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DECLARATION

I, Md. Nazmul Hossain, hereby declares that this thesis is my own work and that, to the best of my knowledge and belief, it reproduces no material previously published or written, nor material that has been accepted for the award of any other degree, except where due acknowledgement had been made in the text.

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Dedicated

To

My beloved Family

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Abstract

Natural dyes are those colorants which are extracted from plants, animals or insects. Recently, the contemporary environmental concerns have stimulated the public interest in natural dyeing that produces less toxic contamination. Mangrove plants generally believed to have a high tannin content which can be a potential source of natural dye (and contribute to the development of local culture). In this experiment, bark of Ora (*Sonneratia caseolaris L.*) was investigated for extraction of the dye and its effects on cotton, silk and wool and the bark was collected from the Sundarbans mangrove forest. The dye was extracted in tree media, namely acid, base and neutral where acetic acid (CH_3COOH), sodium hydroxide ($NaOH$) and distilled water (H_2O) were used respectively. Extracted dye was used in dying fabrics with pre mordanting (*Alum*) and post mordanting (*Copper sulphate and Ferus sulphate*) treatment. Color of fabrics was identified using RGB and CMYK color model using graphical software. From the experiment, it is seen that acidic and neutral extraction media have the good effect of dye extraction. But, the acidic media gave the best result on dye extraction. The basic medium is less effective for the extraction of die from Ora. Extracted dye from all media gave darker shade on cotton. The Darker color of the other fabrics was obtained when ferus sulphate was used as a post mordanting agent.

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Abbreviations

RGB — Red, Green, Blue.

CYMK — Cyan, Yellow, Magenda and Black

ACCu — Acid, Cotton, $CuSO_4$

ACF — Acid, Cotton, $FeSO_4$

ACNu — Acid, Cotton, Neutral

ASCu — Acid, Silk, $CuSO_4$

ASF — Acid, Silk, $FeSO_4$

ASNu — Acid, Silk, Neutral

AWCu — Acid, Wool, $CuSO_4$

AWF — Acid, Wool, $FeSO_4$

AWNu — Acid, Wool, Neutral

BCCu — Base, Cotton, $CuSO_4$

BCF — Base, Cotton, $FeSO_4$

BCNu — Base, Cotton, Neutral

BSCu — Base, Silk, $CuSO_4$

BSF — Base, Silk, $FeSO_4$

BSNu — Base, Silk, Neutral

BWCu — Base, Wool, $CuSO_4$

BWF — Base, Wool, $FeSO_4$

BWNu — Base, Wool, Neutral

NCCu — Neutral, Cotton, $CuSO_4$

NCF — Neutral, Cotton, $FeSO_4$

NCNu — Neutral, Cotton, Neutral

NSCu — Neutral, Silk, $CuSO_4$

NSF — Neutral, Silk, $FeSO_4$

NSNu — Neutral, Silk, Neutral

NWCu — Neutral, Wool, $CuSO_4$

NWF — Neutral, Wool, $FeSO_4$

NWNu --- Neutral, Wool, Neutral

OACu --- Ora, Acid, $CuSO_4$

OAF — Ora, Acid, $FeSO_4$

OAN --- Ora, Acid, Neutral

OBCu --- Ora, Base, $CuSO_4$

OBF — Ora, Base, $FeSO_4$

OBN — Ora, Base, Neutral

ONCu --- Ora, Neutral, $CuSO_4$

ONF — Ora, Neutral, $FeSO_4$

ONN — Ora, Neutral, Neutral

E.M— Extraction Media

P.M— post mordanting agent

Chapter One

Introduction

1.1 Background of the study

Human civilization is dependent on nature. From the very early history, plants were used not only to fulfill the basic needs such as food, fiber, fuel, cloths and shelter, but also as sources of natural dyes for dying cloths, design and painting. The art of making natural dyes from plants is an age old tradition in Indian subcontinent. In this region, natural dyes were used to a great extent in textile industries from the 15^{th} to 19^{th} centuries. The invention of synthetic dye by William Perkins in 1856 declined the use of natural dye. Synthetic dyes are more convenient, cheap, provide a wide range of colors and reproducibility, excellent fastness properties and ability to color different types of fiber (Singh and Purohit, 2012; Chan, 2013).

