Evaluation of dyeing properties of natural dyes extracted from the heartwood of *Prosopis juliflora* on cotton fabric

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Mark Peter Odero
Department of Chemistry and Biochemistry, Moi University, Kenya and African Centre of Excellence in Phytochemicals Textile and Renewable Energy, Eldoret, Kenya

Ambrose Kipchumba Kiprop
Department of Chemistry and Biochemistry, Moi University, Kenya and African Centre of Excellence in Phytochemicals Textile and Renewable Energy (ACE II), Eldoret, Kenya

Isaac Odhiambo K'Owino
Department of Pure and Applied Chemistry, Masinde Muliro University of Science and Technology, Kakamega, Kenya

Milton Arimi
Department of Chemical Engineering, Moi University, Kenya, and

Scolastica Manyim
Department of Chemistry and Biochemistry, Moi University, Kenya

Abstract

Purpose – Considered as one of the most unwanted species by the International Union for Conservation of Nature, the *Prosopis juliflora* plant is a noxious and invasive weed. Recent studies suggest that the heartwood of the plant has an unusually high amounts of flavonoids with potential medicinal properties and dyeing potentials. In this work, acetonic extracts were successfully valorized into a natural dye.

Design/methodology/approach – After extraction and optimization of dyeing conditions, the fabric was treated by using pre-mordanting, simultaneously mordanting and post mordanting techniques. The dyed samples were then evaluated by using standard methods ISO 105-C06, ISO 105-A02, ISO 105-X12 for wash, light and rub fastness, respectively.

Findings – Dye fastness ranged from good to very good with mordants improving both wash and rub fastness. Optimum pH for dyeing was found to be at 7.2 at temperatures of 60°C and a dyeing time of 80 min. Application of mordant in dyeing white fabric showed improved dyeing properties in post mordanting than both pre-mordanting and simultaneous mordanting.

Competing interests: The authors declare that they have no competing interests.

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Originality/value – To best of the authors' knowledge, this paper details for the first time how a noxious weed can be transformed into a natural dye, with potential applications to the textile industry.

Keywords Natural dye, Mordant, Colour strength, Colour fastness

Paper type Research paper

Introduction

The history of the dyeing industry dates back to more than 4,000 years ago, where primitive forms of colourations existed. For a long time, these dyes came from different parts of plants, such as roots, flowers, leaves, barks, selected rocks and insects. This, drastically changed after the year 1856, when the first synthetic dye was produced by William Henry Perkins who synthesized a dye named mauveine as he was attempting to synthesize quinine commonly used for the treatment of malaria (Benkhaya *et al.*, 2020). The dye was cheap to produce, reliable in shade, had a brilliant colour and had good colour and light fastness. His discovery opened doors for more research into the world of synthetic dyes and thus they quickly overtook natural dyes as the main sources of dyes. Currently, the number of known dyes exceed the 100,000 mark with a production exceeding 7×10^{5} worldwide (Bhuiyan *et al.*, 2017; Dwivedi and Tomar, 2018).

Presently, natural dyes have been linked to environmental pollution, allergies, skin cancer and other forms of cancer (Carmen and Daniela, 2012). This has led to an increase in research on the effectiveness of natural dyes and their potentials in different types of fabric (Bhuiyan *et al.*, 2017). The use of natural dyes has been discovered not only to be soothing to the eyes but also to be having unique properties such UV protection, antimicrobial, antifungal and antibacterial finishes (Sarwar *et al.*, 2018). In spite of these advantages, most natural dyes have poor colour and light fastness and, in most cases, will need mordants to improve their dyeing properties. Mordants are special binders of either synthetic or natural origin that are able to on one hand bind to the fibre while on the other hand also bind to the dye, thus making the dyeing process to be effective and colourfast.

