conditions used for this study, i.e. the content of dyestuff content in the plant material; it is therefore a consideration for extraction solvent must be carefully done. This is to avoid the release of huge amounts of chemically contaminated extracted plant material from the extraction step. Direct disposal of chemical should be prevented for costs and environmental reasons. Natural dyes production needs a huge supply of plant material, thus have to be handled during harvesting, storage and extraction. Variability of dyeing results could be minimised by standardisation of dyestuff, the plant materials of same quality must be chosen. Technically, application of natural colorants should be done with the available equipment in modern textile dye house thus evading big additional investment. Studies on natural dyes extraction with various parameters and operation conditions as well as plant materials have been done by some researchers [3-5].

This research is expected to offer clean technology and environmental-friendly textile dyeing system by minimizing the use of hazardous chemicals via application of natural dyes. The application of spray and vacuum drying methods in the production of natural dyes powder for textile application is of important to be investigated. This research will describe the optimal extraction condition to produce high quality natural dyes. This research contributes to the knowledge and technology. The success of this research will be helpful in the application of natural dyes. Apart of achieving clean environment, the utilisation of non-edible and un-utilised sources could enhance the economic value.

2. Materials and methods

2.1. Materials

Brachiaria mutica was used as sample of natural dyes. Deionised water was used for all of the solutions preparation. Ethanol and methanol were purchase from Merck.

2.2. Experimental apparatus

The extractor was designed for capacity of 2 L, made of stainless steel. It is equipped with heater band as heater, stirrer, dryer, detector, pressure and temperature controllers. Spray dryer was made of mild steel in 4 L capacity, equipped with low pressure air heater and temperature controller. In addition to work for dryer, spray dryer also acted as cyclone to separate water vapour and natural dyes powder.

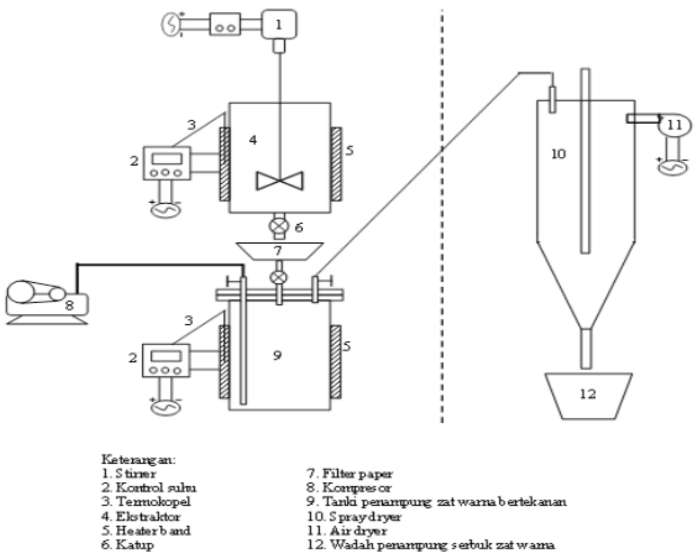

Figure 1. Schematic diagram of natural dyes powder production

2.3. Extraction of natural dyes

This stage was begun by reducing the size of raw materials using chopper and then dried to reduce water content. Furthermore, the particle size was reduced again using the grinder. Thereafter, the amount of the feedstock was mixed with ethanol, methanol, or water as a solvent for extraction in the extractor. Research was conducted on various solvent type, temperature, and extraction time. Extraction temperature was varied at 70, 80, 90, 100, and 110°C, while extraction time was investigated at 60, 90, 120, 150, and 180 min. Volume ratio of dye material to solvent was 1:20. The concentration of the dye before and after the extraction was measured using UV Vis Spectrophotometer to determine the efficiency of extraction.

2.4. Production of natural dyes powder

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After the extraction process, the solution and the dregs were separated using filter paper. Then the dyestuff and solvent were separated by heating. Then dried using a spray dryer and the yield of the solid was weighed using balance. This dye powder production was done at various temperature and fluid flow velocities. The water content and the dye powder gain before and after the process were measured to determine the process efficiency and physical characteristics of the dyestuff powder. Water content was measured using Karl Fischer Titrator.