However, recently there has been a revival of the growing interest of natural dyes due to worldwide environmental consciousness (Samanta *et al.*, 2009). The increasing demand of natural dyes in place of synthetic ones is justified by low toxicity. The use of non-toxic and eco-friendly natural dyes on textiles has received significant importance because of increased environmental awareness in order to avoid hazardous synthetic dyes (Rungruangkitkrai and Mongkholrattanasit, 2012). The synthetic dyes are associated with diseases like cancer as well as when released into the environment takes long time to degrade and the intermediates could be still more toxic (Shuhama *et al.*, 2003).

Development of natural dye sectors required potential source of dye yielding raw material especially of plants that are renewable and can be cultivatable. In this context, mangrove plant are reported as to have high tannin content especially in barks (ranging from 15 to 36%) that is traditionally being used for dying fishing net and leather (Chapman, 1970; Duke and Allen, 2006). *Ceriops decandra* is well known for such use. However, there are a good number of plant species that might have potential to contribute in natural dye sector.

Ora *(Sonneratia caseolaris L.)* is a mangrove plant commonly found in mangrove forest. It is commonly found in freshwater and moderately freshwater zone of the Sundarbans as a pioneer species. No available literature has found to explore its dye yielding potential. Therefore, in this study bark of Ora *(Sonneratia caseolaris L.)* was investigated to see the effect of dye.). The extraction media have influence over the type of dye. To further explore the properties of dyeing, thepresent research has investigated the dyeing and fastness properties of Ora (*Sonneratia caseolaris* L.)using different media.

1.2 Objective of the study

1. To review the available literature on plant dye
2. To investigate the effect of dye extracted from Ora (*Sonneratia caseolarisL.*)using acid, base and neutral media.

Chapter Two

Literature Review

In this chapter, available literature on plant dye was compiled. In this compiled information natural dye, its history, advantages, disadvantages, usages, types, extraction method etc. are discussed. Apart from it common dye yielding plants are also listed.

2.1 Natural Dye

Almost any organic material can produce color when boils, but not all can yield a color. Those colorants which can be fixed on the material are known as dyes (Action, 2002).

Natural dyes can be defined as those colorants (dyes and pigments) obtained from plants, animals or minerals. They attach to the substrate by physical adsorption, mechanical retention and formation of covalent chemical bonds or of complexes with salts or metals, or by solution (Visalakshi and Jawaharlal, 2013). Natural dyes are derived from naturally occurring sources such as plants (e.g., indigo and saffron), insects (e.g., cochineal beetles and Lac scale insects), animals (e.g., some species of mollusks or shellfish) and minerals (e.g., ferrous sulfate, ochre, and clay) without any chemical treatment (Chengaiah and Mallikarjuna, 2010).

2.2 History of Natural Dye

Natural color of plants and minerals has been used by human for several centuries. Ancient people used natural colors for decorating their body, making food, coloring animal skin, furs for their cloth and decorative painting on the cave wall. (Christie, 2001in Nor, 2002). The earliest written record of natural dye was found in China dated 2600 BC (Driessen, 2002).

The cave paintings of Cro-Magnon man are the initial evidences of using color. It was dated in between 30000 and 10000 BC (Seefelder, 1994). Pigment fragments found in Egyptian tombs show the use of natural plant dyes to be more than 5,000 years old, and descriptions of dye extraction procedures are found in hieroglyphs (Zollinger, 2004). Throughout time, weaving cultures around the world have used plant and mineral dyes to dye yarn (Opie, 1992).

There is a long history of using natural dye in Indian subcontinent also. In ancient China, India and Egypt, dyers found sources of useful dyes in plants and animals during 4,000 to 3,000 B.C (Ding, 2013). The people of Indian subcontinent used color in many parts of their daily life. The dyeing process was practiced during the Indus river valley civilization at

Mohenjodaro and Harappa (3500 BC) (Siva, 2007). The indigo blue from India was called "King of dyes" due to its high production levels and bright color (Dean and Jenny, 2010). After the invention of synthetic dye by William Perkins in 1856, popularity of natural dye decreased. But, the usage of natural dye is gaining popularity again with the resurgence of new developed technology in hand crafting, most notably in the fields of weaving, spinning, papermaking, leather craft and basketry (Driessen, n.d.).

2.3 Benefits of Natural Dye

In the last few decades, synthetic dyes have been severely criticized and consumers looked forward to natural products (Nagaraj *et al*, 2000). Now people are more conscious about nature, about the environment and themselves.Because of health and hygiene, nutrition, pharmaceutical activities, fashion and environmental consciousness indicate a relative dependency on natural products (Nagaraj *et al.*, 2000). The main reason of gaining popularity of natural dye is its eco-friendly nature and biodegradable Properties (Ding, 2013).