The *Prosopis juliflora* plant is an invasive plant that can grow to heights of about 12 mwith a trunk of up to 1 m if grown in favourable conditions. Over the years, environmentalists have raised concerns about the *Prosopis* plant mainly because of its ability to colonize large tracts of the land including arid and semiarid land, sometimes leading to loss of biodiversity (Maundu *et al.*, 2009). Apart from this, when the pods of the plant have been ingested by sheep and goats, they affect the dental and respiratory tract in some cases causing death with the thorns also causing injuries that take an extremely long time to heal. Although the plant is noxious and invasive, previous work conducted on how to valorize the plant showed that the plant has a lot of phytochemicals present in different parts of the plant; hence, it has potential medicinal value. The heartwood of the plant is of specific interest which has been shown to be specifically rich in phytochemicals with an unusually high amounts of flavonoids such as mesquitol, epicatechin and catechin. This suggests that the plant can be exploited for medicinal reasons such radical scavenging properties and for its potential dyeing properties (Odero *et al.*, 2017; Sirmah, 2019; Sirmah *et al.*, 2011).

The presence of flavonoids such as catechin, epicatechin, gallo-catechin, mesquitol and other derivatives of flavonoids have already been established to be present in the acetonic extracts of the heartwood of this plant (Chepkwony *et al.*, 2020; Odero *et al.*, 2017). This was of specific interest, given that it has been previously proven that natural dyes that yield the colours yellow and brown are usually composed of flavonoids and flavonoid derivatives such as luteolin, catechin, fisetin, myricetin. Figure 1 shows the structures of some flavanoids extracted from the heartwood of *Prosopis juliflora* that are suggested to be responsible for its dyeing properties.

In this paper, we report for the first time, the successful valorisation of the acetonic extracts of the heartwood of the *Prosopis juliflora* plant into a natural dye of reliable fastness and varying shades based on selected mordanting techniques.

Materials and methods

Chemicals and cotton

All chemicals used in this study were of analytical grade, manufactured by Sigma Aldrich and supplied by Kobian and Gelsap laboratories Nairobi, Kenya. They included Alum [(K_2SO_4.$Al_2(SO_4)$.24H_2O], ferrous sulphate ($FeSO_4$), potassium dichromate ($K_2Cr_2O_7$) and Tin Chloride ($SnCl_2$) which were used as synthetic mordants. Pure white scored and mercerized cotton were sourced from Rivatex East Africa Limited, a facility of Moi University, Eldoret, Kenya.

Collection of plant materials

The *Prosopis juliflora* plant samples were collected from Marigat, Baringo County in Kenya and transported to Moi University for storage awaiting further processing.

Extraction of dyes from Prosopis juliflora

The extraction method was adopted from Sirmah (2018). The heartwood was first separated from the other parts of the trunk and chipped into smaller pieces and finally ground into fine powder using a hammer mill. The powder was then dried at 60°C until a constant weight was achieved. Approximately 500 grams of ground powder of the heartwood was soaked in 1.5 litres of acetone in a 2 litres beaker for approximately 48 h with occasional physical agitations at room temperature. It was then filtered using Whatman filter paper No. 1 and subsequently rota vapoured and finally evaporated to dryness at room temperature.

UV-VIS spectrum of the dye

The UV-VIS spectrum of the dye was measured using a Beckman single beam Coulter Model DU^{R}720 UV-VIS machine between the wavelengths of 200–800 nm.

Optimization of parameters used for dyeing

The following conditions were optimized: pH, dyeing time, dye concentration and dyeing temperature. For all cases, except when the parameter was being optimized, they were held constant, i.e. the dyeing temperature was 60°C, material to liquor ratios were 1:20, dyeing time was 1 h,and the concentration of the dye was 20 g/L at pH 7.2 (Islam *et al.*, 2016; Kundal *et al.*, 2016).

