



Extraction of Natural Dyes from *Delonix regia* Flowers for Cotton Fibres

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Abstract

Textile colouration is among the major industry that pollutes the environment with effluents rich in dye chemicals. Most of the dyes used are synthetics which possess health and environmental impacts. Potentially, the use of bio-based dyes and other chemicals can lessen the environmental challenges of the textile effluents. This study reports on sustainable extraction and dyeing of cotton fabric using extract of *Delonix regia* flowers. The extraction process was done using acidified water as a solvent and the dyeing process was free from hazardous auxiliary chemicals. To improve the dyeing performance, banana sap was used as an affordable and eco-friendly bio-mordant. To optimise energy consumption, dyeing experiments were done between room temperature and 90 °C. The dyeing performance was assessed through wash fastness, rub fastness and colour strength tests. The cotton fabric was successively dyed with the *Delonix regia* flowers extract rich in anthocyanin pigments in the presence of banana sap bio-mordant. The optimum dye performance was obtained at a dyeing temperature of up to 60 °C, above which the performance was negatively affected.

Keywords: Textiles dyeing; *Delonix regia*; Banana sap

Introduction

Natural dyes extracted from plants have been used since ancient times before the invention of synthetic dyes. However, the use of natural dyes for textile applications suffered from performance and economic challenges due to the poor technology associated with the extraction and application. Despite the challenges, natural dyes have fewer negative environmental effects compared to their synthetic counterpart (Nambela et al. 2020). Efforts to overcome the challenges have been reported. Different extraction methods for improving the yield and quality of the natural dyes have been reported (Saxena and Raja 2014, Iqbal and Ansari 2021). Research has been done to improve the natural dye's performance, and search for advanced dyeing methods and techniques such as the use of different mordants (Pizzicato et al. 2023). Despite the efforts

made, most of the reported methods still face challenges in terms of capital investment, energy consumption and non-environmentally friendliness. Most of the non-conventional extraction methods are capital-intensive with high operational costs. On the other hand, the use of metal salt mordants such as alum, potassium dichromate, ferrous sulphate, copper sulphate and stannic chloride to improve the performance of natural dyes results in similar health and environmental challenges as synthetic dyes. In this regard, the environmental challenges of the dyeing industry can be lessened by the use of environmentally friendly mordant. Tannins are polyphenolic compounds which can easily form hydrogen bonds with other compounds through their -OH and -COOH moieties. Therefore, as a natural mordant, tannins bind to natural colourants such as anthocyanins and fibres such as cellulose by

forming hydrogen bonds with the OH groups of the fibres. Thus, tannins can act as an interface to improve the affinity between natural dye and fibre (Figure 1).