Along with the good coloring property natural dye has some other properties that are very beneficial to our health. Some of its constituents are anti-allergens, hence prove safe for skin and are mostly non-hazardous to human health. (Samanta and Konar, 2011). It is reported that some natural dyes not only dye with unique and elegant colors, but also provide antibacterial, deodorizing and UV protective functions to fabrics (Grifoni *et al.*, 2011; Habbal *et al.*, 2011; Lee, 2007; Lee *et al.*, 2010; Mongkholrattanasit *et al.*, 2011a; Reddy *et al.*, 2012). Ultraviolet radiation (UVR) from the sun causes sunburns, tanning, premature skin aging and wrinkling and its overexposure lead to skin cancer (Narayanan *et al.*, 2010). The sun rays blocking properties of a textile are enhanced when a dye, pigment, delustrants, or ultraviolet absorber finish is present that absorbs ultraviolet radiation and blocks its transmission through a fabric to the skin; thus dyed fabrics protect more than undyed ones and their protection levels rise with the increase in dye concentration (Hustvedt and Crews, 2005).

Natural dyes have a range of shades that can be obtained from insects, minerals, fungi and various plant parts including roots, barks, leaves, flowers, fruits and shells of plants (Allen, 1971; Mongkholrattanasit *et al.*, 2011b; Velmurugan *et al.*, 2005; Kamel *et al.*, 2005; Zheng *et al.*, 2011; Vankar and Shukla, 2011; Vankar *et al.*, 2009). Recently natural colorants have become the major alternatives to synthetic colorants due to good market value (Chattopadhyay *et al.*, 2008). The recent ban on the use of azo dyes by the European Union has also increased the scope for the use of natural dyes (Sivakuma *et al.*, 2010). This is a labor intensive industry, thereby providing job opportunities for all those engaged in cultivation, extraction and application of these dyes on textile, food, leather etc. (Samanta and Konar, 2011).

2.4 Limitations of Natural Dye

Though natural dye is beneficial for health and the environment, but it has some limitations. First of all, synthetic fibers usually cannot be dyed with natural dyes. It is difficult to reproduce shades by using natural dyes, as these agro- products vary from one crop season to another crop season, place to place and species to species, maturity period etc. Secondly, it is difficult to standardize a recipe for the use of natural dyes, as the natural dyeing process and its color development depends not only on color component but also on clarity (Samanta and Konar, 2011). Nearly all-natural dyes with a few exceptions need mordant to fix them. This takes a larger dyeing time and excess cost for mordant and mordanting (Ding, 2013). Moreover natural dying process with mordant generate wastewater containing residual toxic metal ions from metal salt mordants which have a negative impact on the environment and public health (Zheng *et al.*, 2011). Finally, dye isolation and the complexity of the dyeing process make these dyes costly to use in industrial production.

2.5 Application of Natural Dye

Natural dyes are known for their use in coloring of food, leather, wood, as well as natural fibers like wool, silk, cotton and flax since ancient times (Mongkholrattanasit *et al.*, 2011). These dyes are mainly being used for:

- Textile
- Dye sensitized solar cell
- Historical staining
- Antimicrobial finishing of textile
- Deodorizing finishing
- UV protecting cloth

2.6 Classification of Natural Dye

Natural dyes have been classified into different ways (Gulrajani and Gupta, 1992). The earlier classification of natural dye was, according to alphabetical order or according to the botanical names. Later, it was classified in various ways, e.g. on the basis of hue, chemical constitution, application, origin etc. (Samanta and Konar, 2011).

Table 2.1: Classification of natural dye

Basis of classification	Type	Source	References
Hue	Red	Roots, Barks, Etc.	(Samanta and Konar, 2011)
	Yellow	Tesu, Turmeric, Kapila	
	Blue	Indigo And Woad	
	Black	Logwood, Harda	
Chemical Constution	Indigoid dyes	Indigo and Tyrian	(Dedhia, 1998)
	Anthraquinone dyes	Madder, Lacs, Kermes, Cochineal	
	Alphanaphthoquinones	Henna, Juglone	
	Flavonoids	Weld	
	Di-hydropyrans	Logwood, Brazil Wood	
	Anthocyanidins	Leaves Of *Bignonia chica.*	
	Carotenoids	Carrots	
Application	Mordant dyes	Madder, Fustic, Persian, Berries	(Gulrajani and Gupta, 1992)
	Vat dyes	Indigo	
	Direct dye	Turmeric, Harda,	
	Basic or cationic dyes	Berberine	
	Acid dyes	Saffron	
	Disperse dye	Henna	
Origin	Vegetable	Root, Bark, Leaf	(Samanta and Konar, 2011)
	Mineral	Inorganic Compounds	
	Animal	Lac, Cochineal And Kermes	