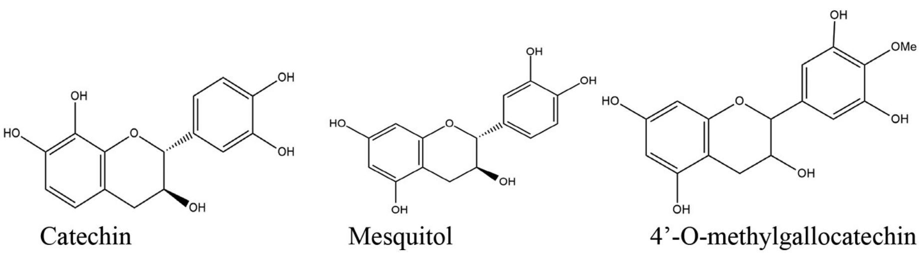


Figure 1. Suggested colouring compounds of *Prosopis juliflora*

Optimization of dye concentration

With all the other variables held constant, the dyeing concentrations were varied as follows: 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L and 50 mg/L. The dyed clothes were then washed for excess dyes to run off and then dried in a shade before their colour strengths were analyzed.

Optimization of dyeing pH

As all other factors were held constant, the pH of the dye was varied at 3.0, 4.2, 7.1, 8.0 and 9.1. The pH was adjusted to the desired value by using HCl and KOH. The dyed fabrics were washed, dried in a shade and evaluated for their dye colour strength.

Optimization of dyeing temperature

Five experiments were conducted to determine the colour strengths at temperatures 25°C, 40°C, 60°C, 80°C and 100°C with all the other factors held constant and the results recorded.

Optimization of dyeing time

With all the other factors held constant, the dyeing times were varied from 10 , 20 , 30 , 40 , 50 to 60 min and the results were recorded.

Dyeing methods

With the exception of the fabric used as a standard, all the other fabrics were either pre-mordanted, simultaneously mordanted or post mordanted. The following four mordants were used: alum [($K_2SO_4.Al_2(SO_4)0.24\ H_2O$], ferrous sulphate ($FeSO_4$), potassium dichromate ($K_2Cr_2O_7$) and tin chloride ($SnCl_2$). The sample size of the fabrics used for the evaluation of mordanting and dyeing properties were kept at 10 × 10 cm. After dyeing, the pieces were then washed with a 1% non-ionic soap before they were rinsed and dried (Islam *et al.*, 2016).

Pre-mordanting

In this method, 0.5 grams of each mordant were dissolved in 100 ml of distilled water at temperatures of 100°C,maintaining the mordant ratios at 5 g/L. The fabrics were then dipped in the mordant solution for an hour and subsequently dipped into the dye bath solution for 1 h at a temperature of 60°C and later removed and dried in a shade (Islam *et al.*, 2016).

Simultaneous mordanting

A total of 0.5 g of mordant were weighed and dissolved in 100 ml of the dye bath to produce mordant concentration of 5 g/L. The dye and mordant were in the same solution and were simultaneously heated to a temperature of 100°C. The fabric was then added to the mixture and dyed for an hour. The fabrics were then removed and dried in a shade (Islam *et al.*, 2016; Janani *et al.*, 2014).

Post-mordanting

In this method, the sample fabrics were first dyed in the respective dye baths at 60°C for approximately 1 h. The dyed fabrics were then post mordanted for an hour and subsequently dried under a shade. The ratio of mordant to solvent was maintained at 1.20 at temperatures of 100°C (Islam *et al.*, 2016; Janani *et al.*, 2014).

Determination of colour fastness
The subsequently dyed samples were subjected to fastness tests based on selected ISO standard tests.

Fastness to washing
The fastness to wash of the dyed fabric were done according to ISO 105-C06:1994. Two pieces of fabric, fabrics the dyed one and another plain fabric were stitched together as per the specifications. The fabrics were then added to 5 g/L non-ionic detergent while maintaining the temperatures at 50°C, for 45 min and the material to liquor ratios at 1.50 in a Rycobel group SDL Atlas Launder-o-meter. The fabrics were then assessed for loss of colour by using a grey scale ISO 105 A02;1993 for change in colour (Yusuf *et al.*, 2015).

Fastness to sunlight
Thinly cut fabric approximately 1 cm by 8 cm, were inserted in a special test tubes and illuminated for 4 hours using 500 W, SDL ATLAS light fastness tester M237. The quality of fastness was assessed via a grey scale ISO 105 A02;1993 for loss of shade (Jihad, 2014; Kumaresan *et al.*, 2011).

