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UTILIZATION OF AQUEOUS, METHANOL AND CHLOROFORM EXTRACTS OF LOCAL PLANTS- IXORA COCCINEA AND HIBISCUS SABDARRIFFA (ZOBO) FROM ABAKALIKI AS FABRIC DYES

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ABSTRACT

This investigation dealt with the dyeing and fastness properties of crude constituents of *Ixora coccinea* and *Hibiscus sabdarriffa* plant species extracted using distilled water, methanol and chloroform. Approximately 150 g of each sample was soaked in the chosen solvent for four weeks, the percentage yields of the aqueous, methanol and chloroform extracts of *Ixora coccinea* were 38.47% and 13.40 % and 3.88 % respectively; *Hibiscus sabdariffa* gave yields of 22.85 % for the aqueous extract, 12.24 % for the methanol extract and 3.79 % for the chloroform extract. Aqueous, methanol and chloroform extracts of *Ixora coccinea* were all pink in colour; *Hibiscus sabdariffa* gave red aqueous extract, red methanol extract and orange chloroform extract. The extracts were used without further purification in dyeing unmordanted and mordanted cotton and polyester fabrics. Different colour shades were obtained after dyeing. The mordanted fabrics using $CuSO_4$, $FeSO_4$ and K_2CrO_7 were fast to acid, alkali and washing.

Key words: dyeing, extract, fastness, mordant, solvent

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1 INTRODUCTION

In the past all dyes, indicators and paint were obtained from nature- directly from animals such as lac, cochineal, kermes; from minerals such as various inorganic metal salts and metal oxides and plants such as roots, flowers, foliage, nuts, trunk, fruit, bark, berries etc(Anjali, Deepali, 2012; Buchanan, 1999).

With the present awareness all over the world of problem posed by synthetic compounds and dyes, natural dyes are gaining attention because of their non toxicity, safe handling and pose no environmental danger or hazard(Pathan and Mazahar, 2011; Adeel, Ali, Bhatti and Zsilla, 2009; Akpuaka, Chukwueneke and Agbo, 1998; Ferricira and Hulme, 1993. Ledakowicz, Olczyk, Polak, Graz and Wilkolazka 2015).

Dyes are intensely coloured substances which when applied to a substance, imparts colour to it by a process which at least temporarily destroys the crystal structure of the coloured substance. The dye must be attractive and capable of being fixed to the substrate directly or with the help of certain reagents called mordants. The dye must be colour fast, produced at a relatively low cost, cheap to obtain and readily available (Bhattacharya and Shah, 2000; Green, 1995; Bhuyan and Saika, 2004).

Hibiscus sabdariffa is an herbaceous and perennial plant cultivated in the tropical and sub tropical regions. It belongs to a family of plants called malvaceae of which over three hundred species have been described (Tease and Evans, 1995). In Africa especially Nigeria it is prepared as a non alcoholic beverage and is known as Zobo. The extract from *Hibiscus sabdariffa* is used in herbal medicine as antiseptic, aphrodisiac, astringent, cholagogue and demulcent. The extract from *Ixora coccinea* has been found to exhibit antioxidant, antibacterial, antidiarrhoeal, antimutagenic properties([Manjeshewar and Poruthukaran, 2012; Bhattacharjee, 1994).

The author decided to look at the use of these common plant species as local/natural dyes for fabrics and by extension their use in the dye industry; cosmetics, food and biology stains considering their medicinal and physiological uses.

2 GENERAL EXPERIMENTAL PROCEDURES

All reagents used were of analytical grade, purchased from BDH, England and Merck Germany. All weighing was done on Mettler P2100.

2.1 Plant Samples

Dry samples of *Hibiscus sabdariffa* (zobo) were purchased from Meat Market Abakaliki, Ebonyi State Nigeria in May 2014 while fresh samples of *Ixora coccinea* were handpicked from the office complex of Faculty of Science Ebonyi State University Abakaliki in June 2014 during the flowering season. Both samples were identified by the Applied Biology Department Faculty of Science Ebonyi State University.

2.2 Sample Preparation

Approximately 1000 g of each sample was washed with distilled water to remove sand and dirt and air dried for seven days. The dried samples were pulverized and stored in air tight cellophane until ready for use.

2.3 Extraction

About 150 g of *Hibiscus sabdariffa* was soaked in 500mL of distilled water, methanol and chloroform respectively. The same 150 g of sample and procedure of soaking was carried out for *Ixora coccinea*. The soaked samples were allowed to stand for four weeks to achieve exhaustive extraction of the constituents of the plant species. Each sample solution was filtered and the filtrate evaporated to dryness by mild heating to reveal dark red *Hibiscus sabdariffa* aqueous extract (HSAE); red *Hibiscus sabdariffa* methanol extract (HSME); orange *Hibiscus sabdariffa* chloroform extract (HSCE); dark brown *Ixora coccinea* aqueous extract (ICAE); Purple *Ixora coccinea* methanol extract (ICME) and brown Ixora *coccinea* chloroform extract (ICCE) (Osabohien and Ukponmwan, 2002). The solubility of the extracts in distilled water and ethanol was determined.