2.7 Collection and Storage of Natural Dyes

Natural dyes are mostly obtained from various plant parts. These dye-bearing materials contain only a small percentage of dye usually 0.5–5 % (Saxena and Raja, 2014). Therefore after collection, the plant materials are dried in shade or hot air drier in low temperature about 40-50°C temperature to reduce its water content to 10-15%. It can be dried in the sun. Dried material is then powdered to reduce particle size and increase surface area to facilitate better dye extraction. Storage under nitrogen can further prolong their shelf-life (Saxena and Raja, 2014)

2.7.1 Extraction Methods

Raw materials for natural dye contain not only coloring components, but also other constituents like water-insoluble fibers, carbohydrates, protein, chlorophyll, tannins etc. So, extraction is an essential step not only for preparing purified natural dyes, (Saxena and Raja, 2014). Moreover, standardization of the extraction process and optimizing the extraction variables, both, for a particular source natural dye material has technical and commercial importance of color yield and the cost of extraction process as well as dyeing cost (Samanta and Konar, 2011). There are many methods that are used to extract dye like Aqueous extraction,Acid and Alkali Extraction Process, Ultrasonic and Microwave Extraction,Extraction by Non-aqueous and other Solvent Assisted System.

2.7.1.1 Aqueous Extraction

Aqueous extraction was traditionally used to extract dyes from plants and other materials that produce dye. In this method, the dye-containing material is first broken into small pieces or powdered and sieved to improve extraction efficiency. It is then soaked with water in metal vessels (preferably copper or stainless steel) for a long time, usually overnight to loosen the cell structure and then boiled to get the dye solution which is filtered to remove nondye plant materials. The process of boiling and filtering is repeated to extract as much dye as possible. When the extraction is to be carried out on a larger scale for preparation of purified dye powders, stainless steel vessels are used and the time of soaking the materials in water may be reduced by boiling the solution for an extended time period. Generally, centrifuges are used to separate residual matter. Use of trickling filters can ensure removal of fine plant material particles and ensure better solubility of the purified natural dye (Saxena and Raja, 2014).

As most of the dyeing operations are carried out in aqueous media, the extract obtained by this method can be easily applied to the textile materials. Disadvantages of this extraction method are long extraction time, large water requirement, use of high temperature, and low dye yield. Moreover, only water-soluble dye components get extracted whereas many dyes have low water solubility. Also, along with dye, other water-soluble substances such as sugars and the like get extracted that may have to be removed if the extract is to be concentrated and converted to a powder form. Yield of heat-sensitive dye substances gets reduced at boiling temperature; therefore a lower temperature should be used for extraction in such instances. (Saxena and Raja, 2014).

2.7.1.2 Acid and Alkali Extraction

As many dyes are in the form of glycosides, these can be extracted under dilute acidic or alkaline conditions. The addition of the acid or alkali facilitates the hydrolysis of glycosides resulting in better extraction and higher yield of coloring materials (Saxena and Raja, 2014).
Alkaline extraction is suitable for dyes, having phenolic groups as they are soluble in alkali, which improves the dye yield. Dyes can be later precipitated by the use of acids. The disadvantage of this process is that some coloring materials may be destroyed under alkaline conditions considering the fact that some of the natural dyes are pH sensitive.

As natural dyes are usually a mixture of different chemical constituents, changing the pH of the extraction medium by adding acid or alkali can lead to the extraction of different dye constituents which can lead to different hues upon subsequent dyeing and differences in colorfastness properties. Many researchers have studied the extraction of natural dyes under various pH conditions and compared the color and fastness properties of dyed fabric to find out the optimum dye extraction conditions and further additions to this information continue to be made every year in the scientific literature (Samanta and Konar, 2011).

Color from euphorbia leaves (Dixit and Jahan, 2005) under acidic pH by adding hydrochloric acid and under alkaline pH by adding sodium carbonate both, in aqueous media are extracted for dyeing silk fabric. Extraction of color in alkali media from the nuts of *Acacia catechu* (Sudhakar, 2006) is carried out for coloration of protein fiber based fabric.