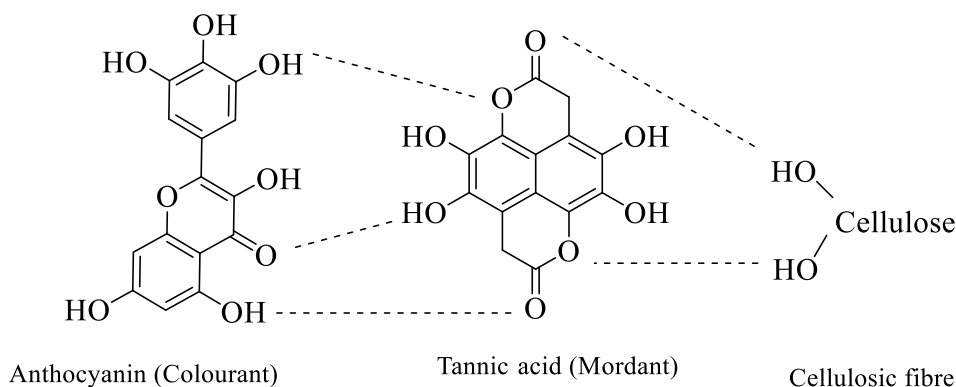


Figure 1: Interaction between anthocyanin colourant, tannin and cellulosic fibre

Delonix regia commonly known as flamboyant, royal poinciana or flame tree is a large flowering plant grown in tropical and subtropical countries across the world. The tree produces brilliant red-orange flowers. Anthocyanins and carotenoids are the pigment responsible for the colour of the flowers. The extraction, isolation and identification of carotenoids and anthocyanins from *Delonix regia* have been reported elsewhere (Veigas et al. 2012). The literature shows that the amount of anthocyanins in the petal of fresh *Delonix regia* red flowers is 8.3 g per 100 g dry flowers (Veigas et al. 2012).

The dyeing properties of anthocyanin pigments extracted from different plants including *Delonix regia* flowers have been studied on cotton (Phan et al. 2020, Wang et al. 2016, Shanker and Vankar 2007), wool (Haddar et al. 2018, Adeel et al. 2022, Shanker and Vankar 2007) and silk fibres (Vankar and Shukla 2011, Yasukawa et al. 2017, Haddar et al. 2018, Shanker and Vankar 2007). However, most of the literature reported the extraction methods which involve high energy consumption and the use of petroleum-based solvents. In addition, textiles dyed using the extracted pigments showed unsatisfactory performance due to the poor affinity. The use of mordants such as iron sulphate, copper sul-

phate, zinc sulphate, alum and tannic acid improves the dyeing properties as reported elsewhere (Wang et al. 2016, Sonali et al. 2022, Shahmoradi et al. 2021). However, these conventional mordants are expensive and environmentally not friendly.

This paper reports sustainable pathway of obtaining anthocyanin rich extract from *Delonix regia* flowers for eco-friendly dyeing of cotton fabric.

Materials and Methods

Materials

Bleached and mercerised (100%) cotton fabric (156 g/m²) was supplied by Tanzania-China Friendship Textile Company Limited (URAFIKI), Dar es Salaam, Tanzania. The bio-mordant used was tannin rich banana sap extracted from banana tree stem. Fresh *Delonix regia* flowers and banana tree stem were collected from the gardens at the University of Dar es Salaam, Mwalimu Julius Nyerere, Campus. Hydrochloric acid (36% analytical reagent – Sigma-Aldrich) was used to acidify the dye extract.

Methods

Extraction of dyestuff

The dyestuff, anthocyanin rich extracts, were obtained from fresh *Delonix regia* flowers. The fresh flowers (20 g) were grounded

thoroughly using a motor and paste then filtered using a Buchner funnel through a filter paper in *vacuo*. Since anthocyanin pigments are stable in an acidic medium, acidified water (40 mL, pH 2) was used to wash the residues to remove any remaining extracts. Then the combined filtrates were kept in the dyeing beaker.

Dyeing procedure

For 60 minutes, flower extracts were used to dye cotton fabric both with and without mordant and without adjusting the pH. The ratio of fabric to dye extract was 1:20. To obtain the optimum dyeing temperature, the dyeing was done at 40, 50, 60, 70 and 90 °C. The dyed fabric was washed with cold water and dried at room temperature.

Mordanting procedure

Pre-mordanting was carried out by exhausting undyed woven cotton fabric in bio-mordant for 60 minutes. The ratio of the amount of fabric to the volume of the mordant was 1:10. To optimize the temperature, mordanting was done at 40, 50, 60, 70 and 90 °C for 60 minutes. For post mordanting process, a similar procedure was done on a dyed cotton fabric.

The co-mordanting was carried out during dyeing at bio-mordant to the dye extract ratios of 1:1, 1:2, and 2:1. During co-mordanting the dye solution was prepared by mixing 20 mL of the dye extract with the required volume of bio-mordant to make up to 1:1, 1:2, and 2:1 dye: mordant ratio. The ratio of fabric to liquor was 1:40, 1:60 and 1:30.

Colour measurement

The colour strength (K/S) was determined using the Kubelka-Munk equation ($K/S = (1 - R)^2 / 2R$), where K is the light absorption coefficient, S is the scattering coefficient and R is the reflectance.