2.4 Preparation of Dye Baths

About 0.4 g of HSAE, HSME, HSCE ICAE; ICME and ICCE were used in the preparation of six different dye baths. HSAE, HSME, ICAE; ICME and ICCE were dissolved in100 mL of distilled hot water while 0.4 g of (HSCE) was first dissolved in 20 mL chloroform before introduction into 80 mL of hot water. To each solution were added 1 g of NaOH and 1g Na_2SO_4 respectively and heated to a temperature of 45^{o}C until a uniform mixture was obtained.

2.5 Dyeing of Cotton and Polyester Fabrics

About six pieces of 30 x 30 cm of each of the white fabrics (cotton and polyester) were prepared by boiling in 10 % NaOH for 15 min to remove starch and other impurities and then introduced into each dye bath and the temperature was raised to 80^{o}C and allowed to simmer at that temperature for 30 min. The dye baths were removed from source of heat and allowed to cool to 40^{o}C for 15 min. The fabrics were removed without rinsing and allowed to dry at room temperature for 6 h, the dried fabrics showed different colours.

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2.6 Fastness Tests of unmordanted fabrics

2.6.1 Colour fastness to Acid

Six pieces of each fabric (5 x5 cm) were dipped in 10 % HCl for 5 min, removed without rinsing and allowed to dry at room temperature.

2.6.2 Colour fastness to Alkali

Six pieces of each fabric (5 x5 cm) were dipped in 10 % NaOH solution for 5 min, removed without rinsing and allowed to dry at room temperature.

2.6.3 Wash Fastness

Six pieces of each fabric (5 x5 cm) were soaked in 10 % soap solution for 6 h, washed for 5 min, rinsed in water and dried at room temperature (Baishya, Talukdah and Sandya, 2012).

2.7 Mordanting and Dyeing of Fabrics

Approximately 0.4 g of Ferrous sulphate, Copper sulphate and Ferric chloride and potassium dichromate were weighed out separately and dissolved in 100 mL of hot water respectively. To each salt were added 0.4 g Na_2CO_3 and Na_2SO_4 and allowed to simmer for 10 min, cotton and polyester fabrics 5 x5 cm were each soaked separately in each metal salt solution for 45 min at a temperature 60 ^oC. The fabrics were removed and introduced into freshly prepared dye baths of HSAE, HSME. HSCE, ICAE, ICME and ICCE and the temperature of the baths gradually raised to 90^oC and allowed to simmer for 1 h. After 1 h the dye baths were allowed to cool for 20 min, and the dyed fabrics removed and spread at room temperature until dry.

2.7.1 Fastness Tests of unmordanted fabrics

The same procedure was followed for the fastness tests of the mordanted fabrics.

3 RESULTS

Percentage yield of the extracts appears on Table I; the solubility of the extracts in water and ethanol is shown in Table II, the result of the dyeing of the unmordanted fabrics appears on Table III; fastness properties of the unmordanted fabrics is on table IV; colours of dyed Fabrics using different mordants is outlined on Table V; the fastness tests are shown on Tables VI, VII and VIII.

Table I Percentage Yield of the Extracts

Extracts	Weight of Plant Powder(g)	Weight of dye	Percentage yield (%)
HSAE	150	34.28	22.85
HSME	150	24.47	12.24
HSCE	150	5.68	3.79
ICAE	150	57.71	38.47
ICME	150	20.10	13.40
ICCE	150	5.82	3.88

Table II Solubility of HSAE, HSME, HSCE ICAE; ICME and ICCE in Water and Ethanol

Extract	Solvent	
	Distilled water	ethanol
HSAE	soluble	soluble
HSME	insoluble	insoluble
HSCE	soluble	soluble
ICAE	soluble	insoluble
ICME	soluble	soluble
ICCE	soluble	soluble

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Table III Colours of the Dyed unmordanted Fabrics

Extract	Colour on fabrics	
	Cotton	Polyester
HSAE	red	pink
HSME	light green	red
HSCE	pink	light yellow
ICAE	red	brown
ICME	red	violet
ICCE	light red	light brown

Table IV Fastness properties of the unmordanted fabrics

Extract	Cotton			Polyester		
	acid	alkali	wash	acid	alkali	wash
HSAE	3	4	3	4	3	4
HSME	3	4	3	4	3	4
HSCE	4	3	4	4	4	3
ICAE	3	4	3	4	3	3
ICME	4	4	4	3	3	4
ICCE	4	4	4	4	4	4

