

Extraction of Eco-Friendly Natural Dyes from Pina Leaves and their Application on Wool Fabrics

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Abstract

Natural dyes comprise of colorants that are obtained from animals or vegetable matters without any chemical processing. Natural dyes can substitute synthetic dye and promotes green technology initiatives in the field of textile dyeing. This study was carried out by extracting dyes from pineapple leaves (*Ananas Comosus*) using three stage mordanting methods using different mordants namely pre-mordanting, post-mordanting and simultaneous dyeing-mordanting. The mordants used were aluminium potassium sulphate, white vinegar and sodium chloride (NaCl). Wool fabrics were used for dyeing. The strength of colour and K/S values of the dyed fabrics were measured before and after washing. The colourfastness to washing, rubbing and light fastness of the fabrics were conducted to investigate the performance of the dye and mordants. The results indicate that the washing, rubbing and light fastness properties of dyed samples were between good to excellent grades.

Keywords: *Ananas Comosus*; Mordants; Natural Dye; Pina Leave; Wool.

1. Introduction

A dye can generally be described as a coloured substance that has an affinity to the substrate to which it is being applied [1]. The dye is usually used as an aqueous solution and may require a mordant to improve the fastness of the dye on the fiber. Dyes are molecules which absorb and reflect light at specific wavelengths to give human eyes the sense of colour. There are two major types of dyes - natural and synthetic dyes. Natural dyes are extracted from natural substances such as plants, animals, or minerals. Natural dyes from plants are generally obtained from the leaves, roots, stems and fruits. Although natural dyes are more cost effective, non-allergic to the skin, non-toxic and environmentally friendly, they have some limitations on the performance in terms of getting bright colours and maintaining the fastness of the colour on the fabrics [2].

Synthetic dyes are chemicals derived from sources like coal tar or petroleum-based substances [3]. There are many categories of synthetic dyes depending on the type of chemical combination and their application on the material. Some examples are such as acid, basic, oxides, pre-metalized, direct, chrome, azoic, sulphur, disperse, reactive, and vat dye. Almost all the synthetic dyes can be modified to add beauty, in terms of brilliancy, to increase the strength of colours and fastness. The colour fastness of synthetic dyes are generally excellent because the dyes strongly bind to the fabric. However, the production and application of these dyes are threatening the environment by damaging the plants in terms of growth and good yield, killing the aquatic animals, affecting human life, and causing skin cancer [4-6].

As mentioned earlier, most of the natural dyes have limitation on the colour yield and colourfastness. Thus, specific mordants are

used to increase natural dye uptake by textile fibres. Different types of mordants will yield different colours even on the same type of dye. The final colour strength and colour fastness properties are not only dependant on the dye itself but are also determined by varying concentration, the types, and method of applying the mordants [19]. This study used the leaves of *Ananas Comosus* to dye wool fabric and determine the best shades and mordanting method.

2. Experimental Setup

Figure 1 shows the overall methodology of the present research starting from material selection. First is the collection of the plant material. Leaves of *Ananas Comosus* were collected from Pineapple Plantation and Pineapple Museum in Pontian, Johor. After collection, the pineapple leaves were collected, washed and cut into small pieces. To extract the dyes, 5 gm of the *Ananas Comosus* leaves were added in 100 ml of water. The extraction was done at 100°C for 15 to 30 minutes at 6-7 pH. The hot solution was filtered to obtained clear solution which is used for dyeing the wool fabrics.

2.1. Materials

In this research, a wool fabric (1:1/2 twill weave, 40/40 ends and picks per inch, 50s count, desized, scoured and bleached) was purchased from Toyobo Textile Mills Sdn. Bhd., Perak. Three types of mordants were used - potassium aluminium sulphate, white vinegar and sodium chloride (NaCl).