Colour Fastness to Washing

The colour fastness of the dyed cotton fabric against washing was assessed following ISO 105 C06 standard. The evaluation was

conducted using greyscale according to ISO-05-A02 to determine the degree of change in shade depth of the stained fabric. A rating of 5 indicates the lowest change, while a rating of 1 signifies the highest change.

Colour fastness to rubbing

The colour fastness of the dyed fabric against wet and dry rubbing was assessed according to AATCC test method 8- 1996 using a manually operated SDL Atlas crockmeter. The visual assessment was done using grey scales for staining. The grey scale rating system was similar to the colour fastness to the washing test.

Results and Discussions

The *Delonix regia* flower extract obtained by using acidified water solvent was red. The literature suggests that *Delonix regia* flowers contain polyphenols, anthocyanins (main component), carotenoids and other flavonoids. The aqueous extraction method used in this work favours the anthocyanins. Therefore, it is speculated that the Ultraviolet (UV-Vis) absorption maxima (Figure 3) is due to the presence of anthocyanin in the extract. Similar findings have been reported in the literature (Skaar et al. 2014, Godibo et al. 2015). Literature shows that the colour stability of isolated anthocyanin largely depends on storage temperature, solvent type and pH. The red colour which is the stable form of anthocyanins is favoured at high flavylium concentrations when the anthocyanin functions as a weak acid (at acidic pH) (Ekici et al. 2014). Therefore, the colour of anthocyanin-rich extract depends on the pH of the solvent, red colour being the stable form. Most of the literature reported on the extraction of anthocyanins using acidified protic organic solvents such as ethanol and methanol. However, the organic solvents are not sustainable. This suggests that acidified water can be used for the extraction of anthocyanin dyes, hence overcoming the shortcomings of the organic solvents. In this regard, acidified water is considered a sustainable solvent.

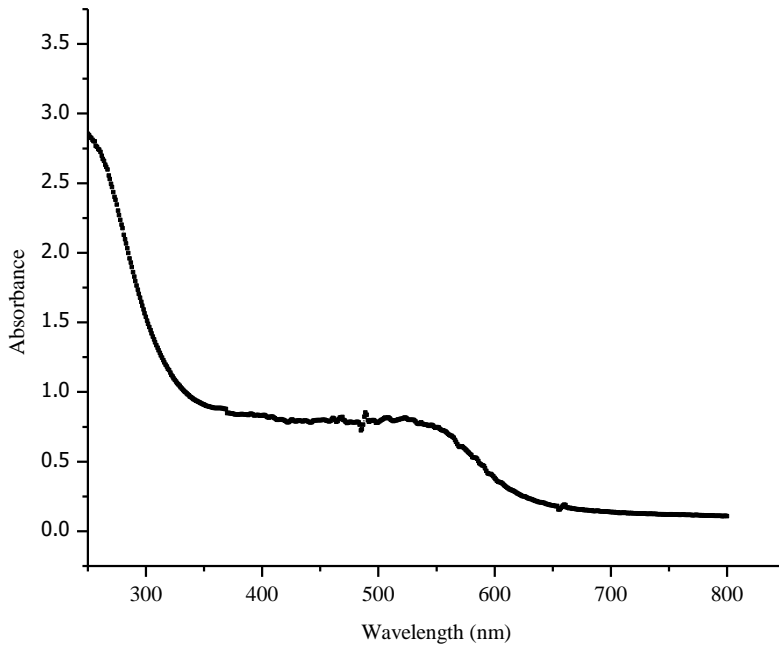


Figure 3: UV-Vis spectrum of *Delonix regia* flower extracts

Assessment of the dyed fabrics indicates that the non-mordanted had a lighter shade than the mordanted fabric (Figure 4). This suggests that anthocyanins have a poor affinity to cotton fabric. Apart from using mordant, the affinity of the dye can be improved by modifying the fabric surface or the dye structure.

