BIOSCIENCES BIOTECHNOLOGY RESEARCH ASIA, December 2011. Vol. **8**(2), p. 447-454

Extraction, Characterization and Application of Natural Dyes From Water Opepe (Nuclea diderricchi) and Water Ekimi (Vanilya Planofolia) on Natural and Synthetic Fibres

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(Received: August 25, 2011; Accepted: October 01, 2011)

Colours from naturally dyed fabrics recently have attracted both consumers and manufacturers attention in fashion fabrics. The extraction, characterization and application of natural dyes from water opepe (Nuclea diderricchi) and water ekimi (Vanilya planofolia) are reported. The dye extracts were applied as direct dyes and in the presence of mordants. The results of the wash fastness showed that the dye extracts have affinity for cotton, acrylic and nylon fibres used for the study, even in the absence of mordants. The presence of mordants, also further improved their fastness properties. The dye extracts were characterized by IR, UV-VIS and NMR analyses.

Keywords: Natural dyes, Textile dyeing, Mordanting, Natural and synthetic fibres, Fastness.

For the past 47 years or so, the use of natural dyes for the coloration of textiles has mainly been confined to craft dyers who have used these colorants for their own pleasure. However more interest has been shown in recent times, in the use of these dyes and limited number of commercial dyers and small businesses have started to look at the possibility of using natural dyes for coloration of both natural and synthetic fibres[1,2]. This trend has been fuelled by increased public awareness about environmental issues over the past decade, and more businesses, both large and small, have started exploring the use of natural colorants as a possible means of producing an ecologically sound product which would also appeal to the "green" minded consumer. The problem with this, however, is that to obtain a full colour range on natural and synthetic fibres for example, mordants must be used, and while the natural dyes are themselves generally harmless, the chemicals used for mordanting or fixing the dyes are generally not environmentally acceptable[3].

However, being a natural product does not necessarily mean being a safe product. The reality of course, is that in any group of products some will be good for you and others bad.

Many research articles[4-9] have been published dealing with re-establishment of traditional dyeing techniques, variability, approaches to the improvement of colour fastness or dyeing affinity for dyed fabrics, and identification of other functional properties such as antimicrobial activities. They are all part of

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possibilities to get good technical performance for natural dyeing.

Even though the current use of natural dyes for textiles is somewhat limited to small-scale industries and markets, their great potential to provide value-added products is encouraged by some noticeable advantages including better biodegradability and higher compatibility with the environment.

In the traditional natural dyeing of textiles, an important part of red/yellow dyes was formed by extraction of anthocyanin/flavonoid dyes from fruits and vegetables. An excellent overview of plant sources and application is given by Schweppe[10,11]. The extraction and quantification of anthocyanin dyes and phenolic components have also been studied in detail[16-17]. Gulrajani et al[18] studied the application of tannin-based natural dyes for dyeing of cotton.

A number of yellow/brown natural dyes from plants have also been extracted and applied to natural fibres[12-13].

The present study was carried out to extract colorants from the barks of two plants called water opepe and water ekimi. An attempt was also made by the author to utilize the extracted dyes for the dyeing of acrylic, cotton and nylon fabrics. The study also tried to identify the effect of mordants on naturally and synthetically dyed fashion fabrics since wool is gradually being replaced by acrylic fibres.

The determination of selected fastness properties (light-fastness, wash-fastness) as key properties for technical and commercial success were also evaluated.

MATERIAL AND METHODS

Extraction of plant dyes

The barks of water opepe and water ekimi were collected, dried and weighed. The dried plant barks were then ground into powdered form and treated with dimethyl formamide in the case of water opepe and ethanol in the case of water ekimi. The ratio of plant bark powder to volume of dimethylformomide or ethanol was set at 1:50, which means that 1.0 g of plant bark was extracted with 50 mL of dimethylformamide or ethanol according to the procedure[14]. The extraction was carried out at room temperature.

Dye Application

Nylon Fabric

Nylon fabric was scoured in a solution of 1.0 g/L of nonionic detergent (Lissapol) for 1 hour at 90^0C. Dyeing was carried out in a shaking water bath under the conditions indicated in Table 1.

The specific pH of the individual mordanting baths were potassium dichromate 6.6, Stannous chloride 6.1, Copper sulphate 6.8, and Ferrous
sulphate.

Acrylic fabric

The dyeing of acrylic material was carried out in a temperature controlled dyeing machine. The fabric samples were dyed according to the conditions shown in table 2 below.

Cotton Fabric

Scoured cotton fabric was dyed by an exhaustion method using a liquor ration of 1:20-22. For 1.0 g of goods, a dye bath volume of 20-22 mL was applied. The dyeing experiment was performed in a beaker at 95^0C dyeing temperature. After dyeing, unfixed dyestuff was removed by rinsing three times with cold water. The detailed dyeing procedure is given in the literature[15].