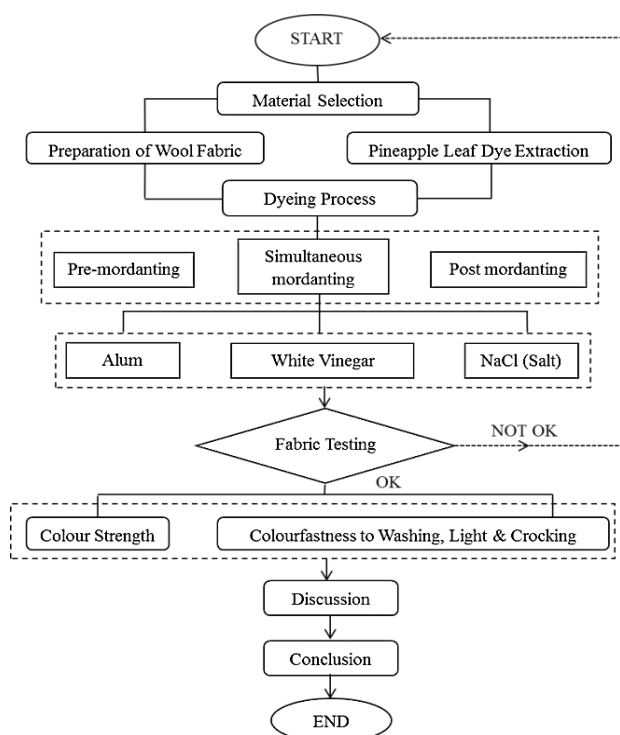


Figure 1: Research Methodology Flowchart

2.2. Mordanting Method Used

Pre-mordanting: The fabric was treated with the mordant for 45 minutes at 60°C. The dyeing was carried out at 1:20 material to liquor ratio, for 45 min at 80°C. Dyeing and mordanting (1%) were done using a Rota-Dyeing machine.

Post-mordanting: In this method, the fabrics were dyed first followed by mordanting. The dyeing method was similar with the dyeing method used in pre-mordanting. After dyeing, the fabrics were rinsed in hot and then cold water and finally treated with mordant solution for 45 minutes at 60°C and rinsed.

Simultaneous-mordanting: In this method, 1 ml of 2% mordant was mixed with 30 ml of dye extracts and then 5 gm of fabric was dyed with it for 45 minutes at 60°C.

2.3. Colour Properties

The K/S values of the dyed and undyed wool and cotton fabrics were determined by measuring the surface reflectance of the samples using a computer aided Mcbeth 2020 plus reflectance spectrophotometer using Kubelka-Munk equation:

$$\frac{K}{S} = \frac{(1 - R_{\lambda\max})^2}{2 R_{\lambda\max}} \quad (1)$$

Where K is the coefficient of absorption, while S is the coefficient of scattering; $R_{\lambda\max}$ is the surface reflectance value of the sample at particular wavelength when maximum absorption occurs for a particular dye / colorant. The K/S values of dyed fabric is directly proportional to the amount of dye present in the material and it has been also found that generally K/S value get increase with the presence of mordant [7].

2.4. Evaluation of Colorfastness

Color fastness to washing: The color fastness to washing of the dyed samples were determined as per AATCC Test Method 61-2009. Fabrics were cut into 10 × 5 cm and placed between two pieces of undyed fabrics (wool and cotton) of the same size. Fabrics were washed with 5% non-ionic soap solution for 45 minute

using washing fastness tester (Launderometer). The samples were rinsed and dried and then measured using grey scale for loss of shade and extent of staining.

Color fastness to light: This test was carried out as per AATCC Test Method 16-2004. The dyed samples with the size of 3 × 6 cm were fixed on a black cardboard in such a way that all samples were half-exposed and half-covered. This frame was placed inside the fadeometer fitted with mercury bulb tungsten lamp (MBTF). The specimens were brought out after 5 hours and the colour fading was assessed against the blue wool standards (BS 1006: BOI: 1978).

Color fastness to crocking: This test was done as per AATCC Test Method 8-2001 using a crock meter. A piece of cotton was rubbed 10 times on a sample using crock meter. Two types of rubbing were done, wet rubbing and dry rubbing. The staining on the wet and dry piece of fabric were evaluated with the help of grey scale.

