Technology in Textile Dyeing: Resurgence of Natural Dyes from *Adonidia Merillii* (Betel Nuts) Fruits Husk

IOP Conf. Series: Materials Science and Engineering **917** (2020) 012013 doi:10.1088/1757-899X/917/1/012013

Technology in Textile Dyeing: Resurgence of Natural Dyes from *Adonidia Merillii* (Betel Nuts) Fruits Husk

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Abstract. Natural dyeing is one of the indigenous pieces of knowledge that has been explored on its potential since prehistoric decade. The process and materials used revolves nature-friendly essential, which are safe to the environment in comparison to the chemical dyeing materials. Currently, the *Adonidia Merillii* or also known as betel nut tree, is only used as an ornamental tree such as in landscaping. However, it has potentials of being more than an ornament and can be utilized as dyes for local textile industries. Thus, this study focused on extracting dye from young Adonidia Merillii fruits husk. Traditional boiling method was applied to extract the natural colours from the fruits. Colour testing was conducted on four (4) types of fabrics which were *rayon*, *Crepe de Chine* (CDC), *China Cotton* and *jacquard* by using post-mordanting treatment. The mordants used were aqueous lime water, alum, and ashes. The colour on the textile samples was tested and evaluated via colour fastness properties. The tests conducted include exposing textile samples to artificial light, washing, rubbing, water and perspiration. *Adonidia Merillii* fruits husk have given hues of brown colour staining on the textiles, but the colour changed after the test, surprisingly on the artificial light exposure. The bright colour of the initial samples appeared to be less bright than those exposed to artificial light. The brightness of the samples was determined using blue scale grading. The findings from this experimental study may contribute to the palette colour of natural dyeing on textile and extended future research about the brightness change would be significant for the Malaysia local textile industries.

1. Introduction

Natural dyeing process is one of the acknowledged technologies from the ancients to produce dyes and colours by utilizing materials from natural resources. Natural dyes pigments can be obtained through extraction process on plants, insect, animals and minerals [1],[2]. In the early age, natural dyes were widely used as media to write and draw images on caves' wall to present story and spiritual belief [3], materials to paint houses, and for textile and craft dyeing applications. Apart of being materials for colouring, dyes also can act as agents to improve durability of textiles and fibres. Appealing colours on textiles certainly symbolize the identity, culture and lucrative local knowledge in dyeing skills. Therefore, re-introducing dyeing process using natural resources can encourage the spirit of appreciating traditional knowledge in dyeing textiles, as well as to boost awareness on sustaining the

environment using alternatively eco-friendly dyes. Disposal and public burning of plastic waste practiced by most banner advertising industry contributes to wastage and environmental pollution [35]. In this paper, natural colouring refers to dyeing process using nature resources in local textile production for plain dyeing or resist techniques dyeing applications.

In recent years, there has been growing interest in natural dyeing research [4] reflecting the effect and affect caused by chemical colour substances widely used in textile production. Uncontrollable dyeing process results in water pollution that leads to deterioration of vicinity [5], [6]. Other than that, usage of chemical dyes can cause safety and health issues, in a long run, as it exposes workers who are working in this industry to hazardous substance for dyeing, especially when the workers are not provided with safety equipments during execution of the works [7]. Synthetic dyes were first introduced in 1856 by William Henry Perkin. This material has gained massive popularity due to advantages it has in production and also the colour it possesses [8]. In Malaysia, synthetic dyes have been utilized since 1920s for local textile productions. Synthetics dyes changed the acceptance rate on process and colours produced by natural dyes. This is because, natural dyeing is time-consuming, complicated and have limited colours for selection [1], [9]. Most manufacturers choose synthetic dyes over natural dyes in the efforts to meet the consumer's demand based because synthetic dyes are cost-effective. Synthetic dyes give solution to intricate dyeing processes, unfortunately it significantly deteriorates environment. On the other hand, not like synthetic dyes, dyeing process using natural resources possesses environmental element of sustainable development. Therefore, this experimental study was undertaken to find alternatives to synthetics dyes. For that purpose, young Adonidia Merillii fruits husk was chosen as raw material for producing natural dye, for this study. The extract as well as the extraction processes involved were then evaluated and studied, in order to determine the best procedures that can produce the best quality textiles with the most and fascinating colours.