Fastness to rub
The fastness to rub was done using a manually operated crock-meter (AATCC Model; M238 AA SDATLAS) as per ISO 105-X12:200.

Evaluation of colour strength and CIELAB
The colour coordinates for the dyed fabric were evaluated in form of CIE L*, a*, b* using an X-rite spectrometer SP60-EB05003377 illuminant D65-10 mode at three points and averaging them (Ding and Freeman, 2017). The Cie C and H° were calculated using equations 1 and 2 as follows (Ali and El-Mohamedy, 2011; Yusuf *et al.*, 2015):

$$\text{Chroma }(\mathrm{C}^*) = \sqrt{a^2 + b^2} \tag{1}$$

$$\text{Hue Angle}(\mathrm{h}) = tan^{-1\frac{b}{a}} \tag{2}$$

The colour strength was calculated using the Kubelka–Munk (K/S) equation defined in equation (3) below; where S refers to scattering, K the samples absorption and R the reflectance (Kundal *et al.*, 2016; Sarkar, 2004; Yusuf *et al.*, 2017).

$$\frac{\mathrm{K}}{\mathrm{S}} = \frac{(1-\mathrm{R})^2}{2\mathrm{R}} \tag{3}$$

Results and discussion

Evaluation of the effects of dyeing parameters on colour strength
The highest values of K/S usually suggest the most effective point of dyeing (Zubairu and Mshelia, 2015). All the points are captured in the Figure 2 below. Figure 2 a, b, c and d being for the optimizations of dye concentrations, pH, time and temperature respectively. As depicted in Figure 2a, as the dye concentration increased, so did the K/S value with the highest value being at 50 g/L and the lowest being at 10 g/L. This, is consistent with other literature which

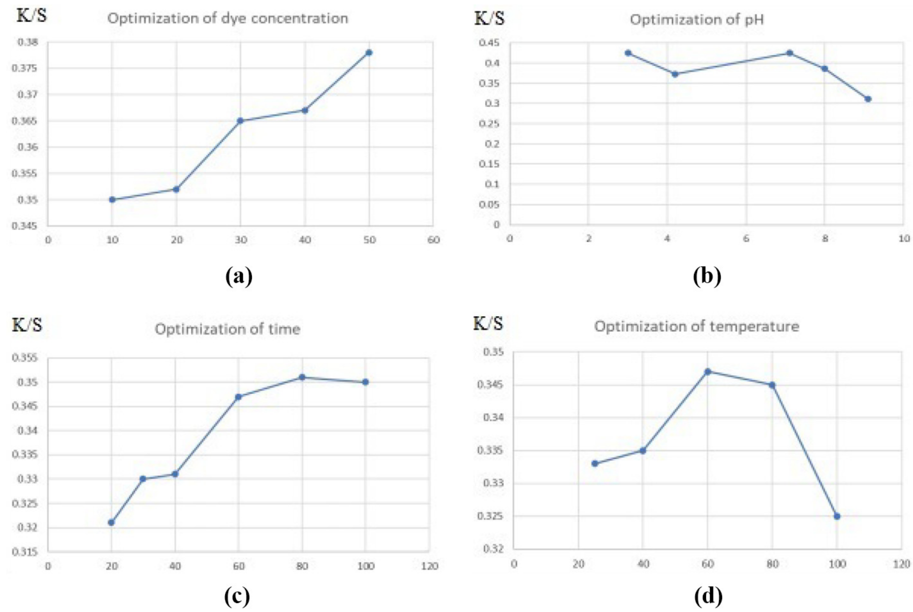


Figure 2. Graphs of K/S vs dye concentration (a), pH (b), time (c) and temperature (d)

explained the trend by suggesting that as the concentration of dyes increase, there is also an increase in the number of dyeing molecules. This implying that there are more dyeing molecules that can be bound to the fabric; hence, the higher the dye concentration, the higher the uptake of fabric in most cases (Paul *et al.*, 2017; Shahid *et al.*, 2016).