3. Results and Discussion

The results show that the leaves of *Ananas Comosus* produce a wide range of colors. Next, the study was further done to standardize the methods of extraction and application of dyes on wool fabric.

The spectral plot (Figure 2) shows the graph of reflectance and transmittance for all samples including the dyed fabrics with different method of mordanting and different mordant (potassium aluminium sulphate, white vinegar and sodium chloride). Pre-mordanting samples with different mordant show reflectance wavelengths from 400nm until 500nm in the reflectance of blue region while the simultaneous mordanting samples show reflectance wavelengths from 500nm until 600nm in the reflectance of yellow region. The post-mordanting samples with different mordant show the reflectance wavelengths from 600nm until 700nm in the reflectance of red region. This may be due to the loss of dye during washed-off of pre-mordanting samples. A study done by [8] also agreed that excessive of dye on the fabric surface may show on the reflectance of the wavelength.

Figure 3 shows that the 2D-colour plot graph for the three types of mordanting method with different mordant samples were towards negative in the blue colour region below the yellow-blue line axis and high in the L value in the lightness axis, L-axis. As a result, the colour values for all samples are somehow lighter in colour.

The results of depth of shade of wool dyed with pineapple leaves are summarised in Table 1. The colour differences for NaCl mordant samples show that the pineapple leaves dyed fabrics with pre-mordanting is darker (L^* , 65.60) as compared to the controlled dyed fabrics with white vinegar and potassium aluminium sulphate mordant. The controlled dyed fabric with white vinegar mordant has high a^* which means the shade is redder and the pineapple leaves dyed fabric with potassium aluminium sulphate as the mordant has high value of b^* which means it is yellower.

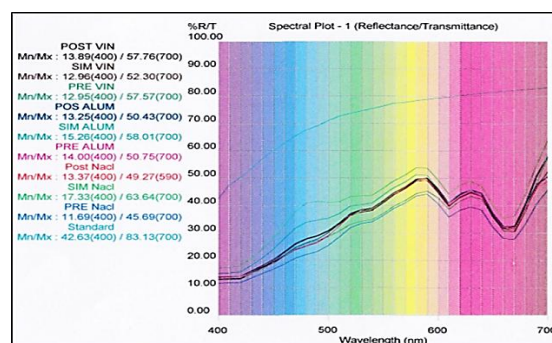


Figure 2: Spectral plot graph for mordanting method with different mordants

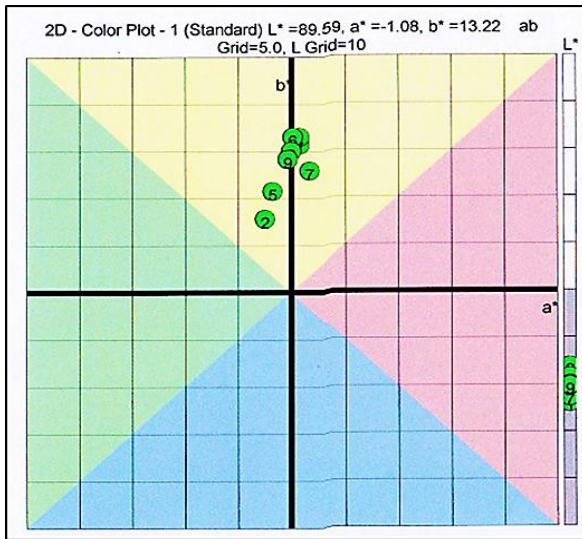


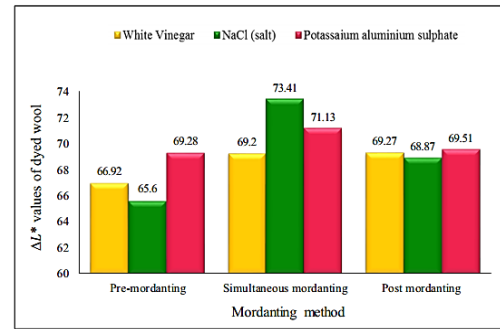
Figure 3: 2D-colour plot graph for mordanting method with different mordants