3. Results and discussion

3.1. Effect of solvent type

Study on colouring matter of natural dyes extraction was done by varying types of solvent. In this stage, optimisation study of solvent for *Brachiaria mutica* extraction was carried out using water, methanol, and ethanol. Solvent determination was considered based on the highest rendement as well as tannin concentration. Figure 2 reveals effect of solvent to rendement of natural dyes powder.

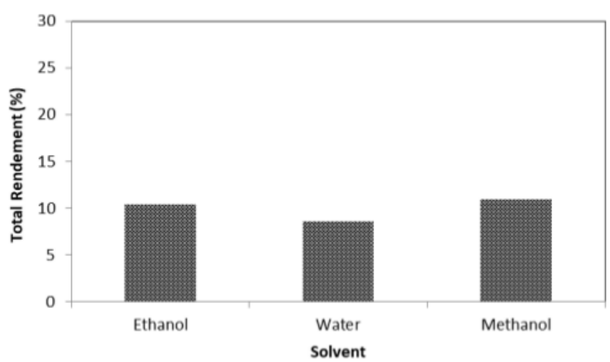

Figure 2. Effect of solvent type to total rendement

Average of total rendement of *Brachiaria mutica* dried extract at different solvent types was in the range of 8.57% to 10.96%. Total rendement of dried extract prepared with ethanol was insignificantly different to that of produced by deionised water. The lowest total rendement was given by system with deionised water as solvent. Delvitasari [6] obtained similar trend in her research. Total rendement of mahagony extract at various solvent types were 8.57%, 10.17%, and 10.56% for deionised water, methanol, and ethanol, respectively. Flavonoid and tannin, as colour-producing compounds in *Brachiaria mutica*, are polar compounds thus dissolve in polar solvents [7]. Polarity of *Brachiaria mutica* is possible closest to that of methanol thus system with methanol as solvent provided the highest total rendement.

3.2. Effect of extraction temperature

It is known that in extraction of natural dyes, temperature is of important. Higher temperature will increase extraction efficiency. However, high temperature also increases the risk of dyes degradation while low temperature limits extraction rate [8]. Higher temperature could result in colour change due to energy change that affect electron conjugation. In the end, colour stability would also decrease. Higher time and temperature stimulate compounds accumulation of colour degradation [9]. Effect of extraction temperature has been investigated; the results are shown in Figure 3. Effect of extraction temperature was studied at 70°-110°C.

Temperature change effects operation condition in spray dryer. Using initial gas temperature at 120°C, the obtained powder increased with the increase of feed temperature and retention time in spray dryer. The addition of retention time increased the contact time of hot air and *Brachiaria mutica* extract thus more powder was produced. Higher feed temperature also improving yield due to lower temperature gradient of feed and hot air. However, yield of powder decreased at feed temperature of 100°C. It could be driven by the vaporisation of volatile materials by high operation temperature. The best result was achieved by extraction temperature of 90°C and retention time of 180 minutes at total rendement of 12.51%. Similar result was obtained by Paryanto [10] in the extraction of *Bixa orellana* that achieved total rendement of 9.8% at extraction temperature and time of 90°C and 180 minutes, respectively.

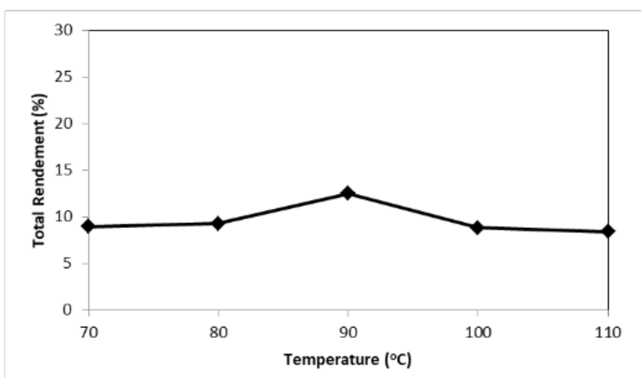

Figure 3. Effect of extraction temperature to total rendement

3.3. Effect of extraction time

Further study of extraction of *Brachiaria mutica* was carried out to effect of extraction time. In this stage, extraction time was varied at 60, 90, 120, 150, and 180 minutes. This step is of important; the best yield could be produced at optimal extraction time. Theoretically, higher yield would be obtained at longer extraction time. However, excessive lengthening of extraction time tends to degrade natural dyes. Moreover, evaporation potentially occured that resulted in the loss of solvent by longer extraction time [11].