2.7.1.3 Ultrasonic and Microwave Extraction

In microwave and ultrasound-assisted extraction processes, extraction efficiency is increased by the use of ultrasound or microwaves thus reducing the quantity of required solvent, time, and temperature of extraction. When the natural dye containing plant materials are treated with water or any other solvent in the presence of ultrasound, very small bubbles or cavitations are formed in the liquid. These increase in size, but upon reaching a certain size, they cannot retain their shape. When this happens, the cavity collapses or the bubbles burst creating high temperature and pressure. Millions of these bubbles form and collapse every second. The creation of very high temperature and pressure during extraction increases the extraction efficiency within a short time. Also the process can be performed at lower temperature and therefore extractions of heat-sensitive dye molecules are better. As an exploration of new dye sources and attempts to optimize the dye extraction process is continuing, use of this extraction technique has been recently reported by many researchers (Liu *et al.*, 2009; Mishra *et al.*, 2012; Pradeep *et al.*, 2012; Rahman *et al.*, 2013). In microwave extraction, the natural sources are treated with a minimum amount of solvent in the presence of microwave energy sources. Microwave increases the rate of the processes so the extraction can be completed in a shorter time with better yield. It is reported that the extraction of annatto colorant is possible with microwave energy (Sinha *et al.*, 2013). Earlier their group had reported microwave-assisted extraction of blue pigment from the butterfly pea (Sinha *et al.*, 2012). Microwave and ultrasound extractions can be considered as green processes due to reduction of extraction temperature, solvent usage, and time which results in lower consumption of energy.

2.7.1.4 Enzymatic Extraction

As plant tissues contain cellulose, starches, and pectins as binding materials, commercially available enzymes including cellulose, amylase, and pectinase have been used by some researchers to loosen the surrounding material leading to the extraction of dye molecules under milder conditions. This process may be beneficial in the extraction of dye from hard plant materials such as bark, roots, and the like (Saxena and Raja, 2014).

2.7.1.5 Extraction by Non-aqueous and Other Solvent Assisted System

Due to increasingly stringent environmental regulations, supercritical fluid extraction (SFE) has gained wide acceptance in recent years as an alternative to conventional solvent extraction for separation of organic compound in many analytical and industrial process. In recent past decade, SFE has been applied successfully to the extraction of a variety of organic compounds from herbs, other plant material as well as natural colourant from source natural dye material. With increasing public interest in natural products, SFE may become a standard extraction technique for source natural dye material and other herbs and food items. Supercritical fluid extraction using carbon dioxide as a solvent has provided an excellent alternative to the use of chemical solvents. Over the past three decades, supercritical CO_2 has been used for the extraction and isolation of valuable compounds from natural products.

Extraction of dye from food is best achieved with ethanol/oxalic acid. The comparative behavior of other red food dyes is also studied and a process is developed for the extraction of natural dye from the leaves of teak plant is carried out using aqueous methanol (Nanda *et al.*, 2001). A brick red shade from dyeing for silk/wool using the isolated dye in presence of different mordants is achieved. Attempts (Bhattacharya, 2002; Patel and Agarwal, 2001) has been made to standardize colorant derived from arjun bark, babool bark and pomegranate rind. Extraction (Agarwal *et al.*, 1992; Singh and Kaur, 2006) of well grounded henna leaves, directly in a solvent assisted dyeing process, employing organic solvent: water (1:9) as the dyeing medium is studied and superior dyeing properties are obtained, when applied to polyester. Natural dye (Raja and Kala, 2005) is obtained from the grape skin waste by using soxhlet extractor, and latter on distilled it under vacuum to obtain the concentrated dye solution. Colorant/dye is extracted by using a reflux condenser; source dye material is refluxed for 1 hour and filtered it to yield natural colorant (Eom *et al*, 2001).

2.8 Mordants

All natural dye can't be applied directly to fiber. It needs mordant to fix the color on the fibers and form strong chemical bonds.

Mordants are substances which are used to fix a dye to the fibers. They also improve the take-up quality of the fabric and help improve color and light-fastness (Rees, 1998). The mordant is a sort of bridge between the fiber and the dyestuffs that are extracted from the plants or animals (Bohmer, 2002). The mordant enters deeply into the fiber and combines with dyestuff to form the color (Brennan, 2004).