Yet, most modifications involve the use of non-green chemicals. Therefore, the bio-mordant used in this study is green compared to the other available methods for improving the dyeability of cotton using anthocyanins.



Figure 4: Shades of (a): unmordanted dyed fabric; (b): mordanted dyed fabric

Color Fastness to Washing

Findings on the effects of mordanting and temperature on wash fastness indicate that in the absence of mordant, the grey scale rating was 1, regardless of the dyeing temperature, Figure 5. This implies that in the absence of mordant, the wash fastness of the cotton fabric dyed with anthocyanin-rich extract is poor.

However, the grey scale rating of the fabric dyed with mordant was 2-4, which suggests that the wash fastness of the cotton fabric dyed with anthocyanin-rich can be improved by the use of banana sap bio-mordant. The characterisation of pseudostem banana sap has been reported elsewhere (Akpabio et al. 2012). Apart

from tannin, studies show that pseudostem banana sap contains some colourless flavonoids which have less/no contribution to dyeing and mordanting (Gupta et al. 2022).

Further examination of Figure 5 indicates that the grey scale rating of the pre-mordanted was higher compared to post post-mordanted fabric. The grey scale rating for the pre-mordanted fabric was increased remarkably with temperatures up to 60 °C, thereafter it decreased. This implies that the wash fastness of the fabric dyed with anthocyanin-rich extract decreased with an increase in dyeing temperature up to 60 °C. A similar trend was observed for the mordanted post-mordanted fabric but at

a marginal rate. This marginal increase is because under pre-mordanting, the presence of the mordant on the fabric increases the number of potential sites for hydrogen bonding between the fabric and dye. While under post-mordanting, most of the sites for the formation of hydrogen bonds are occupied by the dye, hence the limited possibility of the mordant contributing to the interactions between the dye and the fabric. The decrease in wash fastness with an increase in dyeing temperature at a temperature above 60 °C is due to the instability of the anthocyanin.

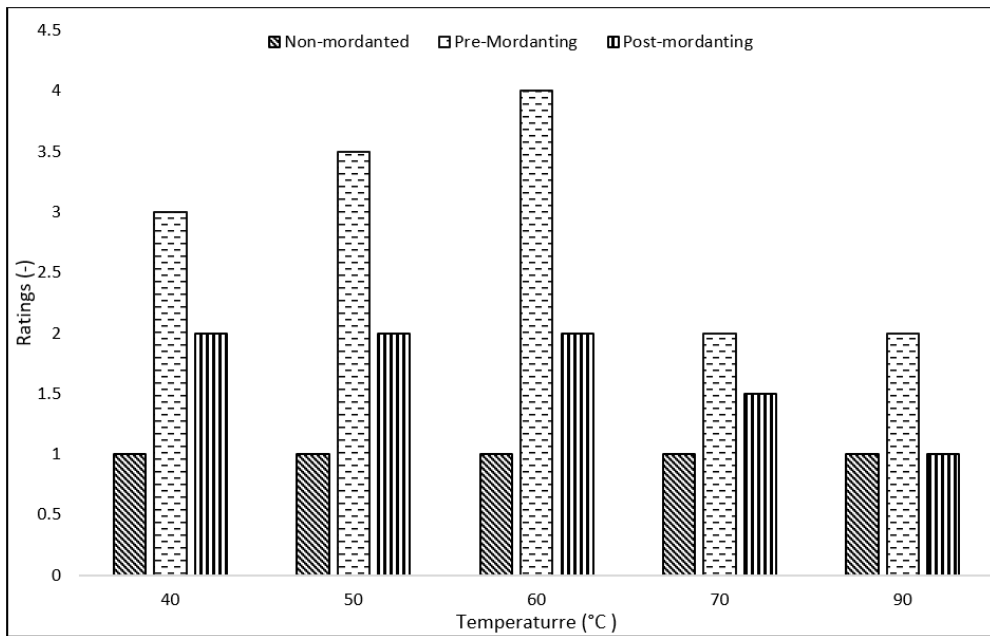


Figure 5: Effects of temperature and bio-mordant on wash fastness

Findings on the effects of the amount of bio-mordant indicate that at a temperature less than 60 °C the wash fastness was higher when the dye to bio-mordant ratio was 1:2. Whereas at 60 °C the 2:1 ratio gave the highest wash

fastness results. Poor results were obtained at equal amounts of dye and bio-mordant (Figure 6). This trend is due to the effect of dyeing temperature as just explained.

