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EXTRACTION OF NATURAL DYES, ITS APPLICATION IN TEXTILE COLORATION AND COMPARATIVE STUDY WITH REACTIVE DYES

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ABSTRACT

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The worldwide demand for natural dyes is of great interest due to the increased awareness of medicinal properties. Also non-toxic, biodegradable and eco-friendly properties make them exceedingly popular. It is due to the technical drawbacks, such as, color yield, complexibities of dyeing process, limited shades, reproducibility and fastness properties which restricts their usage in textile dyeing. So standardization of dyeing techniques needs to be explored for successful commercial use of natural dyes. Turmeric, Neem and Henna are one of the most ancient spices is commonly used as a dye. These are perennial plants found in the tropical regions of Southern Asia. These give vibrant yellow-orange, light brown and dark brown color. These can be also used to dye fabrics. The dye solution used for dyeing was extracted by boiling the meshed form of Turmeric, Neem and Henna individually. Then the extracted solution used dyeing 9(nine) derivatives of knitted fabric and one woven fabric by exhaution process in lab dyeing machine under different dyeing conditions. Next to it the individual shade of dyed fabrics were scanned through spectrophotometer to get recipe for reactive dyes. The color fastness to wash, color fastness staining, rubbing, perspiration for the dyed fabrics with Turmeric, Neem & Henna were done to compare with the fabrics dyed with reactive dyes. Again, due to shortage of time the costing for both natural dyed fabric and reactive dyed fabric were not done.

Key words: water, dyes, treatment, dying, coloration

INTRODUCTION

For thousands of years people have dyed textiles and for most of the time the dyes have come from nature. Only in the past 100 years has our textile industry depended only in synthetic dyes. The worldwide demand for natural dyes is nowadays of great interest due to the increased awareness on therapeutic properties of natural dyes. Although known for a long time for dyeing as well as medicinal properties, the structures and protective properties of natural dyes have been recognized only in the recent past. A renewed international interest has arisen in natural dyes due to increased awareness of the environmental and health hazards associated with the synthesis, processing and use of synthetic dyes. Natural dyes comprise colorants that are obtained from animal or vegetable matter without any chemical processing. During the last decade the use of natural dyes, obtained from animal or vegetable matter without any chemical processing, has gained momentum due to increased demand for these dyes by the food, pharmaceutical, cosmetic as well as the textile coloration industry (http://en.wikipedia.org/wiki/Turmeric).

Textile processing industry is one of the major environmental polluters. In order to process a ton of textile, one might have to use as much as 230 to 270 tons of water. The effluent generated by this much water would pollute the environment as it contains a heavy load of chemicals including dyes used during textile processing. Over 7 x 105 tones and approximately 10,000 different types of dyes and pigments are produced world-wide annually. It is estimated that 10-15% of the dye is lost in the effluent during the dyeing process. Thus, there are two main ways to limit the environmental impact of textile processing (http://en.wikipedia.org/wiki/Neem). One is to construct sufficiently large and highly effective effluent treatment plants, and the other way is to make use of dyes and chemicals that are environment friendly. Natural dyes are mostly eco-friendly, biodegradable, less toxic, and less allergenic as compared to synthetic dyes. However, studies have shown that certain natural dyes may have detectable mutagenic effects e.g., elderberry color and safflower yellow; others, like carmine, can cause asthma by continuous inhalation, but it can be said that most of the natural dyes are safe and some even have curative effect e.g., curcumin in turmeric has antibacterial properties.

However, in spite of the merits of natural dyes as compared to the synthetic ones, the use of the former is still not widespread due to non-availability of standard shade cards and standard application procedures. Most of the natural dyes have no substantively for the fiber and are required to be used in conjunction with mordants. A mordant, usually a metallic salt, is regarded as a chemical, which itself be fixed on the fiber and which also combines with dyestuff. A link is formed between the fiber and the dye, which allows certain dyes with no or little affinity for fiber to be fixed (http://en.wikipedia.org/wiki/Henna).

MATERIALS AND METHODS

Henna leaves: For extracting Henna dye Turmeric rhizomes: For extracting Turmeric rhizomes Neem leaves: For extracting Neem dye Potassium di chromate ($K_2Cr_2O_7$): as mordant Reactive dyes [Reason Yellow-2R-X, Reason Red-2R-X, Reason Blue-2R-X, Reason Br Yellow 3GL, Reason Br Yellow 3GL]: For making comparative study with natural dyes.