Table 2.2: Different types of mordants

Type of mordants	Example
Metallic mordants	Alum, Copper, Chrome, Iron, Tin
Tannins and Tannic acid	Hydrolysable tannins, Condensed tannins
Oils type mordents	Vegetable oils, Turkey red oil (TRO)

2.9 Mordanting Methods

There are three types of methods for application of mordants based on the time of their usage (Saxena and Raja, 2014). They are:

1. **Premordanting:** As suggested by the name, in premordanting, the mordants are applied to the fabric prior to dyeing. It is most common for cotton and cellulosics as in the unmordanted state they do not have affinity for many natural dyes.
2. **Postmordanting :** In the postmordanting method, the fabric after dyeing is treated with mordant in a separate bath. The final color is developed during the last phase. Iron salts are very often applied in this manner for producing grey and black colors.
3. **Metamordanting or simultaneous mordanting**: In the metamordanting or simultaneous mordanting method, both dyeing and mordanting processes are carried out in the same bath itself. Usually for cotton and cellulosics, mordant is also added to the dye bath at the start of dyeing so that both dyeing and mordanting processes take place simultaneously in the same bath.

2.10 Common Dye Plants in Bangladesh.

There are numerous plants in Bangladesh which is used for plant dye extraction.

Table 2.3: Scientific and local name of dye producing plants of Bangladesh

Local Name	Scientific Name
Zafran	*Crocus sativus*
Bokkan	*Haematoxylon campechianun*
Chandon	*Santalum album*
Kushum Ful	*Carthamus tinctorius*
Babul	*Acacia hexandra*
Khoyer	*Acacia catechu*
Lotkon	*Bixa orellana*
Neel	*Indigofera Sumatra and Indigofera arekta*
Bokom	*Caesalpinia sappan*
Divi divi	*Caesalpinia coriaria*
Supari	*Areca catechu*
Gab	*Diospyros peregrine*
Amloki	*Phyllanthus emblica*
Bokul	*Mimusops hexandra*
Bohera	*Terminalia belerica*
Jhau	*Casuarina littorea*
Dak	*Butea monosperma*
Tun	*Toona ciliata*
Hena	*Lawsonia inermis*
Daruhoridra	*Berberis asiatica*
Kamela	*Mallotus phillippinesis*

Chapter Three

Materials and Methods

3.1 Sample Collection

Bark of Ora (*Sonneratia caseolaries L.*) was collected from the Sundarbans mangrove forest by strips from different trees.

3.2 Sample Preparation

After collecting, the sample was dried in the room temperature. After drying, all the bark was grinded using a grinder. Then all the powdered materials were screened using common kitchen mesh. The screened powders were stored in an airtight container with appropriate labeling.

3.3 Chemical Preparation

In this experiment dye extraction was carried in three media: distilled water, alkali solution and acidic solution. Distilled water was considered as control. These three media were considered as three treatments. Each treatment was replicated for three times. 0.1M Sodium hydroxide (NaOH) and Acetic acid (CH3COOH) solution were prepared for alkaline and acidic solution respectively (Furry and Viemont, 1935).

3.4 Pre Mordanting

Mordants are essential to fast dyes with fabric. Alum was used as a pre-mordanting agent. For pre-mordanting, fabrics were soaked in 135 ml distilled water with 1.25gm alum and boiled for 30 min with 70^{0} C temperatures.

3.5 Extraction of Dye and Dying

The dye was extracted using three media, i.e. acid, base and distilled water. 200ml of each media was taken in an oven proof beaker. 25gm of powdered bark was poured into the beaker and stirred gently with a glass rod. In case of acidic or basic media, 0.1M of acidic acid (CH_3COOH) and sodium hydroxide (NaOH) solution was used respectively. The amount of solvent and solute was determined according to (Furry and viemont, 1935). Each medium was put into a halogen hotplate for (30) minutes for boiling with $(70\text{-}80)^{0}$C temperature. Glass beads were used to prevent foaming of liquid dye solution.Extracted dye was used for dying of fabrics. Fabrics of pure cotton, wool and silk were collected from local market. Fabrics were washed using soft detergent to get rid of any filler or starch material and dried in room temperature. Before dipping fabrics into dye solution, pre mordanting was carried out using alum. The dye solution was filtered and the cloth from alum solution was put into filtered dye liquid. The beaker with liquid dye and cloth was put on hot plate for 1 hour at $(70\text{-}80)^0C$ temperature.

3.6 Post Mordanting

Copper sulphate and Ferous sulphate were used as post mordanting agents to discover shades of color. 0.1g copper sulphate and Ferous sulphate were mixed with 250ml distilled water respectively. The cloths from dye solution ware rinsed and then put into the respective post mordanting solution. The solutions with the cloths were boiled for 30 min with $(60\text{-}70)^0C$ temperature. After boiling it was kept for overnight.

3.7 Washing and Drying

After dying process all cloths were washed with tap water first and then with soft detergent and dried at room temperature for washing fastness test.