2. Adonidia Merillii (Betel Nuts)

Adonidia Merillii of *Arecaceae* is a palm tree species. It originated from Palawan, Philippines. *Adonidia Merillii* is commonly known in several names as Christmas Palm, Chinese betel nuts, Dwarf Royal Palm, Kerpis Palm, Manila Palm, Veitchia Palm, Bunga de China and Oring – oring [10],[11]. This palm tree can be utilized as raw material for many areas like sugar production, vegetable oils production and building materials. It is also known as a source for edible fruits, buds and used as ornament plant for landscape. In Malaysia, this palm tree is commonly used as an ornamental to beautify landscapes, beaches and houses, as well as providing the cooling effect to the environment [12]. *Adonidia Merillii* is a resemblance to *Areca Catechu*, but the difference of these two species can be detected by looking at their fruits (refer Figure 1). Usually *Areca Catechu* mentioned are lack on testifying the types of palm tree used and in Malaysia it generally known as *'buah pinang'* or betel nuts.

Adonidia Merillii is a fast-growing palm. It can reach a height of 8 meters with foliage that can grow up to 2 meters long which normally equipped with 40 to 50 pairs of leaflets and it produces fruits from the buds [11], [12]. Young betel nuts have green-coloured husk whereas ripen betel nuts have bright scarlet or bright crimson colours [11], [13]. Betel nuts of *Adonidia Merillii* sometimes used as a substitute to *Areca Catechu* fruits in preparing traditional rituals like '*makan sirih'* or chewing the piper betle or also called '*buyo'* in Philippines. The ingredients are betel nuts, piper betle leaves, gambier (*Uncaria gambir*) and lime [11], [14], [15]. In Malay culture, betel nut is one of the important elements in '*tepak sirih'* or ornamental objects in rectangular wooden or brass box. It is used in Malay community especially for special ceremonies such as marriage proposal, engagement, wedding and also royal ceremonies as non-verbal communication [16],[17]. Malays believe that Betel nuts symbolize good characters and betel nut tree symbolize high level of good characters among Malay people [18],[19].

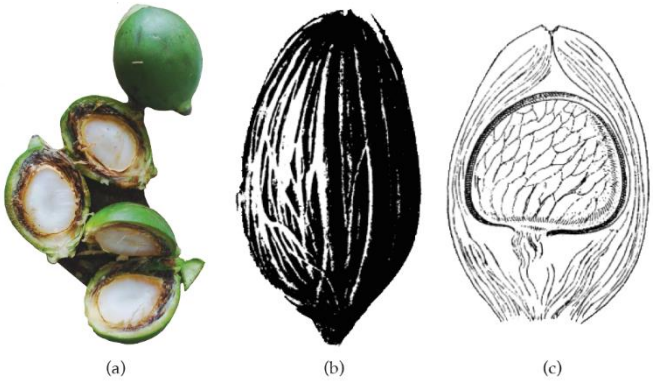

Figure 1. (a) show young *Adonidia Merillii* fruits used in this study. (b) show *Adonidia Merillii* fruits without husk and (c) show the *Areca Catechu* fruits.

Nowadays, betel nut trees are more known as ornament plants, therefore the fruits are not fully utilized and usually left, ripen and fall to the ground. Therefore, in this study betel nut is seen to have low economic value but at the same time provide no adverse effects to the environment [20]. Furthermore, previous study shows betel nuts can be used as alternative materials to produce eco-friendly dyes for fabrics made of kenaf [21]. Betel nuts extract has antioxidant and cytotoxicity properties [13] and can also be used as hair dye that produce ivory to chocolate colour [22]. Then this study alternatively using the wasted and abandon nature resources to produce natural dyes for colouring the selected silks and cotton fabrics. Thus, this paper reports the processes and procedures involved in betel nuts extraction which was intended to be used as natural dye for textile.

3. Methodology

This experimental study was conducted in a laboratory of Malaysian Handicraft Development Corporation (MHDC), Kelantan Branch, Malaysia in April 2019.

3.1. Materials

Two materials were involved for this study: 1) potential raw materials for production of natural colorant / dye namely young Adonidia Merillii fruits husk and 2) textile samples. The young *Adonidia Merillii L.* fruit husks were obtained from Irama Beach in Bachok, Kelantan, Malaysia. The freshly collected fruits with green concentration colour used in this experiment. Meanwhile, the fabric samples used were *Rayon*, *Crepe de Chine* (CDC), *China Cotton* and *Jacquard* with the size of 43 inches x 18 inches. The mordants utilized in this experiment were lime water, alum and ashes water.