The graph of optimization of pH is also caputred in Figure 2B. In this case, the highest K/S values were at pH 3 and pH 7.2 and dropped thereafter. The pH 7.2 was thus chosen as optimum. The optimization of dyeing time was analysed for 20, 40, 60, 80 and 100 min, as in Figure 2C. This showing that as time increased, the colour strength of the dye also increased upto the 80th minute and remained fairly constant thereafter showing that the amount of time is directly proportional to amount of dye uptake upto a point where an increase in time no longer adds the intake (Paul *et al.*, 2017). This is because as temperature increases, so does the physical and chemical interaction between the dye and fabric until an equilibrium is established (Dayioglu *et al.*, 2015).

Optimization of temperature was done at temperatures 25°C, 40°C, 60°C, 80°C and 100°C (Figure 2D). The optimum temperature was at 60°C which had the highest K/S value this results are similarly mirrored in (Paul *et al.*, 2017). From the graph, it is seen that the colour strength increases as the temperature increases upto 60° where afterwards, the curve starts dipping. This is because, as the temperature increases, so does the molecular structure of the cotton fabric which unravels and opens it up, leading to an increased dye uptake (Paul *et al.*, 2017; Shahid *et al.*, 2016).

UV-VIS spectrum of the Prosopis juliflora dye

Figure 3 shows the UV spectrum of the extracts of the *Prosopis juliflora* dye. The dye extracts were brown in colour and had several points of absorption around 255 nm, 270 nm, 300 nm, 320 nm and 360 nm. This pointing to the probability of co-pigmentation because of several compounds being responsible for the dyeing properties. This is not unique to this dye, as previous brown dyes have also been reported to have multiple absorption peaks (Selvam *et al.*, 2015). For example, a brown dye from the *Trigonella foenum* plant, had absorptions of 259.2, 265.6, 358.4, 371.2 and 396.8 nm.

The peaks around 255 and 270 nm, are characteristic of flavonoid dyes and can therefore be attributed them (Yunus *et al.*, 2011). The suspected presence of flavonoids in the *Prosopis juliflora* can be corroborated by previous studies done on the heartwood of the plant that have shown the presence of unusually large amounts of mesquitol and other flavonoids (Chepkwony *et al.*, 2020; Odero *et al.*, 2017; Sirmah, 2019).

Fastness tests

The results of overall fastness properties, colour fastness, wash fastness and rub fastness were recorded and are detailed well in Table 1.

Wash fastness. From the Table 1 above, the overall wash fastness of the dye without any mordant was three which is considered fair. It can be suggested that the overall wash fastness properties of the dyes were improved by the presence of the various mordant. This

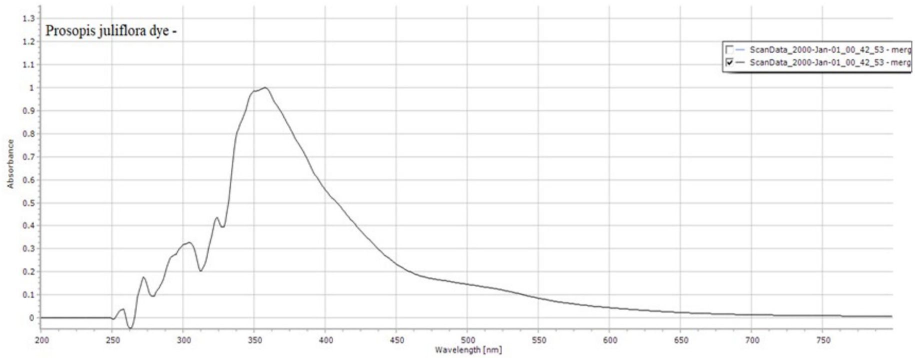


Figure 3. UV spectrum of the *Prosopis juliflora* dye

Method	Mordant	Wash *ISO 105-C06*	Light *ISO 105-A02*	Rub *ISO 105-X12* Dry	Rub *ISO 105-X12* Wet
Control/Standard	None	3	5	5	4
Pre-mordant	Alum	3/5	5	5	4
	$SnCl_2$	3/5	5	5	4
	$FeSO_4$	3/5	5	5	4
	$K_2Cr_2O_7$	3/5	5	5	4
Simultaneous Mordanting	Alum	4	5	5	4/5
	$SnCl_2$	4	4/5	5	4
	$FeSO_4$	4	5	5	4/5
	$K_2Cr_2O_7$	4	5	5	4/5
Post mordanting	Alum	5	5	4	4
	$SnCl_2$	5	5	5	5
	$FeSO_4$	5	5	5	5
	$K_2Cr_2O_7$	5	5	4	4