Figure 4(a) shows the value of ΔL^* while Figure 4(b) shows the value of Δa^* and Figure 4(c) shows the value of Δb^* . The colour strength of the dyed fabric usually depends on the K/S values. The ‘K’ and ‘S’ are the absorption and scattering coefficients of dyed samples respectively. The ratio of K/S values for samples can be expressed as the percentage when comparing the standard at the same wavelength [7]. For simultaneous mordanting samples, the dyed fabrics with NaCl as a mordant has the highest lightness (L^* , 73.41). For post mordanting method, the colour differences for NaCl mordants samples showed that the dyed fabrics with post-mordanting was darker (L^* , 68.87) as compared to the controlled dyed fabric with white vinegar and potassium aluminium sulphate mordant. The controlled dyed fabric with white vinegar mordant has high a^* while the dyed fabrics with potassium aluminium sulphate as a mordant has high value of b^* .

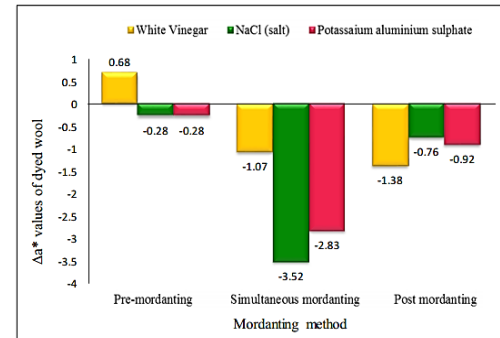
The K/S values as shown in Figure 4(d) for all pre-mordanting and post-mordanting are almost similar because the fabrics were being treated, with mordant, for the same duration and temperature, which were 30 minutes at 60°C (wool was dyed using the same method as dyeing natural dyes). Besides that, the fabrics were dyed first then mordanting as the method used above and rinsed in hot and then cold water and finally treated with mordant solution [8]. Nonetheless, natural dyed fabric with NaCl as a mordant in simultaneous mordanting has slightly higher K/S values as compared to others. This is because the NaCl in simultaneous mordanting helped to fix the colour and increases the depth of shade [9].

Table 1: Colour differences of L^* , a^* , and b^* , K/S values and shade between the mordanting method with different mordants dyed wool samples

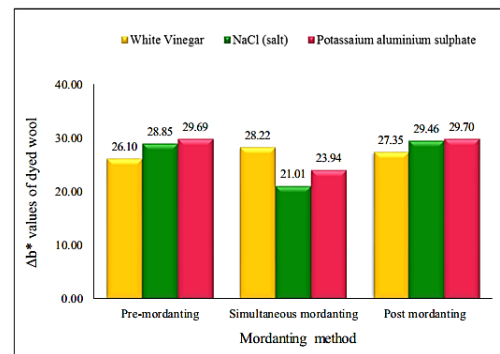
| Mordant used | Mordanting method | L^* | a^* | b^* | K/S |
|------------------------------|-------------------|-------|-------|-------|------|
| White Vinegar | Pre | 66.92 | 0.68 | 26.10 | 2.08 |
| | Sim. | 69.20 | -1.06 | 28.22 | 2.19 |
| | Post | 69.27 | -1.38 | 27.35 | 2.02 |
| Sodium Chloride (NaCl) | Pre | 65.60 | 0.28 | 28.85 | 1.89 |
| | Sim. | 73.41 | -3.52 | 21.01 | 5.49 |
| | Post | 68.87 | 0.76 | 29.46 | 1.95 |
| Potassium Aluminium Sulphate | Pre | 69.28 | 0.28 | 29.69 | 2.78 |
| | Sim. | 71.13 | -2.83 | 23.94 | 3.18 |
| | Post | 69.51 | 0.92 | 29.70 | 2.84 |