3.2. Instruments

Instruments included in this study were for extraction and dyeing preparation, as well as for the preparation of colour fastness testing. Tools used for extraction and dyeing were knife, cutting board, pots, stoves, sieve cloth, basin, wrapped block stone (functioning as ballast to let the attire fabrics soaked during dyeing process), digital scale and 1.2-liter jar. As for the colour fastness testing involved the following materials, apparatus and machineries. The apparatus involved are distilled water, 150 ml beaker, stainless steel rod, clear flint rod, acid solvents, alkali solvents, SDC Multifiber DW (BN EN ISO 105 F10), magnetic mixer, FBA Free detergent and heating plate. Meanwhile, according to the Malaysia Standard MS 692:2007 (Malaysia Batik), the machinery is used for the quality controls method for textile testing. The machine involved in this study are Crockmeter (MS ISO 105-X12), Xenontest machine (MS ISO 105-B02), Perspirometer and Oven (MS ISO 105-E04), Linitest machine (MS ISO 105-C01) and for colour evaluation used Grey Scale for Assessing Staining (ISO 105 A03:1993) and Grey Scale for Assessing Change in Colour (ISO 105 A02:1995).

3.3. Natural Dyeing Process

In this experimental study, there were phases involved in the processes of preparing natural dyes as well as dyeing the textiles. The phases were fabric treatments, husk extractions, textile dyeing and mordanting. In addition, two (2) more phases were testing and evaluating the coloured textiles. **Table 1** shows the phases, process flow and estimated time taken in completing this study.

Table 1. The flow of natural dye process and the durations taken for this experiment.

Phases	Process Flow	Durations
1: Fabric Treatment	Pre - Fabric Treatment	15 minutes
2: Extraction Preparation	Boiling	8 hours 26 minutes
3: Dyeing	Soaking	3 hours
4: Mordanting	Post - Mordanting Ashes / Lime Water / Alum	3 hours
5: Testing	Colour Fastness	Artificial light : 24 hours Washing : 30 minutes Rubbing : 30 seconds Water : 5h 30mins Perspiration : 5h 30mins
6: Evaluation	Grading	

3.4. Preparation of Fabric Treatment

Textile samples used were – silk-based textiles i.e *Crepe de Chine* (CDC), *Jacquard* and cotton-based textile i.e *Rayon* and *Cotton China*. The textile samples were soaked and scoured with clear lime water for 15 minutes. Then, the textile samples were rinsed with tap water and air dried at room temperature [23],[24]. This initial treatment or bleaching process was intended to remove stains that may exist on the textiles, hence encourage the textiles to absorb dye more efficiently [25].

3.5. Dye Extractions

The extraction started by weighing [26] and grinding the young betel nut husks. The husks were separated from the freshly collected fruits before they were grinded into smaller particles. 4.6kg of the particles were then placed in a pot. The pot was added with 9 liters of tap water until the particles were fully soaked with the water. The boiling process for extraction was conducted by adopting the traditional method. The boiling process was terminated when the remaining water was half of its original level. The total duration of the boiling process was 8 hours 26 minutes. Previous study showed that higher temperature and longer extraction duration can produce higher dye colour intensity [27]. The aqueous extract then was filtered and stored for dyeing process.

3.6. Dyeing

Dye extracted from betel nut husks was reheated for 30 minutes. The textiles then were placed into the hot dye solution for 3 hours. The textiles were flipped and turned from one side to another, every 30 minutes to expedite the dye absorption and achieve even colouration. Once the process has completed, the textiles were removed from the solution and were rinsed with tap water until the water ran clear. The textiles were then hanged and air-dried at ambient temperature in a room which was away from direct sunlight, because sunlight can effect the colouration and causes the final product to have duller colour [28].

3.7. Mordanting

Natural dyes involved the binder substances called mordant. It acts as catalyst to change the colour of extracted dyes and also functions as colour fixation [1], [29]. There are several conditions can be carry with mordanting process and gives different results for the colour for pre-mordanting, simultaneous mordanting and post-mordanting [30], [31]. This study used post-mordanting, which means the textiles were dyed with the betel nut husks extract, prior to soaking into the prepared mordants (ashes water, alum and lime water) for 3 hours and turning over the fabrics frequently.

