A COMPARISON OF BOILING AND SOXHLET EXTRACTION METHOD IN NATURAL DYEING

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ABSTRACT
A natural dyeing of silk using boiling and soxhlet extraction method in obtaining the dyestuff using leaves of Morinda citrifolia (mengkudu) and fruits of Melastoma malabathricum (senduduk) was studied. Types of mordant chosen were chemical-based mordant and natural mordant in order to obtain various colours. The purpose of this study is to prepare the dyestuff using both methods and to compare the colour obtained by the two methods and between the two types of mordant. As the dyestuff was obtained, the dyeing process was carried out at initial temperature of 30ºC for 30 minutes and raised up to 85ºC for another one hour. The results in terms of colour analysis, time to obtain the dyestuff and the fastness properties were analyzed and compared within these two methods. It was found that the boiling method produced a deeper shade of colours and the dyestuff was obtained in a shorter time. It was found also that both methods obtained good fastness properties in terms of staining but less in change in colour after washing for some mordant. Most of the results also depend on the mordant used. This study has proven that boiling method has a better shade of colors and saves in time but the shelf life of the dyestuff is very short. However, the soxhlet extraction method could be used in order to commercialize the natural dyestuff.

Keywords: boiling, soxhlet extraction, natural dyeing

1.0 INTRODUCTION
The ability of natural dyes to colour textiles has been known since ancient times. The earliest written record of the use of natural dyes was found in China dated 2600 B.C. History relates that natural dyes were used in the early days of the Roman Empire the so-called Tyrian purple was used for the royal family. The substance that first produced the colour purple was derived from a kind of snail. For the past 30 years or so, the use of natural dyes has mainly confined to craft dyers, who use natural dyes for their own personal textiles (Wingate, I.B. 1964). Obtaining natural dyestuffs and mixing them to obtain the desired colour is a slow process compared with the present methods of commercial dyeing, in which all the dye are called synthetic or chemical. They are man-made by the mixing of certain chemicals whose bases are salts or acid. Nowadays, the synthetic dyes have replaced natural dyes (Brito, K.K. 2002). However, there is an increasing awareness of protecting environment in textile industry. Finishing and dyeing section are the major sections that contribute pollution to the
environment. In dyeing section for example, the toxic effluent usually contributed from the dyes, mordant and other various chemicals that are not treated before being thrown away into the source of water. For the reason of this, it has persuaded chemist and scientist to reassess the feasibility of natural colorants. Although it might not be able to overcome the problem as a whole but perhaps could reduce the level of contamination (Omar, K, Tumin, S.M, Ghani, S.A. 2003). The reason of saying that using natural dye could not totally safe the environment is that most natural dyes are not particularly toxic in themselves, but they will not stick to fabric unless a mordant is used. Typically, a mordant is a heavy metal. Heavy metals are extremely toxic and bad for the environment as well. The protection of the environment has become a challenge for the chemical industries worldwide (Omar, K, Tumin, S.M, Ghani, S.A. 2003).

There were four objectives of doing this study. They were to prepare natural dyes using boiling and soxhlet extraction methods, to compare the dyed samples in term of colours and shades obtained from extraction and boiling method, to compare colours and shades obtained from environmental friendly mordant with chemical based mordant and to analyze the colourfastness of the dyed samples. The scope of the study was confined to two types of plants that were Melastoma malabathricum (senduduk) and Morinda citrifolia (mengkudu). Melastoma malabathricum dye source was from fruits and Morinda citrifolia dye source was from leaves. The reason for choosing those parts was due to the availability of the sources. Two types of mordant were chosen that; environmental friendly mordant which were wood ash and tawas while chemical based mordant were aluminium potassium sulphate (alum), potassium dichromate (chrome), stannous chloride (tin). The fabric used in this study was plain silk fabric and the dyed materials were analyzed under Malaysian Standard on the colourfastness testing. The testing conducted were washing testing, rubbing/crocking testing and perspiration testing.

2.0 METHODOLOGY
This study was carried out by stages of procedures. The first method was to obtain the dyestuff using boiling and soxhlet extraction. After obtaining the dyestuff, it was followed by dyeing process and lastly colourfastness testing. The dyed fabrics were analyzed under Malaysian Standard on the colourfastness testing. The testing conducted were washing testing, rubbing/crocking testing and perspiration testing.

2.1 Boiling method
The leaves and fruits were freshly plucked and blended. The amounts were about 30 g. After that, they were blended and then put into a beaker where 150 ml of distilled water was poured in. The beaker was then heated about 15 minutes. In order to obtain the clearest solution of dyestuff, the mixture was cooled down before being strained.

2.2 Soxhlet extraction method
The leaves and fruits were blended at about 1 kg and they were put into a porous bag. The bag was placed in an extraction chamber, which was suspended above a flask containing methanol as a solvent. The flask was heated for 10 hours and the solvent evaporates and moves up into the condenser where it was converted into a liquid that trickles into the extraction chamber containing the sample. The extraction chamber was designed so that when the solvent surrounding the sample exceeds a certain level it overflows and trickles back down into the boiling flask. At the end of the
extraction process, the flask containing the solvent was removed. Then, the sample was dried using Rotary Evaporator.