100% Woven cotton fabric (100×80/40×40), Single Jersey, 1×1 Rib, 2×2 Rib, Lycra Rib, Pointer Rib, hani PK, Terry Fleece (70% cotton + 30% polyester), Terry Fleece (100% cotton), Double Lacoste: For dyeing (with natural and reactive dyes both)

Gluber Salt: For dyeing (with natural and reactive dyes both)

Soda ash: For dyeing (with natural and reactive dyes both)

Leveling agent: For dyeing (with natural and reactive dyes both)

High Temperature Laboratory scale dyeing machine: For exhaust dyeing

Datacolor SF 600 Spectrophotometer: To get recipe for reactive dyeing form natural dyed fabric.

Crockmeter: For rubbing fastness.

Multi fiber fabric: For color fastness to staining.

Standard soap solution: For color fastness to wash & staining.

Incubator: For perspiration test.

Mordanting Methods

A mordant is a substance used to set dyes on fabrics or tissue sections by forming a coordination complex with the dye which then attaches to the fabric or tissue. It may be used for dyeing fabrics, or for intensifying stains in cell or tissue preparations. The term mordant comes from the Latin word, "mordere", to bite. In the past, it was thought that a mordant helped the dye bite onto the fiber so that it would hold fast during washing. A mordant is often a polyvalent metal ion. The resulting coordination complex of dye and ion is colloidal and can be either acidic or alkaline.

Common mordants

Mordants include tannic acid, alum, urine, chrome alum, sodium chloride, certain salts of aluminium and, copper, iron, iodine, potassium, sodium, and tin. Iodine is often referred to as a mordant in Gram stains but is in fact a trapping agent.

The three methods used for mordanting are:

- Pre-mordanting (on chrome): The substrate is treated with the mordant and then dyed.
- Meta-mordanting (met chrome): The mordant is added in the dye bath itself.
- Post-mordanting (after chrome): The dyed material is treated with a mordant.

The type of mordant used changes the shade obtained after dyeing and also affects the fastness property of the dye.

Testing Methods

Colorfastness to Rubbing

Principle:

This test is designed to determine the degree of color which may be transferred from the surface of a colored fabric to a specify test cloth for rubbing (which could be dry and Wet).

Equipment that needed for measurement:

Crock Meter.

Cotton Rubbing Cotton.

Grey Scale Stop Watch

Color Matching Cabinet.

Size of Fabric:

Need to have 14 cm \times 5 cm pieces of textile fabric sample (one warp direction/wale direction and other weft/course direction).

Test Procedure of Color Fastness to Rubbing:

Lock the test specimen (textile sample) onto the base of the crock meter.

Using the spinal clip, set $5 \text{ cm} \times 5 \text{ cm}$ of the white cotton fabric to the finger of the crock meter.

Lower the covered finger on the test sample.

Turn hand crank at the rate of the one turn per second.

Remove the white rubbing test cloth and e valuate with grey scale.

Evaluation:

In this stage compare the contrast between the treated and untreated white rubbing cloth with grey scale and rated 1 to 5.

Colorfastness to Wash

Washing conditions may vary from one country to another.

The methods on the use of dyed goods.

To evaluate repeated washing accelerated test methods are used.

The degree of fading and staining of dyed goods for washing depends upon the following factors:

Temperature range may be from 40 Degree centigrade to 95 Degree centigrade. The type and amount of detergent added to the washing bath. In many testing procedures a standard detergent is used. The extent of mechanical action which can be varied by changing the agitation speed in a washing machine or by adding steel ball to revolving the bath. The washing liquor to goods ratio is 50:01. The hardness of water. The rinsing, drying, or pressing methods used to restore the sample after the washing test.

Procedures of Fastness Measurement

A Specimen in contact with specified adjacent fabric or fabric is laundered, rinsed and dried. The specimen/composite sample is treated under appropriate condition in a chemical bath for short time. The abrasive action is accomplished by the use of a liquor ration and an appropriate number of steel balls. The change in color of the specimen (Dyed Sample) and the staining of the adjacent fabric is assessed by recommend Grey Scales (1-5).