Notes: KEY 1: Very Poor; 2: Poor; 3: Fair; 4: Good; 5: Very Good

Table 1. The fastness properties (colour, wash and rub fastness) of the extract dye

is also reported in other literature (Kumbhar *et al.*, 2019; Lee and Kim, 2004). The most effective of the mordanting techniques was post-mordanting which improved the colour fastness of the dye significantly (5) with pre-mordanting and simultaneous mordanting techniques also improving the fastness properties of the dye.

Light fastness. From Table 1, it can be concluded that the overall light fastness property of the dye was not specifically improved by the presence of the mordants. This is because the value was already good at 5.

Rub fastness. The overall rub fastness for dry rub and wet rub are as in Table 1. It can be noted that he presence of mordants in some cases improved the rub fastness of the dye while in some cases it did not.

Colour strength (K/S)

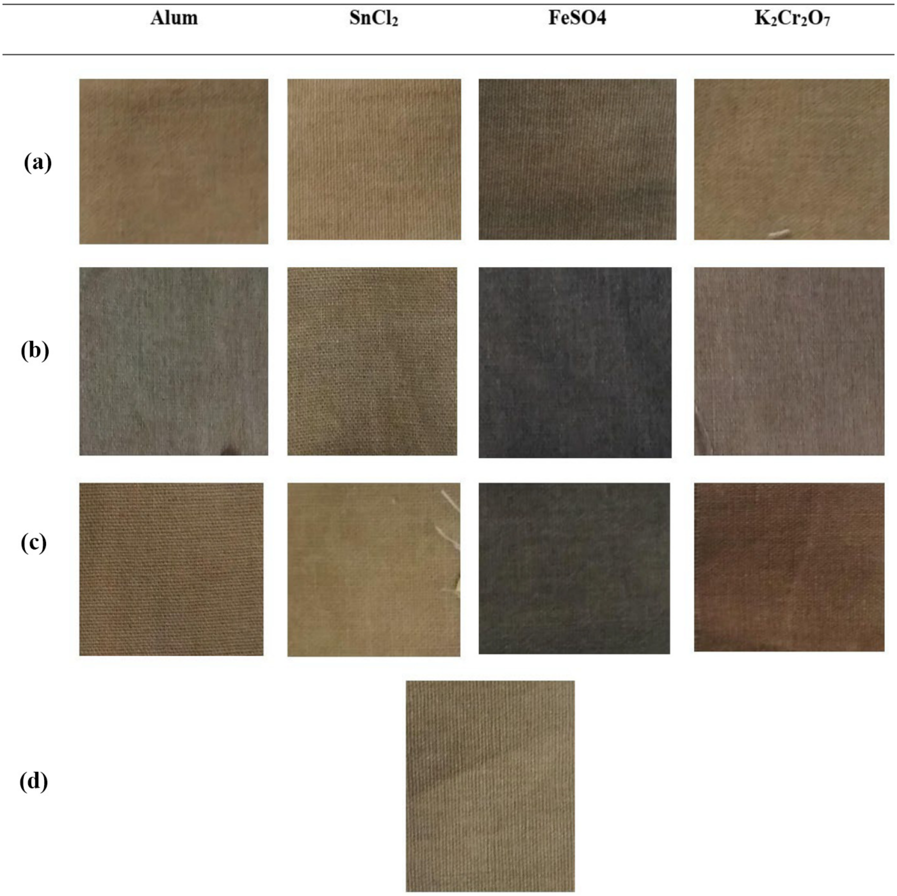
The summary of colour strength (K/S) of the dye extract from *Prosopis juliflora* plant is given in Table 2. Analysis of the K/S values suggest that the optimum mordanting techniques are between simultaneous mordanting and post mordanting techniques which have higher values of colour strengths as compared to pre-mordanting techniques. It can also be concluded that based on the difference between the colour strength and lightness (L) of the standard and the mordanted fabric, mordants generally increased the uptake of dyes on the fabric (Kumbhar *et al.*, 2019).