Effect of extraction time to total rendement is described in Figure 4. The figure reveals the increment of total rendement by time lengthening. The highest total rendement was achieved ata extraction time of 180 minutes. Based on the previous research of Dent et al [11], extending extraction time by more than 3 hours was not recommended due to colour degradation reason. Longer extraction time is also not economically feasible. Extraction time of 180 minutes managed to obtain total rendement of 12.46%.

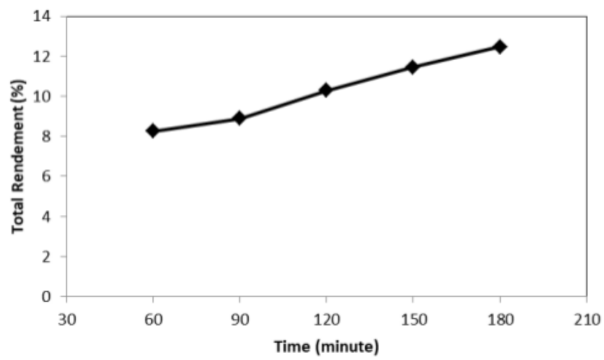

Figure 4. Effect of extraction time to total rendement

3.4. Effect of solution pH to total rendement

pH of extraction solution was varied at 2-12. Effect of solution pH to total rendement is revealed in Figure 5. The figure indicates the increment of yield by the increase if solution pH. Insignificant increase of yield was monitored in the increase of solution pH from 2 to 10. Steep increment of total rendement was seen in the increase of solution pH of 12. This result agreed the research of Shauki et al [12]. Their study of pomegranate extraction revealed the similar trend. Tannin compound in *Brachiaria mutica* is acidic in nature; it is therefore pH increment induced its leaching towards extraction medium. Neutralisation of tannin occurred due to the solution alkalinity thus releasing more colouring compounds. The breaking up of plant cell membranes, increased by solution pH increment, highly enhances chromophore transport in the solvent thus supports dye extraction [12].

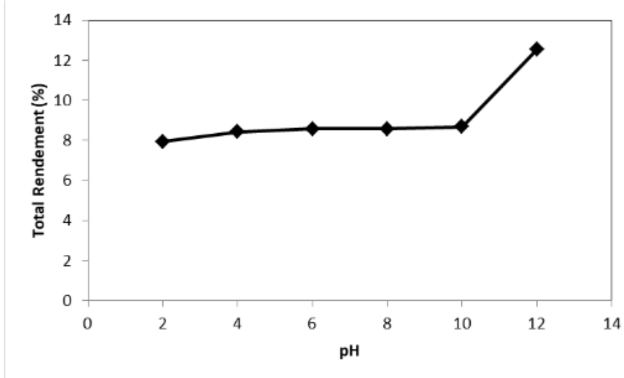

Figure 5. Effect of solution pH to total rendement

4. Conclusions

Brachiaria mutica, abundantly available plant material, has potential use of natural dyes. Production of natural dyes powder would increase the application of natural dyes as an alternative of synthetic dyes. The study succeeded in optimising parameters and operating conditions for the production of *Brachiaria mutica* dyes powder. Gaining insignificant difference of total rendement, methanol was the most suitable solvent for *Brachiaria mutica* extraction. *Brachiaria mutica* extraction must be carried out in temperature and time of 90^{o}C and 180 minutes, respectively. Solution pH strongly affects dye extraction. Investigation on solution pH revealed the optimum pH of 12.

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Acknowledgments

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