Apparatus & Materials used for measuring Color Fastness of Textiles Materials:

Wash-wheel with a thermostatically controlled water bath and rating speed of 40+-2 rpm. Stainless steel container. Stainless steel ball. SDC, Multifiber Fabric (Acetate/Cotton/Nylon/Polyester/Acrylic Fiber/Wool). Thermometer, Sewing Machine. Drver. Color Matching Cabinet and ISO Scales. **Reagents:** Reference Detergent. Distilled Water (Grade-3) Sodium Carbonate/Soda Ash.

Test Specimen:

A Textile material (Dyed Goods) sample should be cut at 10 CM into 4 Cm and sew it with same size multifiber fabric. This is the composite test sample.

Test Procedure:

The color fastness procedure I am going to tell you is ISO certified and recommended -ISO-105-C01: Composite sample is treated in a wash wheel for 30 minutes at 40 plus minus Degree centigrade with 5 Gram/Ltr standard soap.

Evaluation:

Compare the contrast between the treated and untreated sample with Grey scales for changing color of dyed sample and staining of adjacent fabric in a color matching cabinet. Numerical rating for color changing is the shade and staining to adjacent fabric. Number of method used.

Reporting Format of Color fastness measurement:

Color change in shade staining in Acetate – Grade 4.

Color change in shade staining in Cotton - Grade 4.

Color change in shade staining in Polyamide — Grade 4.

Color change in shade staining in Polyester — Grade 4-5.

Color change in shade staining in Acrylic – Grade 4—5.

Color change in shade staining in Wool — Grade 4.

Colorfastness to Perspiration

Principle

The garments a\which come into contact with the body where perspiration is heavy may suffer serious local discoloration. This test is intended to determine the resistance of color of dyed textile to the action of acidic and alkaline perspiration.

Equipment for Fastness Measurement

Perspiration tester

Oven, Maintained at 37+-2 Degree centigrade Multifiber test fabric Grey scale Color matching chamber Acidic and Alkaline solution Glass or Acrylic plat3e 8. Weight. **Reagents:** Histidine mono-hydrochloride mono-hydrate ($C_6H_9O_2N_3HCl.H_2O$)

Sodium chloride (NaCl)

Disodium hydrogen orthophosphate dihydrate (Na₂HPO₄.2H₂O) Distilled Water (Grade-3)

Sample size will be 10 CM × 4 CM

Test Procedure

Wet-out the composite test sample in mentioned alkaline or acidic solution at room temperature. The Material ration will be 1:50 and leave for 30 minutes. Pour off excess solution and place the composite sample between two glass plate or acrylic plate under a pressure of 4.5 KG and place in a oven for 4 hours at 37+- and 2 degree centigrade temperature. Remove the specimen and hang to dry in warn air not exceeding 60 Degree centigrade. **Evaluation**

Evaluation is done by Grey scale in a dyed color matching cabinet and rate from 1 to 5.

RESULTS AND DISCUSSION

Collection and processing of sample

Among the three core ingredients, Henna leaves and Neem leaves were collected from gardens and the Turmeric were collected from market. They were washed and made into fine paste. These pastes were boiled with water and stared to get the dyes solution. Now these solutions were used to dye nine derivatives of knitted fabrics and one plain woven fabric, where dyeing conditions were using only the extracted solution, using the solution with salt, soda and leveling agent and using only solution on mordant fabrics. Next to it we took recipe for one sample from each process using Spectrophotometer (Data Color) against Reactive dyes. Then we dyed the ten samples with the same procedure. After that we find out a comparison on the color fastness to wash, perspiration, and rubbing (Crocking) for each sample along between reactive dye and natural dyes (Henna, Neem and Turmeric).

Extraction Conditions

Various experiments were conducted for the extraction of natural dye from Henna, Neem and Turmeric in distilled water. Here we boiled 500gm of each ingredients and boiled it with 1000ml of distilled water at temperature 60° C for one hour at pH 6-7. Then we stared out the solution to get dyeing solution.

Dyeing Conditions

For the optimization of dyeing conditions the following parameters were selected:

- 1. Temperature
- 2. Dyeing time
- 3. M : L ratio
- 4. pH
- 5. Salt
- 6. Soda
- 7. Mordant process

Dyeing temperature

All experimental dyeing processes were performed at temperature 60° C for 1 hour.

Dyeing time

All experimental dyeing processes were performed at temperature 60°C for 1 hour.