Mordanting

The mordants that were used are Potassium dichromate, tin (II) chloride, Copper (II) sulphate and iron (II) sulphate. A weighed amount of mordant was dissolved in water and stirred until a clear solution was obtained in each case. Dyed fabric samples were added to the bath and mordanting was carried out in a shaking water-bath as per the conditions in the tables as in the case of postmordanting.

In pre mordanting, the fabric was first treated with the metal salt and then dyed[16].

Instruments

The UV-visible spectra of the dye extracts were evaluated on a Genesys IOS. VL 200 series spectrophotometer. The infra-red spectra were determined by the use of Nicolet Averser, 300 series spectrophotometer. The melting points of the solid dye extracts were evaluated on a gallenkemp block apparatus and are uncorrected. Nuclear magnetic resonance spectra were recorded on the mercury 200 BB MHz spectrometer equipped with an oxford wide Bore magnet, sun 3/160 based computer with an array processor and GE Omega 6.0 software. Chemical shifts are given in δ values (ppm) with tetramethylsilane (TMS) as an internal reference standard.

Fastness Properties

Light Fastness

Light fastness of dyed fabric sample was evaluated by using a method[17] based on the standard procedure issued by ISO. Samples (2 x 6cm) were rated against standard blue wool samples (grade 1-8)

Wash ˘ Fastness

The wash-fastness of the dyed fabric samples was determined according to ISO washing test number 3 [18].

RESULTS AND DISCUSSION

Characterization of Dye Extracts

The ‘HNMR spectrum of the water opepe (Nuclea didierricchi) was characterized by two singlet peaks at 1.25-1.80 ppm which corresponds to the amine protons. Also the spectrum was characterized by a multiplet peak at 7.20-7.40ppm which correspond to phenyl protons. The ‘HNMR spectrum of water ekimi (Vanilya planofolia) showed two singlet peaks at 1.20-1.35 (ppm) assignable to amine protons.

The infra-red spectra (Table 3) of the dye extracts showed bands at 3441-3443 cm^{-1} region which are assignable to OH and NH groups. There is the presence of strong bands at 1800-1650 cm^{-1} regions indicating the presence of carbonyl group in the dye extracts. The band at 1629 – 1517 cm^{-1} suggests the presence of benzene ring. The band at 1102-1075 cm^{-1} region for the water ekimi dye extract is indicative of the presence of sulphur containing compounds. The small band at 2926 cm^{-1} for C%H stretch of aryl compounds further confirms the presence of benzene ring structures in the dye extracts[19-20].

The UV-visible spectral (Table 3) results of the dye extracts showed broad absorption curves in the range 380-800 nm with lmax of 413 nm (0.298) for water ekimi and lmax of 431 nm (0.325) for the water opepe. The area under the absorption curve is therefore large indicating that the two dye extracts have high tincterial strength [21]. The results of the melting point determination of the solid dye extracts gave 260^{0}C for the water opepe and 137-139^{0}C for the water ekimi dye extract. Thus, the heat stability of the water opepe dye extract is higher than that of the water ekimi. The relevant analytical data are shown in table 3

Light and Wash-fastness

Dyes are rather variable in their behaviour and their light-fastness depends on the depth of dyeing and on many environmental factors such as temperature and relative humidity around irradiated dyed fabric samples. A light fastness of at least 2-3 was set as a lower limit for positive selection, however, values of light fastness of 2 perhaps could be increased by further research activities to exceed the lower limit of 2-3 [22].

The light fastness of natural dyeing is strongly influenced by the type of mordant used. The light fastness grading confirm the earlier findings. Unmordanted dyed samples[23] had lower fastness to light whereas mordanted ones showed improved light fastness. The results in tables 4, 5 and 6 showed that light fastness of dyeing on acrylic fabric gave higher values when compared with those of nylon and cotton fabrics.

The results of the wash-fastness gradings showed good performance on the three substrates used for the study. In general, poor or insufficient wash-fastness could be identified to be of minor importance as a limitation for the use of natural dyes extracted from the two different representative sources. Sufficient dyestuff fixation also can be seen as good marks for wash-fastness bleeding. The extracted dyes, however, showed very good wash fastness bleeding except in a few cases. The high fastness to washing of the unmordanted dyed fabric samples showed that the dye extracts from the two plant sources have good affinity for cotton, acrylic and nylon fibres.

Effect of mordanting

Mordanting has been usually applied to improve colour fastness of naturally dyed fabrics. In addition, it is well-known that colour characteristics of the fabric, may change after mordanting[24] thus, the results in tables 4, 5 and 6 showed that without mordanting the colourant dyed fabrics gave lighter hues compared, to the mordanted-colorant dyed fabrics. The results also showed that the chrome mordanted dyed fabrics showed deeper hue on acrylic fabric compared to those of cotton and nylon fabric. The reason for this could be attributed to the fact that the acrylic fabric sample had higher exhaustion at the liquor-to-material ratio used.