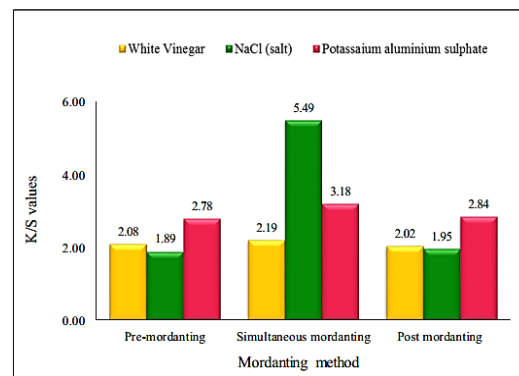
(a)



(b)



(c)



(d)

Figure 4: (a) ΔL^* values, (b) Δa^* values, (c) Δb^* values and (d) K/S values of dyed wool

3.2. Fastness Tests

For colorfastness to washing, the rating of colour fastness is given in Table 2. The wool fabrics recorded good to excellent (4 to 4-5) fastness grading for the three mordanting methods with different mordants. In the case of wool, change in colour results for NaCl samples showed poor rating with score of 3 in simultaneous and post mordanting dyed fabrics as compared to those of other mordant samples. This is due to the loss of colour because of the dyes were only held loosely by the fibre or held by weak forces such as

hydrogen bonds or Van der Waals forces [10]. The potassium aluminium sulphate mordanted fabrics recorded better results compared to fabrics from other mordants. Although it had the same average value with fabrics from white vinegar in the category of simultaneous mordanting fabric, but the value of fabrics from aluminium was more consistent. Thus, it can be said that alum is a better mordant for the pineapple leave dyeing process. Figure 5 shows the colour change rating for washing fastness.

Table 2: The colour change rating for washing fastness and colour staining to cotton and wool

| Mordant used | Mordanting method | Colour change | Staining (Wool) | |
|------------------------------|-------------------|---------------|-----------------|------|
| | | | Cotton | Wool |
| White Vinegar | Pre | 4 | 4/5 | 4 |
| | Sim. | 4/5 | 4 | 4 |
| | Post | 4 | 4/5 | 4/5 |
| Sodium Chloride (NaCl) | Pre | 3/4 | 5 | 4/5 |
| | Sim. | 3 | 4 | 4 |
| | Post | 3 | 5 | 5 |
| Potassium Aluminium Sulphate | Pre | 5 | 4/5 | 4 |
| | Sim. | 4/5 | 4/5 | 4/5 |
| | Post | 5 | 5 | 5 |

For colorfastness to light, the results indicate that for wool, dyeing along with NaCl gave good to very good (5 to 6) fastness grade and good to excellent (6 to 7) with potassium aluminium sulphate. Dyeing with potassium aluminium sulphate showed very good fastness with value of 6. The colourfastness samples to light in general have good rating values as shown in Figure 6. This is due to metal protecting the chromatophore from photolytic degradation. The substitution of dyes may increase the electron density around the reaction site of the molecule aiding oxidation, or it may reduce the electron density with a resultant increase in case of reduction which influence the result of light fastness. It can be seen that the structure of pineapple leaves having two hydroxyl groups, gives good fastness. Table 3 presents the rating of colourfastness to light for wool fabric.