3.8. Colour Fastness Testing Procedures

Colour fastness refers to the resistance of colour to fade to numbers of influences which usually exist in our daily life or even in manufacturing process such as water, light, rubbing, washing and perspiration. Colour fastness results could help us to get insights on how to handle the tested materials in order to minimize the effects of the influences mentioned earlier to the colour quality. Colour fastness test was conducted in accordance with the Malaysia Batik Standard from Malaysia Handicraft Development Corporation (MHDC), Kelantan Branch which is commonly utilized for quality control. The test involves are (1) colour fastness to artificial light, (2) colour fastness to washing, (3) colour fastness to rubbing, (4) colour fastness to perspiration and (5) colour fastness to water. The procedures were summarized in the Table **2**.

3.9. Colour Evaluation

The colour quality of the samples tested with colour fastness test were then evaluated by using colour grading with the following procedures: (1) blue wool colour standard scale to evaluate the textile samples exposed to artificial light [32], the evaluation scale ranges from 1 to 4 which indicates as failed and 5 to 8 which indicates passed. (2) Grey Scale for Assessing Staining (ISO 105 A03:1993) was used to evaluate colour stained on white fabrics or multifiber fabrics, and (3) Grey Scale for Assessing Change in Colour (ISO 105 A02:1993) was used to evaluate the fading colour of the dyed fabrics. The scale for staining and changes in colour evaluations range from 1 to 3 (represent failed) and 4 to 5 (represent passed).

Table 2. The colour fastness test procedures on fabrics samples include its machinery used and the time taken to get the result.

Colour Fastness Testing	Machine	Procedures	Durations
1. Colour Fastness to Artificial Light (MS ISO 105-B05)	Xenotest	: This test exposed the dyed fabrics to the artificial light which resembled exposing the fabrics to the direct sunlight. : ***Sample size for test*** – 1cm x 4.5cm stapled to the 'sample holder'.	24 hours
2. Colour Fastness to Washing (MS ISO 105-C01)	Linitest	: This test exposed the dyed fabrics by washing in hot water to determine how much the colour fading. : ***Sample size for test*** – 10cm x 4cm for 3 pieces samples which are white fabrics with the same fabric with the dyed dyed fabric, and coarse fabric. Then, it was stitched 'sandwiches' respectively and inserted with 5 pieces of bearing balls. : ***Preparation*** – 2 liters of distilled water boiled with FCE Phosphate weighted for 10g and stirred. The soap was detergent free. 100 ml mixtures poured into the washing case and ready to be tested.	30 minutes
3. Colour Fastness to Rubbing (MS ISO 105-X12)	Crockmeter	: It purposely tested in wet and dry for rubbing test to see the colour staining after the scrub. : ***Sample size for test*** – 29cm x 3.75cm dyed fabrics and 5cm x 5cm white fabric. The white fabric will be attached on the weight and dyed fabrics on the crockmeter plate, and both are rubbed for 20 seconds.	30 seconds
4. Colour Fastness to Perspiration (MS ISO 105-E04)	Perspirometer and oven	: This test wants to test the effect (colour fading) of sweating on dyed fabrics by resemblance the sweat using acidic solution with the pH 5.5 and alkali solution with the pH 8.	30 minutes
5. Colour Fastness to Water (MS ISO 105-E01)		: ***Sample size for test*** – 10cm x 4 cm for 2 pieces samples which are dyed fabrics and multifiber fabrics and stitched together. : ***Preparation*** -The samples were placed in the beaker and poured with 50ml acidic solutions and stirred. Tossed on the clear flint glasses for 30 minutes. The samples were arranged on AATCC Perspiration Tester and weighted with 5kg pressure weight AATCC. Then, the samples left in the perspirometer machine for 4 hours. Lastly, dry the samples by putting them into the oven for 1 hour. The procedure for acid, alkali and water test are same respectively different with the solution used.	5 hours

4. Result and Discussion

Results were exhibit in Table 4, Table 5 and Table 6 respectively using post-mordanting with lime water, alum and ashes water. The discussion on the result and findings for this experiment elaborated into two states which are (1) the colour obtained and (2) the colour fastness properties.