3.8 Color identification

Color was identified using color picker of graphics software. To determine the color and effect of natural dye, different fabrics such as cotton, silk and wool were used. At first fabrics were dyed with natural dye extracted in different extraction media and with or without post mordanting treatment. After drying the fabrics were scanned. After that using Gimp2 software, RGB and CMYK color model were prepared. Acid (CH_3COOH), Base (NaOH), and Neutral (Distilled water) were use as extraction media. Ferus Sulphate and Copper Sulphate were used as post mordanting agent. RGB and CYMK values were recorded for each sample. Color naming is done using hex code obtained from the software.

3.9 Wavelength analysis

UV visible spectra of any dye show its peaks at predominating wavelength, indicating the main hue. For natural dye, spectra specially indicate the peak available in visible region. Various absorbances in different wavelength was measured by using photospectrometer model U-2900 Spectrophotometer and different color and values at different length were found. Scan speed was 100 nm/min.

Chapter Four

Result and discussion

4.1. Determination of Color

To determine the obtained color of the natural dye, two methods were used. The first one is graphic software based where RGB and CMYK models were used to determine the color. Another method is spectrophotometry test.

4.1.1 Wavelength Analysis

To get the peak and absorbance in different wavelength of natural dye, spectrometry test was conducted.

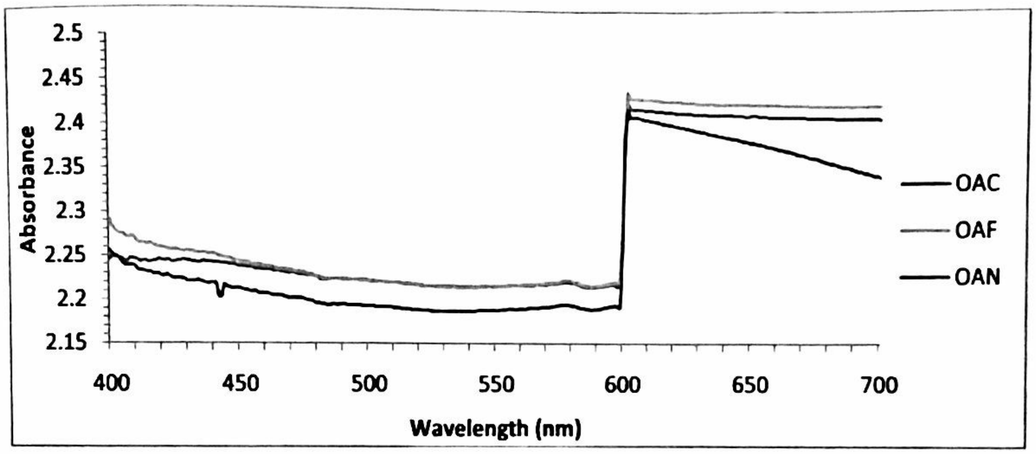


Fig 4.1: The peak points and absorbance of color in visible wavelength in acidic media

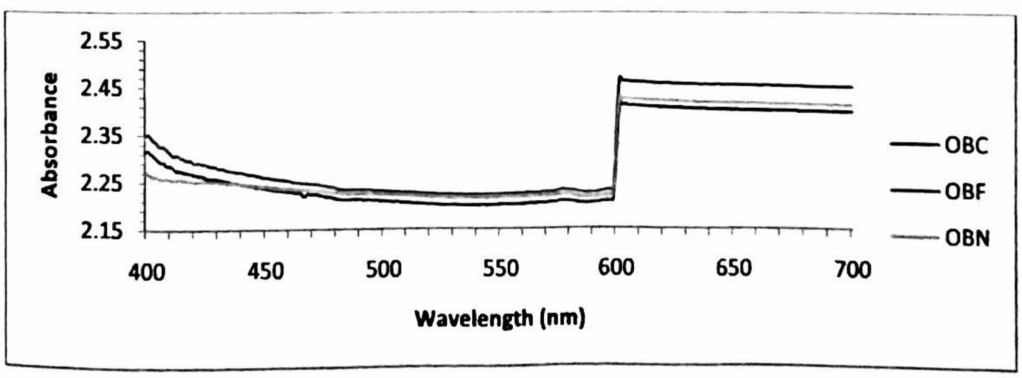


Fig 4.2: The peak points and absorbance of color in visible wavelength in basic media