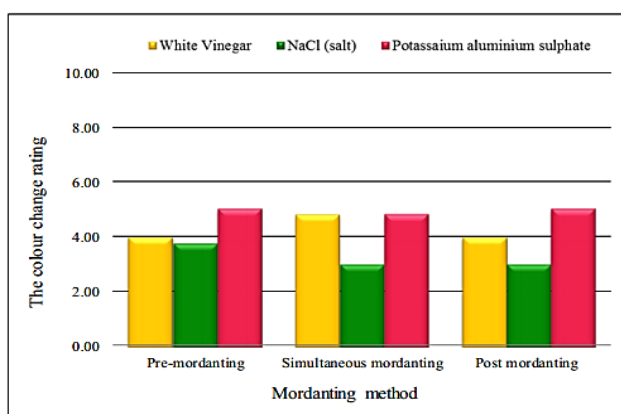


Figure 5: The colour change rating for washing fastness

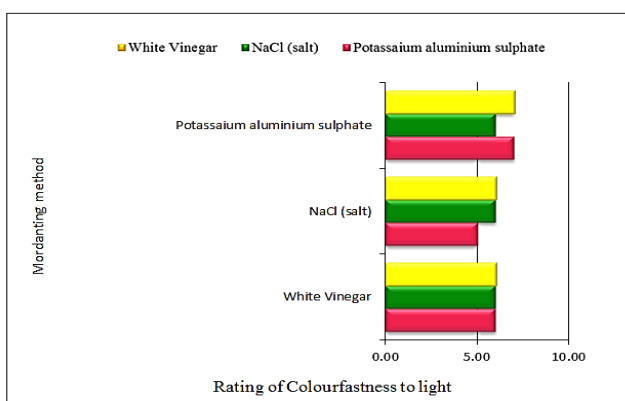


Figure 6: Rating of colourfastness to light for wool

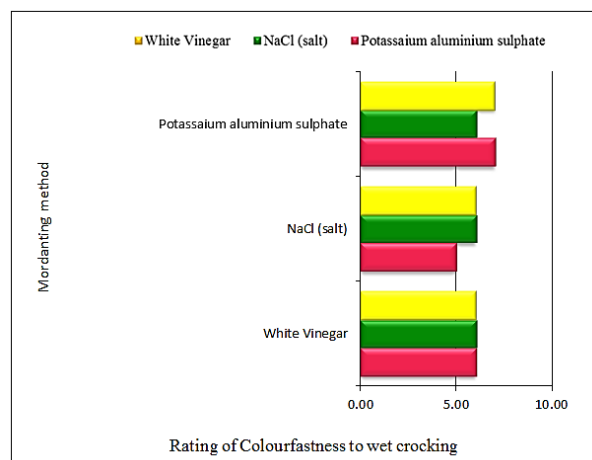
Table 3: Rating of colourfastness to light for wool

| Mordant used | Mordanting method | Rating values (Wool) |
|------------------------------|-------------------|----------------------|
| | | |
| White Vinegar | Pre | 6 |
| | Sim. | 6 |
| | Post | 6 |
| Sodium Chloride (NaCl) | Pre | 5 |
| | Sim. | 6 |
| | Post | 6 |
| Potassium Aluminium Sulphate | Pre | 7 |
| | Sim. | 6 |
| | Post | 7 |

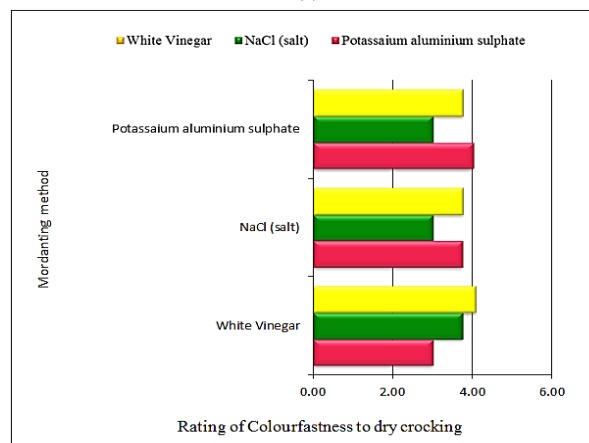
Colorfastness to crocking is used to determine the stability of the dyed color on the fabrics. Wet crocking fastness was found slightly better than dry crocking fastness (Table 4). All the staining rating of wet crocking fastness was ranged within 3/4-5. Samples dyed with pineapple leave extract by using potassium aluminium sulphate as a mordant with post-mordanting method have an excellent light fastness followed by NaCl. Figure 7(a) and (b) shows the rating value for wet crocking and dry crocking.