The next process was to remove the latex element in the solution using petroleum ether. The dried solution was mixed with the petroleum ether in a separator funnel with a ratio of 1:1. After a moment, the mixture formed two layers whereby the upper layer was the petroleum ether and the lower layer was the crude sample without the presence of latex. The lower layer was then put inside a round bottom flask and dried again using Rotary Evaporator.

![Image of the sample in the separator funnel](image)

**Figure 1: The sample in the separator funnel**

After drying, distilled water was added to the sample in order to freeze it. This was carried out as the freeze temperature of the methanol is -98°C, which is hard to be obtained and takes longer time. Water on the other hand has freeze temperature of 0°C. The mixture was shook until all the samples dissolve in the water. After that, the sample was put in a freezer. The frost sample was then defrosting using freeze dryer for about three to five days depending on the water being added in the previous step.
The more the water, the longer the process needed. At the end, a powder form of the sample obtained. The sample was then kept in vacuum desiccators. At the end, the weight of sample was about 7 g.

**Figure 2: The Freeze Dryer**

**Figure 3: Keeping the sample in vacuum desiccators**

### 2.3 The dyeing process

The dyeing method process used in this study was the metachrome mordanting process or simultaneous mordanting process. This could save time by cutting out mordanting process as a separate stage and resulted to only one bath was required. The dye bath solution was measured based on the weight of the fabric. A liquor ratio of 1:50 was used. The calculation for total volume solution needed is as shown below:

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\text{**Total volume of dyeing solution} = \text{O.W.F} \times \text{Liquor ratio (mL)}
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The solution was then poured into a metal tube, which was then placed in the Roaches Dyeing Machine. Dyeing was conducted at 30°C for 30 minutes and the temperature was then increased to 85°C. At that temperature, dyeing was continued for another hour. After the dyeing process was completed, all the dyed materials were rinsed with cold running water and then let dry.
2.4 Colourfastness testing
Colourfastness refers to dyes that do not shift hue or fade when exposed to light and other environmental factors and that does not migrate onto other fabrics or material during storage, processing, use or care (Kadolph S.J., and Langford A.L., (2002). All the samples of dyed fabric were tested for perspiration, rubbing/crocking and washing fastness.

2.4.1 Colourfastness to washing
For colourfastness to washing, the Standard Method: MS 308: Part 14: 1975 was used. One composite sample per testing of size 10 cm x 4 cm was prepared. The composite sample consists of the dyed sample placed between the 100% silk and 100% cotton of equal size. The four sides were then sewn together. A soap solution was prepared and heated to 60 ± 2°C using liquor ratio of 1:50. The composite sample was then placed in a metal container containing the soap solution. The container was tightly closed and then placed inside the Autowash set at 60 ± 2°C for 30 minutes. After 30 minutes, the container was taken out and the sample removed and rinsed in running tap water. The stitches on the two long sides and one short side were unpicked leaving the three fabrics attached only at the short side. Samples were let dry. The results of the testing were then evaluated for change in colour (CIC) and staining using the grey scales.

2.4.2 Colourfastness to perspiration
Colourfastness to perspiration conducted in two chemical solutions, one alkaline and another one was acidic. The alkaline solution consisted of 0.5 g L-histidine monohydrochloride monohydrate, 5.0 g sodium chloride, 5.0 g crystallized disodium hydrogen orthophosphate, 0.1 N sodium hydroxide (pH 8.0), and 1.0 L distilled water. The acidic solution consisted of 0.5 g L-histidine monohydrochloride monohydrate, 5.0 g sodium chloride, 2.2 g crystallized sodium dihydrogen orthophosphate, 0.1 N sodium hydroxide (pH 5) and 1.0 L distilled water. Two composite samples similar to that of the washing test were prepared. Each composite sample was then placed in 2 small beakers into which of the acidic and alkaline solution using liquor ratio of 1:50 were poured separately. The mixture was stirred for 30 minutes and the composite samples were then taken out and squeezed out between two glass rods. These samples were then placed on the spirometer and put in the oven set at 37 ± 2°C for 4 hours. Then the samples were dried and evaluated using the grey scales.

2.4.3 Colourfastness to crocking
Two white cotton of 5 x 5 cm were prepared to be tested for dry and wet rubbing. The sample was then placed on the crocking machine and rubbing of the white cotton to and fro was allowed for 10 times. For the wet rubbing, the process was repeated with the white cotton being wetted first in distilled water. Evaluation was done based on the staining of the white cotton due to rubbing on the fabric.