M : L ratio

All the experiments were carried out with M : L = 1:10

Dyeing pH

We had three set of experiments where the bath pH was a great point to maintain. Such as

The bath with extracted solution and normal fabrics had pH of 4.5 - 5.5.

The bath with extracted solution with salt, soda and leveling agent and normal fabrics had pH of 9 - 10.

The bath with extracted solution and mordant fabrics had pH of 4.5 - 5.5.

Salt concentration

All the experiments were carried out with salt = 5 cc

Soda concentration

All the experiments were carried out with salt = 8 cc

Turmeric (Curcuma longa)

Turmeric (*Curcuma longa*) is a plant native to south India and Indonasia. It is also cultivated in China and the whole of South East Asia. It is also called "Haldi". Its tuberous rhizomes have been used as a condiment, a colorant and an aromatic stimulant since antiquity. Turmeric consists of various molecular constituents, including three gold color alkaloidal curcuminoid, curcumides methoxy curcumin and bisdemethoxy curcumin. The curcuminoid content responsible for color, depends upon the turmeric variety and within a variety on the maturity at harvest. It may be present to the extent of 4to 8% in turmeric harvesting at the right maturity being an important factor for colorant aroma. Some isomeric forms of curcumin are displayed below:



Isomeric forms of Curcumin

Curcumin has anti-inflammatory, antifungal and anti-tumorous. It is also widly used as food colorant. It is called C.I Natural Yellow 3, WHO (World Health Organization) and FAO (Food and Agricultural Organization) committees have approved it as food additive, its color index number is C.I, 75300, E100. The plant of turmeric in given in Fig. 1.



Fig. 1. Turmeric Rhizome

Synonyms:

Holud (Bangladesh), Curcuma (Sp. It. Fr.), acafrao da India (port.), geelwortel (Dutch), kurkum Arab. Manjano (East Africa), haldi (Hindi) manjal (Tamil), kunyit (Indonesia) temu kunyit (Malaysian), iyu-chin (Chin.).

Scientific Specification:				
Kingdom	: Plantae			
Division	: Magnoliophyta			
Class	: Liliopsida			
Subclass	: Zingiberidae			
Order	: Zingiberales			
Family	: Zingiberaceae			

: Curcuma

: C. longa

Description:

Genus

Species

Turmeric belongs to the family of Zingiberacea, the ginger family. Anybody familiar with this plant family will readily recognize this affiliation even by simple superficial examination. Turmeric is an upright, relatively short and stout plant that rarely grows more than about 1 meter in height. Its leaves are elongated, dark green, and pointed, often curling slightly along the margins. Each leaf arises on an individual stalk directly from the fleshy rhizome at their base. The rhizome appears scaly due to the remaining rings of previous leaves. Its outer skin is brownish, but its flesh is deep orange-yellow inside. Rhizomes grow to about 5-8cm x 1.5-2.5cm. When bruised they omit a spicy scent. The flower stalk will appear among the leaves, also directly rising from the rootstock. The yellow-reddish flowers are arranged spirally along the cylindrical spike, which may be partially protected by a leaf sheath. The flowers poke out from protective bracteoles, which form little 'pockets' along the flower spike. Turmeric propagates mostly vegetatively by means of rhizome segments.

Cultivation:

Pot on the turmeric roots as soon as the shoots are 5cm high, into shallow 15cm pots. Keep them damp and warm, in a slightly shaded position and feed during the growing season weekly with a general purpose liquid fertilizer. In dry weather plants will benefit from being lightly misted with rainwater daily.

During the autumn reduce watering, and keep the plants fairly dry over winter, when they will need more light. Turmeric hates being in a draught, so site your plants carefully.

Composition:

Turmeric contains up to 5% essential oils and up to 5% curcumin, a polyphenol. Curcumin is the active substance of turmeric and curcumin is known as C.I. 75300, or Natural Yellow 3. The systematic chemical name is (1E, 6E)-1, 7-bis (4-hydroxy-3-methoxyphenyl)-1, 6-heptadiene-3, 5-Dione.

It can exist at least in two turmeric forms, keto and enol. The keto form is preferred in solid phase and the enol form in solution. Curcumin is a pH indicator. In acidic solutions (pH <7.4) it turns yellow, whereas in basic (pH > 8.6) solutions it turns bright red.