Table 4: The colour change rating for colourfastness to crocking

| Mordant used | Mordanting method | Rating values | |
|------------------------------|-------------------|---------------|-----|
| | | Wet | Dry |
| White Vinegar | Pre | 3/4 | 3 |
| | Sim. | 4/5 | 3/4 |
| | Post | 3/4 | 4/5 |
| Sodium Chloride (NaCl) | Pre | 4 | 3/4 |
| | Sim. | 4/5 | 3 |
| | Post | 4/5 | 3/4 |
| Potassium Aluminium Sulphate | Pre | 4/5 | 4 |
| | Sim. | 4/5 | 3 |
| | Post | 5 | 3/4 |



(a)



(b)

Figure 7: The colour change rating for colourfastness to (a) wet crocking and (b) dry crocking

Different shades of colour were obtained with dyeing of the leaves of *Ananas Comosus* on wool fabrics, with different mordant and mordanting methods as shown in Table 5 and Figure 8. Dyeing with and without mordants yield yellowish colour, thus it could be said that pre-mordanting with white vinegar and potassium aluminium sulphate was not a good method.

Table 5: The various shades obtained from mordanting method with different mordant

| Mordant used | Mordanting method | Colour shades |
|------------------------------|-------------------|-----------------------|
| White Vinegar | Pre | Yellowish green |
| | Sim. | Light yellowish green |
| | Post | Light yellowish green |
| Sodium Chloride (NaCl) | Pre | Light yellowish green |
| | Sim. | Light yellowish green |
| | Post | Light yellowish green |
| Potassium Aluminium Sulphate | Pre | Yellowish green |
| | Sim. | Light yellowish green |
| | Post | Light yellowish green |

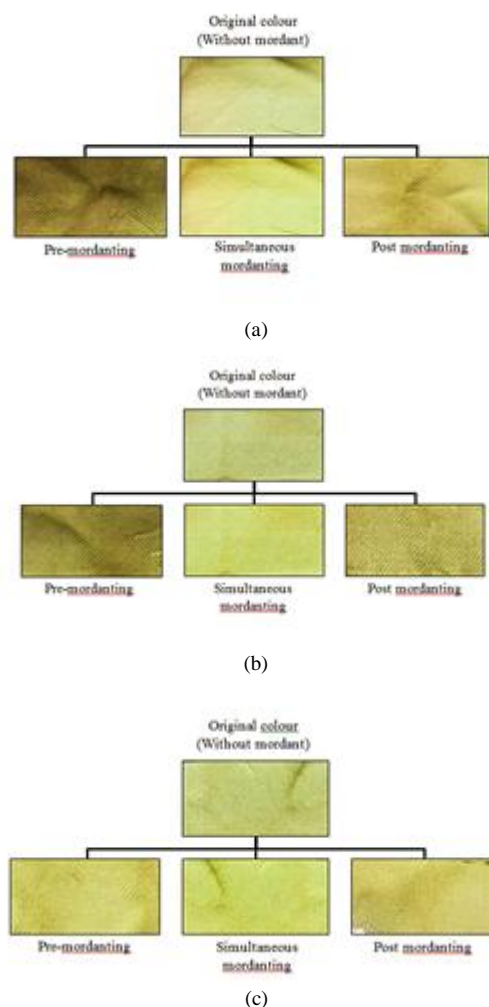


Figure 8: The various shades obtained from mordanting method with different mordant (a) potassium aluminium sulphate, (b) NaCl (salt) and (c) white vinegar

4. Conclusion

The methods of mordanting on wool fabric and three different mordants had shown certain result on the spectrophotometer and colourfastness to washing. For the spectrophotometer, the fabrics with three stage mordanting method with all mordant showed a trend of yellow shades and had stronger colour strength. Thus, it may be concluded that the mordanting method had turned the green pigment of the pineapple leave into yellow colour. Alum

had shown consistent result but the shades of the colour were very pale. White vinegar shows good result on both types of the fabrics in terms of brighter colour shades. NaCl was not a suitable mordant as it yields very inconsistent result. In terms of the colourfastness to washing, the untreated fabrics with three different mordant did not perform better than the treated fabric. This may be due to the colour strength of treated fabrics were stronger, thus the difference of colour before and after washing process was larger than the untreated fabric. On the other hand, potassium aluminium sulphate had shown a better colourfastness in three types of mordanting method. It also had less staining effect to the wool fibres. Thus, it is a most appropriate mordant to be used in pineapple dyeing process.

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