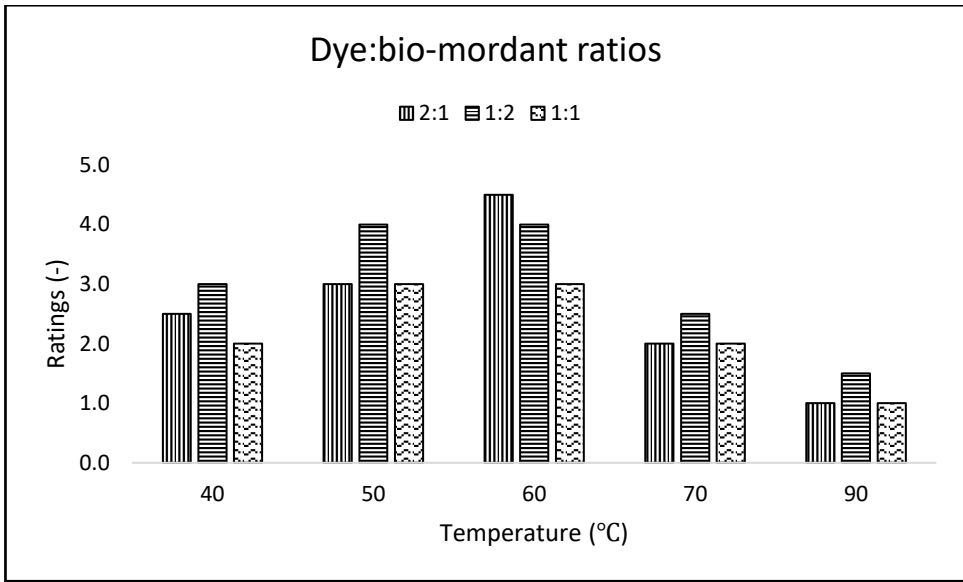


Figure 6: Effects of amount of bio-mordant on wash fastness

Colour Fastness to Rubbing

Findings on the effects of mordant on colour fastness to rubbing indicate that in the absence of the bio-mordant both wet and dry rub fastness ratings is very low, Figure 7. However, the colour fastness to rubbing increases

with an increase in dyeing temperature up to 60 °C above which it decreases due to the instability of the anthocyanin as just explained.

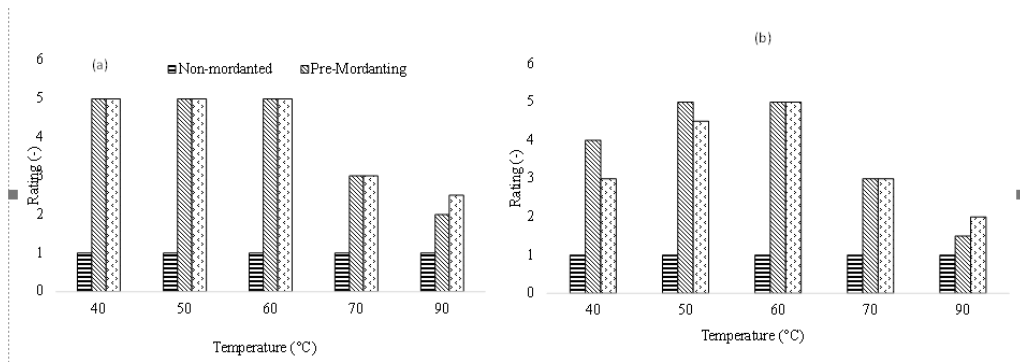


Figure 7: Effects of temperature and bio-mordant on (a) dry rub fastness (b) wet rub fastness