3 = slightly fast; 4 = not fast

Table IV Colours of Mordanted Dyed Fabrics

Extracts	Colours of Mordants on Cotton				Colours of Mordants on Polyester			
	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7
HSAE	White	Brown	Orange	Yellow	White	Brown	Orange	Pink
HSME	Grey	Brown	Yellow	Orange	Grey	Brown	Yellow	Pink
HSCE	Green	Orange	Grey	Yellow	Green	Orange	Grey	Yellow
ICAE	Pink	Brown	Orange	Red	Pink	Brown	Orange	Red
ICME	Pink	Brown	Pink	Orange	Pink	Brown	Pink	Orange
ICCE	white	brown	brown	violet	white	brown	brown	pink

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Table V Acid Fastness of the mordanted Dyed fabrics

Extracts	Colours of Mordants on Cotton				Colours of Mordants on Polyester			
	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7
HSAE	2	1	1	1	3	2	2	1
HSME	2	2	1	1	3	1	1	1
HSCE	1	1	2	2	1	2	1	1
ICAE	1	1	2	2	1	2	1	2
ICME	1	2	1	1	2	1	2	1
ICCE	2	2	4	4	2	4	4	4

1 = very fast; 2 = fast; 3 = slightly fast; 4 = not fast

Table VI Alkaline Fastness of the mordanted Dyed fabrics

Extracts	Colours of Mordants on Cotton				Colours of Mordants on Polyester			
	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7
HSAE	2	1	1	1	2	2	1	1
HSME	1	1	2	1	2	1	2	2
HSCE	2	1	2	2	1	1	2	2
ICAE	1	1	2	1	1	2	1	2
ICME	1	2	1	1	2	2	1	2
ICCE	3	1	4	4	4	1	4	3

1 = very fast; 2 = fast; 3 = slightly fast; 4 = not fast

Table VII Wash Fastness of the mordanted Dyed fabrics

Extracts	Colours of Mordants on Cotton				Colours of Mordants on Polyester			
	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7	$CuSO_4$	$FeSO_4$	$FeCl_3$	K_2CrO_7
HSAE	2	1	1	1	2	4	1	1
HSME	1	1	2	1	2	1	2	1
HSCE	2	1	2	2	1	2	2	2
ICAE	1	1	1	1	1	1	1	2
ICME	1	2	1	1	2	2	1	1
ICCE	3	1	4	4	2	1	4	4

1 = very fast; 2 = fast; 3 = slightly fast; 4 = not fast

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4 DISCUSSION

The powdered plant species after extraction in the three different solvents gave the highest yield using water; 22.85 % HSAE and 38.47% ICAE. The methanol extract of the plant materials gave 12.24 % for HSME and 13.40 % for ICAE. The yield using chloroform was low; 3.79 % HSCE and 3.88 % ICCE. The implication of the result obtained using chloroform was that chloroform was not a good solvent to use for the extraction of the constituents of these plant materials.

The extracts were appreciably soluble in water except HSME and in ethanol except HSME and ICAE. All the extracts gave colours ranging from red, green, pink, yellow and brown on unmordanted cotton and polyester fabrics. The acid, alkaline and wash fastness of the unmordanted fabrics was poor.

Fresh white cotton and polyester fabrics were treated with metal salts $CuSO_4$, $FeSO_4$, $FeCl_3$ and K_2CrO_7 as mordants and dyed to help to make the colours able to be fixed on the fabrics. It was observed that the use of mordants gave different colours from the ones obtained without the use of the metal salts. HSAE, HSME, HSCE, ICAE, ICME and ICCE gave colours that vary from white, brown, orange, yellow and pink using $CuSO_4$, FeSO4, $FeCl_3$ and K_2CrO_7 on the two fabrics used. That is different shades were obtained from a single extract, using different mordants.

Cotton and polyester mordanted fabrics when dyed with HSAE, HSME, ICAE and ICME and treated with 10 % HCl, showed excellent fastness property except HSAE and HSME which gave poor result with polyester mordanted with $CuSO_4$ and also ICCE which gave poor result with $FeCl_3$ and K_2CrO_7 mordanted cotton and $FeCl_3$ and $FeSO_4$ mordanted polyester.

The mordanted dyed fabrics were all fast to 10 % alkali except ICCE on cotton mordanted with $CuSO_4$, $FeCl_3$ and K_2CrO_7 and ICCE on polyester mordanted with $CuSO_4$, $FeCl_3$ and K_2CrO_7.

All the mordanted dyed fabrics were fast to washing except ICCE which was only wash fast on FeSO4 mordanted cotton and polyester.

The results of the investigation showed that *Hibiscus sabdariffa* and *Ixora coccinea* extracts could be used in dyeing fabrics as a source of natural dyes for natural and synthetic fibers. Though used as beverage in the case of *Hibiscus sabdariffa* the plant material could be of immense help in the dye industry especially the aqueous extract for cosmetics. It could also find use in the food and drug industries. The methanol extract could be used in paints and other materials that are non-edible. The colour exhibited by the unmordanted and mordanted dyed fabrics, acid, alkali and wash fastness showed that these plant dyes have affinity and showed acceptable fastness properties.

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