3.0 RESULTS AND ANALYSIS

3.1 Colour analysis
The colour analysis of the samples was carried out manually using lighting cabinet by evaluating the change in colour. The boiling method samples were chosen as the control samples. The evaluation
shows that the boiling method samples have darker shade as compared to the soxhlet extraction method. This is due to the process of dyeing directly in the solution of dyes from the leaves etc. Therefore, the true colour of the leaves was obtained and thus produced deeper shade. The evaluation also shows that colour produced by natural mordant almost in the same hue of the colour produced by the chemical-based mordant. It was observed also that the chrome and tin mordant produce a moderate change in colour meanwhile other mordant had shown a poor change in colour. This may be because it depends on the mordant properties itself. The function of the mordant is to fix a dye to the fibers or fabrics. However, the reaction of the mordant towards the natural dyes is different. Therefore, the change in colour was varying for different mordant. Samples being dyed by senduduk produced a colour of purple but the tone of the hue varies when different mordant was used. Tin mordant for example produced a colour of reddish purple while alum mordant produced a bluish purple. Meanwhile mengkudu produced a colour of greenish yellow and the colour obtained was not obviously different when different mordant was used.

3.2 Time of obtaining the dyestuff
In boiling method, the source of dyestuff was boiled for 15 minutes. After that, the solution was cooled down in order to obtain the clearest solution. This process took about 30 minutes. Therefore, the total time needed to obtain the dyestuff was approximately 45 minutes. However, soxhlet extraction method needed a longer time. The process of extracting the colour itself took about 10 hours. After that, the process of drying, removing of latex and freezing took about 24 hours or a day. The last process was the transferring the frozen samples into powder which took about 5 days to be completed. Therefore, the total time needed to obtain the dyestuff in soxhlet extraction method was about 7 days.

3.3 The ease of application
The boiling method produce dyes in liquid form whereas in soxhlet extraction the dyestuff was in the form of powder. Therefore, it was found that the powder form is easy to be applied, as it is easy to be stored. However, the liquid form needs to be used in a short time and it needs to be stored in a cool temperature for example in a refrigerator. The shelf life of boiled natural dyestuff is very short.

3.4 Color fastness analysis

3.4.1 Analysis of rubbing/crocking fastness
Samples dyed by senduduk and mengkudu showed a good colourfastness to rubbing and crocking. In conjunction with that, both methods that are boiling and soxhlet extraction show an approximately same rating of colourfastness. In term of mordant, overall, both natural mordant and chemical-based mordant gives a rating of 5 and 4/5 in dry and wet condition of testing which indicate good colourfastness too.

3.4.2 Analysis of washing fastness
The rating of staining for silk and cotton was 5 for mengkudu in both methods for all of the mordant used. Meanwhile, dyeing with senduduk gives a range of moderate to good rating of staining for both methods. Most of the mordant showed a rating of 5 and 4/5, which indicate a good rating except alum with a rating of 3 and 3/4 for boiling and soxhlet method respectively. Both methods showed a good
colourfastness to washing in change in colour for *mengkudu* and this indicated by all of the mordant used. Meanwhile for *senduduk*, a poor colourfastness results were obtained by using tin, alum and tawas mordant. All of the mentioned mordant showed a rating of 1/2 by both methods. This is may be due to the different reaction of mordant towards natural dyes. One of the factors that determine the fastness to washing is the rate of movement of the dye outward from the fibre in the presence of soap. Therefore, the degree of fixation of the mentioned mordant may be lower so that causes the dye molecule moving outward of the fabrics at a higher rate in the presence of soap solution. Only chrome and wood ash obtained a good colourfastness with a rating of 5.

### 3.4.3 Analysis of perspiration fastness

Colourfastness to perspiration was tested in two conditions that were acidic and alkaline. Both plants in both methods showed a good rating of staining in silk and cotton for both conditions. All of tested samples showed a rating of 5. In term of change in colour, *senduduk* and *mengkudu* had obtained a range of moderate to good colourfastness. This applied to both acidic and alkaline condition. Such example was dyeing with *senduduk* using tawas mordant, whereby the rating for boiling method and soxhlet extraction method in acidic condition was 3 and 3/4 respectively. The same result also obtained in dyeing with *mengkudu* in the same condition.

### 4.0 CONCLUSION

In term of colours, boiling method produced a darker shade of colour as compared to the soxhlet extraction method. In term of mordant type, both natural mordant and chemical-based mordant produced approximately same colours. This means that natural mordant has potential to be commercialized. In term of colourfastness, both methods produced good properties of colourfastness especially in rubbing and perspiration. However, both methods indicate poor colourfastness to washing for some mordant. This may be due to the properties of the mordant itself when reacting with natural dyes. Both natural and chemical-based mordant produce approximately same rating of colourfastness to rubbing, perspiration and washing. Overall, this study had achieved the objectives and boiling method shows a better result in colours, shade, time of producing the dyestuff and the colourfastness analysis. However, in term of commercialization of natural dyeing, soxhlet extraction method should be considered as the shelf life of the dyestuff is longer and it promotes ease of application and storage.
5.0 BIBLIOGRAPHY