Evaluation of Colour Strength

Findings on the effects of temperature on the colour strength, Figure 8 indicate that the colour strength values of the mordanted were

higher than the non-mordanted fabric regardless of the dyeing temperature. Further observation on the effects of temperature indicated that the colour strength values were increasing with an increase in temperature up to 60 °C with a subsequent decrease above 60 °C, which is similar to the trend observed under the assessment of the fastness properties.

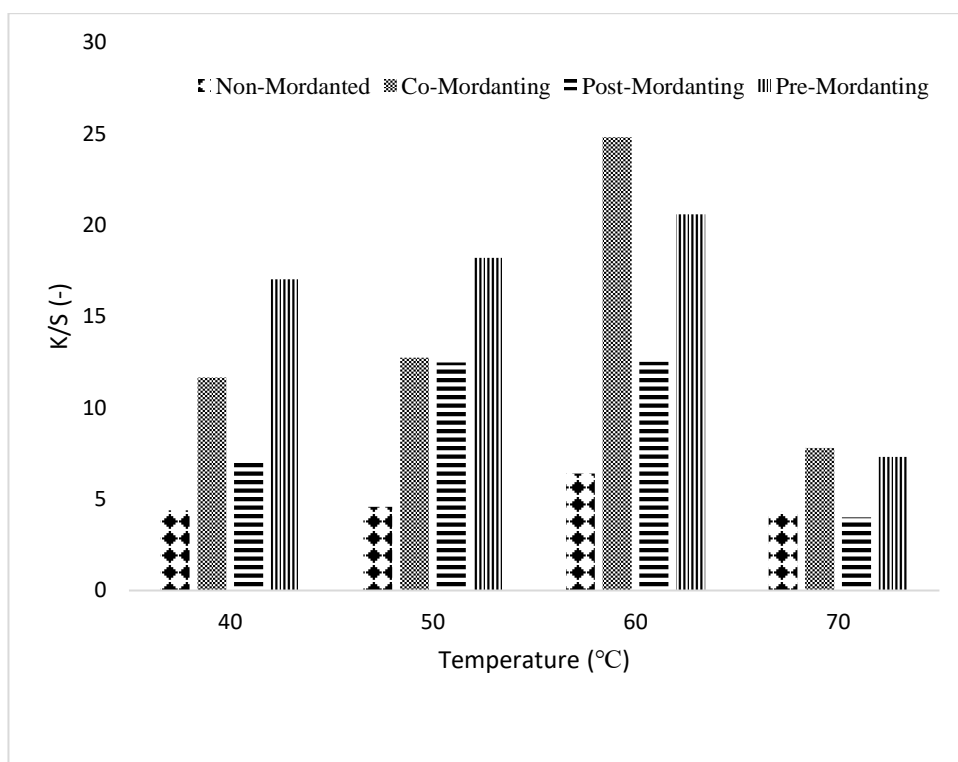


Figure 8: Effect of mordanting type on colour strength of the dyed cotton fabric

Conclusions

To conclude, the anthocyanin-rich extract was successfully extracted from *Delonix regia* flowers by using acidified water as a solvent. This suggests that water can be used as an alternatively sustainable solvent for the extraction of dyes. From the study, it has been established that the cotton fabric was successively dyed with the anthocyanin-rich extracts and the dye performance improved by green bio-mordant banana sap. The results further indicated that the optimum dye performance was obtained at a dyeing temperature of up to 60 °C, above which the performance was negatively affected. Therefore, sustainable dyeing of cotton fabric using *Delonix regia* flowers is possible. To promote the green textile industry, further research on the optimisation of extraction of anthocyanin dyes from plants and dyeing parameters is required.

Declaration of Competing Interest

The authors declare that there is no conflict of interest regarding this work.

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