4.1. Colour Obtained

The results showed that post-mordanting with lime water gave vibrant and dark brown colour to Crepe de Chine (CDC) and Jacquard, compared to Rayon and Cotton China which exhibited soft and pastel brown colours. Meanwhile, post-mordanting with alum and ashes aqueous on CDC, Jacquard, Rayon and Cotton China has produced soft and **pastel brown shades** of colour on the textiles.

Table 3. pH values of solutions used for dyeing process.

Materials	pH Value
Adonidia Merilli L. Aqueous	5
Lime Water	13
Alum	3
Ashes Water	8

Table 3 shows pH values of solutions used in the experiment. *Adonidia Merillii* fruits husk extract and ashes water appeared to have low acidity to low alkalinity values [33]. The pH values were 5 and 8 respectively. The pH value for alum was strong acidity and meanwhile, for lime water, it was strong alkalinity. The study showed that, the Adonidia Merillii fruits husk extract and lime water was the best dyeing to give dark brown colour on silk fabrics. However, depending on textiles and mordants used, young *Adonidia Merillii* fruits husk extract may also give variety colour hues of brown staining.

4.2. Colour Fastness Properties

Colour fastness for fabrics colourant evaluationare using the technical instruments to show the durability of the dyes staining on the textile samples. The test includes the srtificial approach concerning the reaction towards the sunlight, pressure, washing and perspiration.

The study showed that young *Adonidia Merillii* fruits husk extract had poor resistance to artificial light, where the score was 1 to 2 (failed). Lime water which was used for mordanting was found to give the best colour absorbance rate for better colour obtain. Rayon and Cotton China smaples mordanted with ashes water werealso found to have poor colour fastness to light. However, surprisingly, the Crepe de Chine (CDC) samples mordanted with alum and then exposed to artificial light as well as Jacquard samples mordanted with ashes gave opposite results, where the samples appear to have better colour staining results or brighter colour than the original dye staining.

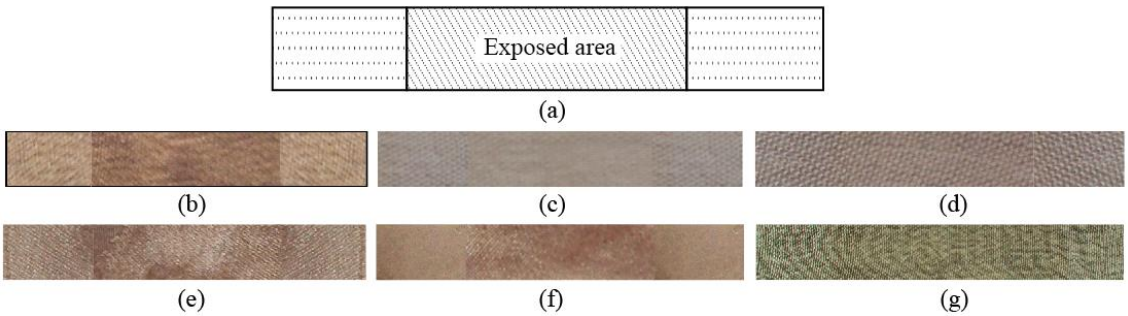

Figure 2. The illustration and results of colour fastness to artificial light.

Figure 2 shows the illustration of how the colour fastness was performed and the results of the textiles samples exposed to artificial light. The labeling of the figure are as follows: (a) the textile size on the sample holder, the center of the textile is the exposed area whereas both right and left areas are

the control samples. (b), (c), (d) and (e) are the result of Crepe de Chine (CDC), Cotton China, Rayon and Jacquard mordanted with alum respectively. Whereas, (f) and (g) are the results of Jacquard and Crepe de Chine (CDC) mordanted with ashes respectively. There were slightly changes detected in comparison to the control samples and that changes are significant enough to grade the colour of the samples with score 5 because the samples are not fading but reacted by giving other hues of brown colour. The exposure to light show excellent too good with the score 4-5, 5-6, 6-7 and fair result for scoring 1-2 and 2-3 [34].

The results for colour fastness of the textiles mordanted with different mordants, and then were subsequently tested with washing, rubbing, perspiration and water were briefly summarized in Table 4, Table 5 and Table 6.