Table 1. Experimental conditions used for dyeing and mordatning

Parameters	Dyeing	Mordanting
Temperature	90°C	90°C
Liquor ratio	1:30	1:30
Timie (min.)	60	30
Concentration (%0wf	2	1, 3
pH of bath	42 ±0.2	Near neutral

Table 2. Experimental conditions used for Dyeing acrylic fabrics

Parameters	Dyeing	Mordanting
Temperature	110°C	110°C
Liquor ratio	50:1	50.1
Timie (min.)	45	45 min
Concentration (% 0wf	1	1
pH of bath	4-5	Near neutral

Table 3. Spectroscopic Properties

Source	Physical State	Wavelength (nm)	IR (KBr)cm^{-1}	'HNMR (d ppm) ($CDCl_3$)	MP(^{0}C)
Water opepe	Solid	431 (0.325)	3.443 (OH, NH) 1635 (C-O-C) 1500(Ar-Hg)	s, 1.15 (NH Proton) m, 7.25 (Phenyl proton)	260^0
				s, 1.35 (NH proton) m, 7.20	
Water ekimi	Solid	413 (0.298)	3441(OH, NH) 1629 (C-O-C) 1075(Sulphur)	(Phenyl proton	137-139

Table 4. Results of dyeing experiments on Cotton

Plant Material	Mordant	Sample	Fastness to light	Wash fastness	
				Colour	Bleeding
Water opepe		UM	1-2	3-4	5
	Cr	PreM	2-3	4	5
	Cr	Posm	3	4-5	5
	Sn	PreM	2-3	4-5	5
	Sn	Posm	2	4	4.5
	Cu	PreM	2	4-5	4.5
	Cu	PosM	2	4-5	4-5
	Fe	PreM	2	4-5	5
	Fe	PosM	3	5	5
		UM	1-2	4	4-5
Water ekimi	Cr	PreM	3	5	5
	Cr	PosM	3	5	5
	Sn	PreM	2-3	4-5	4-5
	Sn	PosM	2-3	4-5	4-5
	Cu	PreM	2	4-5	5
	Cu	PosM	2	4	5
	Fe	PreM	2-3	4-5	5
	Fe	PosM	2-3	4-5	5

Table 5. Results of dyeing experiments on Nylon

Plant Material	Mordant	Sample	Fastness to light	Wash fastness	
				Colour	Bleeding
Water opepe		UM	1-2	3-4	4-5
(Nuclea	Cr	PreM	2	5	5
didevricchii)	Cr	PosM	2	5	5
	Sn	PreM	2	4-5	5
	Sn	PosM	2	4-5	5
	Cu	PreM	2-3	4	4-5
	Cu	PosM	2	4-5	4-5
	Fe	PreM	3	4-5	5
	Fe	PosM	2-3	4-5	5
Water ekimi		UM	-2	3-4	4
	Cr	PreM	2	5	5
(Vanilya	Cr	PosM	3	5	5
planofolia)	Sn	PreM	2-3	4-5	4-5
	Sn	PosM	2-3	4-5	4-5
	Cu	PreM	2	4-5	4
	Cu	PosM	2	3-4	4-5
	Fe	PreM	2-3	3-4	4-5
	Fe	PosM	3	4	

Plant material, type of mordant (Fe iron (II) salt), Sn (tin (Ii salt) Cu, (copper (II) salt), Cr (chrome, salt) UM = unmordanted, PreM = Premordanted, PosM = Postmordanted;
Wash - fatness colour/bleeding 1= poor, 5= excellent, fastness to light 1 = poor; 8 = excellent

Table 6. Results of dyeing experiments on Nylon

Plant Material	Mordant	Sample	Fastness to light	Wash fastness	
				Colour	Bleeding
Bark of water		UM	2	3-4	4-5
opepe	Cr	PreM	3-4	4-5	5
(Nuclea	Cr	PosM	3-4	5	5
didevricchii)	Sn	PreM	3-4	4-5	5
	Sn	PosM	3-4	4-5	5
	Cu	PreM	2-3	4-5	4-5
	Cu	PosM	2-3	4-5	5
	Fe	PreM	2-3	5	4-5
	Fe	PosM	2-3	5	5
		UM	2	4	4
Bark of water	Cr	PreM	3-4	4-5	5
ekimi	Cr	PosM	3	5	5
(Vanilya	Sn	PreM	3	4-5	4-5
planofolia)	Sn	PosM	3	5	4-5
	Cu	PreM	2-3	3	5
	Cu	PosM	2-3	3	5
	Fe	PreM	2-3	4-5	5
	Fe	PosM	2-3	4-5	5