Chemistry:

Turmeric contains about 5% of volatile oil, resin and yellow coloring substances known ascurcuminoids. The chief component of curcuminoids is known as "curcumin". Chemically curcuma species contain volatile oils, starch and curcumin (50–60%). Curcumin and other related curcuminoids are reported to be responsible for yellow color of the dye.



Medicinal importance:

Curcumin from *Curcuma longa* has antioxidant, anti-inflammatory, anti cancer and hepatoprotective. The pharmacological activities of curcumnoids are due to unique molecular structure. The phenolic yellow curry pigment curcumin used in the Alzheimer's disease, it involves amyloid (Abeta) accumulation, oxidative damage and inflammation potent. It has anti-inflammatory effects in arthritis, possibly inhibits prostaglandin synthesis pathway of Cox-2 without causing ulcers in the GI tract. Finally it has anti-platelet, anti-viral, anti-fungal, anti-bacterial effects (inhibits Helicobacter Pylori) and powerful antiseptic agent.

Turmeric is a mild digestive, being aromatic, a stimulant and a carminative. An ointment base on the spice is used as an antiseptic in Malaysia. Turmeric water is an Asian cosmetic applied to impart a golden glow to the complexion. Curcumin has been shown to be active against Staphylococcus aurous (pus-producing infections).

Extraction of Turmeric

Extraction is usually used to recover a component from either a solid or liquid. The sample is contacted with a solvent that will dissolve the solutes of interest. Solvent extraction is of major commercial importance to the chemical and biochemical industries, as it is often the most efficient method of separation of valuable products from complex feed stocks or reaction products. Some extraction techniques involve partition between two immiscible liquids; others involve either continuous extractions or batch extractions.

Water Extraction

Due to ecological and economical aspects the extraction of natural dyes from plant material is limited to water as solvent. The use of water brings lower costs and simplifies the waste water treatment requirements. If other solvents are used and/or chemicals are added, considerable amounts of inputs are lost. This may lead to complicated after-treatments of extracted plant material, to additional costs from solvent/chemical consumption and deposition of contaminated wastes. Dyes can be extracted with water by boiling. Our selective natural dye plants are individually grained into powder form and then boiled with water. Then the liquor is separated by filtration. The process is repeated for the same solid material to extract the dye portion as much as possible. *Dyeing*



Dyeing Conditions for Neem leaves:				
Neem leaves	50 ml			
Fabric	5 gm			
Temperature	$60^{\circ}C$			
Time	60 minutes			
M:L	1:10			
pН	As in medium			

Dyed Sample

		1		
Single Jersey	2×2 Rib	Honey PK	Pointer Rib	Terry Fleece (70% + 30%)
Double Lacoste	Terry Fleece (100%)	1×1 Rib	100% Woven cotton fabric	Lycra Rib

CONCLUSION

The present research work was carried out to evaluate the dyeing of cotton with natural dyes extracted from indigenous natural dyes Henna leaves, Neem leaves and Turmeric by keeping environment friendly procedures as well. The neutral conditions for extraction with other variables including extraction temperature, time and M: L of natural dyes from Henna leaves, Neem leaves and Turmeric were optimized. The optimum extraction medium for 60 minutes at 100°C with M : L ratio 1 : 10 for Henna leaves, Neem leaves and turmeric. The resulting extracts were used to further optimize its dyeing condition for cotton fabric. Dyeing with natural dyes and development of same shades with reactive dyes on same procedures was compared with those obtained with reference to shade, color strength, fastness properties. Finally, the comparative studies were conducted between synthetic reactive and these natural dyes. We tried to show the costing of both reactive dyes and natural dyes but it was not possible due to shortage of time. In whole process we extracted the dyes solution from Henna leaves, Neem leaves and Turmeric to dye nine derivatives of knitted fabrics and one woven fabric on different process and took different colorfastness results. Then we tried to develop the same shades of natural dyes using reactive dyes on same types of fabrics and same process and took comparative result of same fastness test of these shades against natural dyes. Again we tried show the acceptable sides of natural dyes compared to reactive dyes. We know the European countries shifted the dyeing mills to undeveloped countries because using of synthetic dyes is not good for environment at all. So we have shown a way to save our environment using natural dyes. If we could get a long time then it would be possible for us to gather more results.

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