Colour fastness results of the textiles tested with washing showed good to excellent resistance with the score for colour change and colour staining were 4 – 5. Meanwhile, colour fastness results of the textiles exposed to rubbing also showed good to excellent resistance (score 4 – 5 and 5) for all textile samples except for Rayon and Jacquard mordanted with lime water and Cotton China mordanted with ashes aqueous which showed fair results (score 3) for colour staining. The results of exposing the textile samples to perspiration which was to test the effects of alkalinity and acidity to the samples showed good to excellent resistance score on colour change and colour staining. The scores were 4 – 5. Lastly, the water test results on colour change and colour staining also showed good to excellent scores which were 4 – 5. Obviously, young Adonidia Merillii fruits husk extract had good results on the colour fastness properties but otherwise on the light exposure which also give significant results on poor colour with lime water mordanting, but it is giving new colour hues for alum and ashes aqueous. These showed that young Adonidia Merillii fruits husk extract can be used for alternative textile dyeing.

Table 4. The results of colours obtained and colour fastness properties with regards to different types of textiles which were dyed with *Adonidia Merillii* fruits husk extract and post-mordanting with **lime water**.

Experiment Properties / Fabrics	Colour Obtain on Sample	Colour Fastness to Artificial Light	Colour Fastness to Washing		Colour Fastness to Rubbing		Colour Fastness to Perspiration		Colour Fastness to Water	
					Wet	Dry	Acid	Alkali		
			CC	CS	CS	CS	CS	CS	CC	CS
Rayon		**1**	4	4	**3**	4	4/5	4/5	5	4/5
CDC		1/2	4/5	4	4/5	4	4/5	4/5	4/5	4/5
China Cotton		1/2	4	5	4	4/5	4/5	4/5	4/5	4/5
Jaquard		2/3	4	4/5	3	3	4/5	4/5	5	4/5

CC = Colour Change ; CS = Colour Staining

Table 5. The results of colours obtained and colour fastness properties with regards to different of textiles which were dyed with *Adonidia Merillii* fruits husk extract and post-mordanting with **alum**.

Experiment	Colour	Colour Fastness	Colour Fastness	Colour Fastness	Colour Fastness to Perspiration	Colour Fastness to

Properties / Fabrics	Obtain on Sample	to Artificial Light	to Washing		to Rubbing				Water	
					Wet	Dry	Acid	Alkali		
			CC	CS	CS	CS	CS	CS	CC	CS
Rayon		5*	4	4/5	4/5	5	5	4/5	4/5	5
CDC		5*	4/5	4/5	4/5	4/5	4/5	4/5	4/5	5
China Cotton		5*	4	5	4/5	5	4/5	4/5	5	5
Jaquard		5*	4	4/5	4/5	4/5	5	4/5	5	5

CC = Colour Change ; CS = Colour Staining

Table 6. The results of colours obtained and colour fastness properties with regards to different types of textiles dyed *Adonidia Merillii* fruits husk extract and post-mordanting with **ashes aqueous.**

Experiment Properties / Fabrics	Colour Obtain on Sample	Colour Fastness to Artificial Light	Colour Fastness to Washing		Colour Fastness to Rubbing		Colour Fastness to Perspiration		Colour Fastness to Water	
					Wet	Dry	Acid	Alkali		
			CC	CS	CS	CS	CS	CS	CC	CS
Rayon		2	4	5	4/5	5	5	4/5	4/5	4/5
CDC		5*	4/5	4/5	4	4	5	4/5	5	4/5
China Cotton		2	4	5	3	4/5	4/5	4/5	4/5	5
Jaquard		5*	4	4/5	4/5	4	4/5	4/5	5	5

CC = Colour Change ; CS = Colour Staining

5. Conclusion

The result of the experiment fulfilling the objective of the study in extracting dyes from young *Adonidia Merillii* fruits husk to dye several fabrics samples. This study has conferred that Adonidia Merillii which commonly categorized as wasted known to have low economic value possessed value-added characteristic such as dyeing a dyeing agent for textile. Adonidia Merillii extract gave dark brown and soft pastel of brown colour staining on the textiles. The colour fastness properties towards the artificial light, wshing, rubbing, perspirations and water tested on dyed fabrics of Crepe de Chine

(CDC), Rayon, Cotton China and Jacquard were found to be satisfactory. Hence, it is inferred that young *Adonidia Merilli* fruits husk have good dyeability for silk and cotton fabrics.

6. Acknowledgement

The authors wish to thank Malaysian Handicraft Development Corporation (MHDC) for providing laboratory facilities. This study supported in part by the Ministry of Education Malaysia (KPM) for sponsoring the study.

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