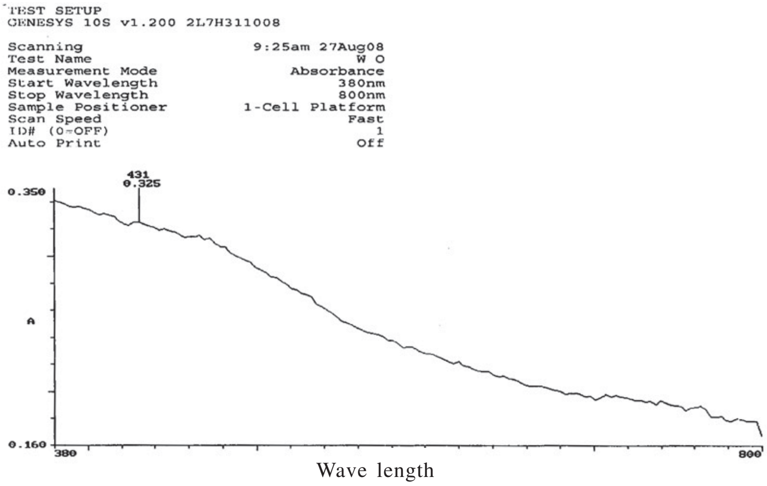

Fig. 1. UV Spectrum of Water Opepe

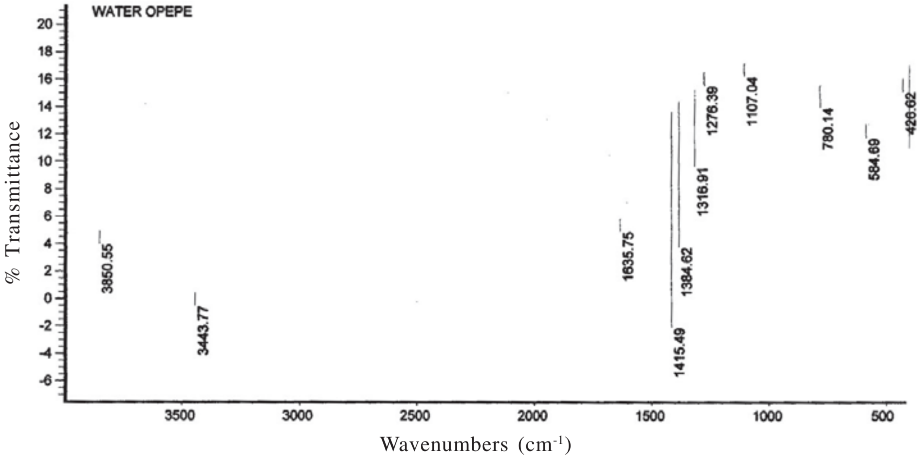

Fig. 2. IR Spectrum of Water Opepe

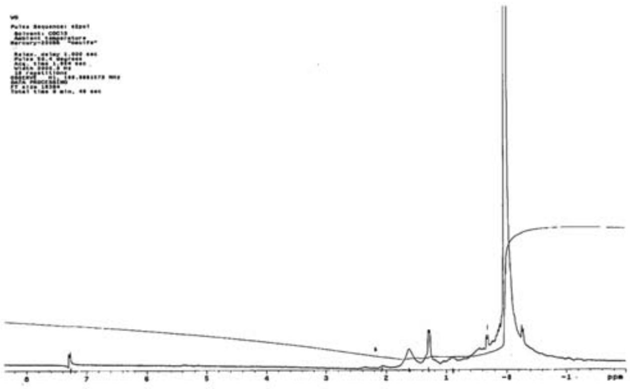

Fig. 3. [1]HNMR of Water Opepe

The water ekimi dye extract gave brown colour on the three substrates. Also the water opepe dye extract gave a brown colour on cotton and nylon but a green colour on acrylic fabric. On premordanting with potassium chromate, water opepe dye extract gave deeper brown shades on the three fabrics. The depth of brown shades is greater on acrylic fabric. The depth of brown shades increased in the order nylon < cotton < acrylic. Postmordanting with potassium chromate gave similar results with water ekimi dye extract. Copper (II) sulphate and iron (II) sulphate salts also gave deeper shades in both premordanting and post mordanting using the two dye extracts.

CONCLUSIONS

In this study, the extraction, characterization and application of natural dyes from the barks of water opepe and water plants ekimi were carried out. From the results of the selected fastness properties, the wash fastness of the two dye extracts showed that the colorants have affinity for cotton, nylon and acrylic fibres. The presence of phenyl protons in the 'HNMR spectra of the dye extracts gave an indication that they could be potential colorants for the petroleum industry especially gasoline. Further research in this area is ongoing.

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