From Table 2, the lightness value (L) of the dye, did not show a particular trend although, there was a significant increase in colour strength when a mordant was used in the dyeing process. Post mordanting technique had lower L values suggesting that although the dye had a higher colour strength, they were duller. The effects of mordants was evident with the stannous chloride and potassium dichromate mordants, producing brighter shades tending towards a bright shade of brown and with ferrous sulphate dulling the shades to shades of green, this effects of the said mordants are similar to those reported by other studies of yellowish, brownish dyes (Janani *et al.*, 2014; Wanyama *et al.*, 2010). Figure 4 shows samples of dyed fabric, in combination with the differents and mordanting techniques, whereas Figure 5 further shows a schematic representation of cotton-mordant and dye complex.

The dulling of shades by $FeSO_4$, has previously been associated to the reaction of the ion into ferric form due the interaction with oxygen from the environment leading to a spectral

Method	Mordant	L	A	B	C	H	K/S
Standard	NONE	63.5	3.3	19.5	19.8	80.4	0.43
Pre-mordant	Alum	62.4	6.3	27.8	28.5	77.1	0.54
	$SnCl_2$	57.6	7.1	25.7	26.7	74.7	0.72
	$FeSO_4$	46.7	2.6	14.3	14.6	79.7	0.65
	$K_2Cr_2O_7$	63.6	5.5	27.1	27.7	78.5	0.63
Simultaneous mordanting	Alum	47.4	6.7	24.5	25.4	74.4	0.64
	$SnCl_2$	56.5	7.7	31.7	32.6	76.4	0.68
	$FeSO_4$	46.7	2.6	14.3	14.5	79.7	0.82
	$K_2Cr_2O_7$	55.5	8.1	22.3	23.7	70.0	0.54
Post-mordanting	Alum	58.4	7.7	23.4	24.6	71.8	0.58
	$SnCl_2$	65.0	4.0	29.6	29.8	82.2	0.44
	$FeSO_4$	44.1	1.86	9.2	9.4	78.6	0.86
	$K_2Cr_2O_7$	41.0	6.29	16.71	17.9	69.4	0.74

Table 2. Summary of the colour strength of the dye from the extract



Notes: (a) Pre mordant; (b) simultaneous mordanting; (c) post mordant (d) standard

Figure 4. Dyed fabric in combination with different fabric

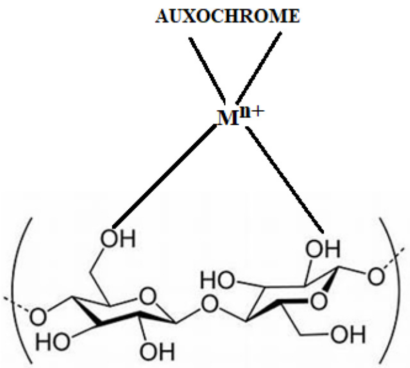


Figure 5. Schematic representation of potential cotton-mordant and dye complex

overlap between the Ferrous and the ferric forms existing together leading to a colour transition in this case dulling the colour (Janani *et al.*, 2014; Wanyama *et al.*, 2010).

Conclusion

Acetonic extracts of *Prosopis juliflora* were investigated for their dyeing properties with the results showing that it can satisfactorily be used as natural dyes that have reliable fastness to light, colour and rub. The effects of mordant on the dyeing properties of the fabric were clear with all the mordants increasing the colour strength of the dye to the fabric. Different dyeing conditions were optimized with post-mordanting techniques proving to be a better than the others. In this study, we have highlighted the potential of the dye extract from *Prosopis juliflora* as a natural dye with positive results.

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Corresponding author

Mark Peter Odero can be contacted at: miakitah@gmail.com