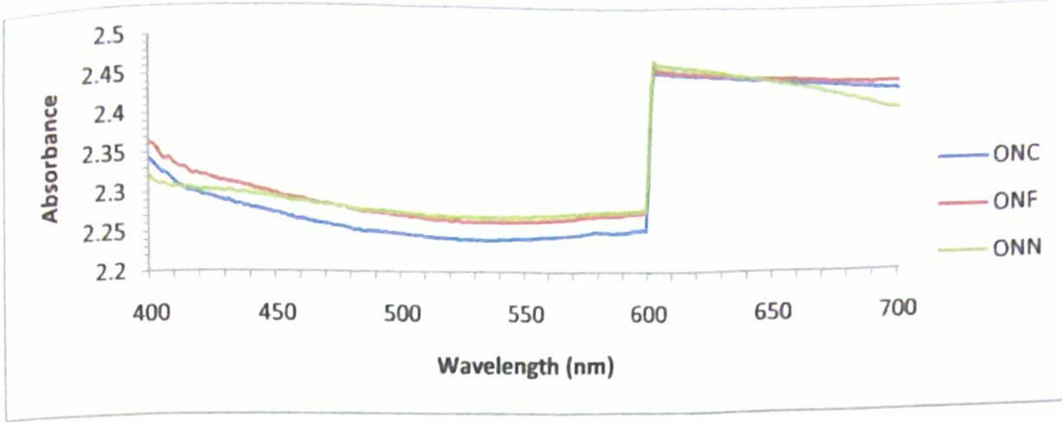


Fig. 4.3: The peak points and absorbance of color in visible wavelength in neutral media

From the test it is observed that peaks were the same in all extraction media and it was found at the wavelength of 602. Natural dye from Ora gives an orange color in all extraction media. But, absorbance at the peak point was different due to using different post mordanting agents. For acidic extraction (Fig.4.1), absorbance at the peak points were 2.425 for copper sulphate, 2.438 for ferrus sulphate and 2.417 absorbance were found where no post mordanting agent was used. For basic extraction media (4.2) 2.419 for copper sulphate, 2.496 for ferus sulphate and without post mordanting agent 2.431 absorbance were found in peak point. On the other hand for the neutral extraction media (Fig.4.3), absorbance at the peak points were observed 2.461 for copper sulphate, 2.464 for ferus sulphate and 2.47 absorbance was found where no post mordanting treatment was used.

4.1.2. Determination of Color with RGB and CMYK Color Model

Red, Green and Blue are the basic colors. In the RGB color model the combination of these three colors is used to determine the color. This ranges from 0 to 255. Where the lower RGB combination indicates the darker color and the higher RGB combination denotes the lighter color. The CMYK color model was used to indicate the mixture of four colors: Cyan, Magenta, Yellow and Black required during printing. In the CMYK color model, proportionate mixture of CMYK determines the ultimate color. In this color model 'K' is denoted as 'key color' that is black in nature. CMYK combination ranges from 0 to 100%. Where the lower combination indicates the lighter color and higher value indicates the dominant color for printing.

4.1.2.1RGB and CMYK Color Model for Neutral Extraction

Distilled water was used as the extraction media for neutral extraction. Obtained colorswere different in different fabrics for neutral extraction.

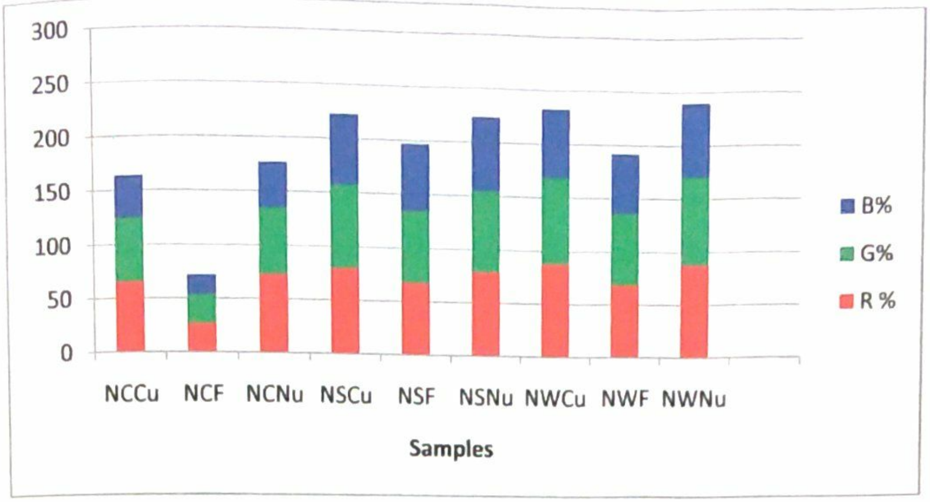


Fig. 4.4: RGB color model for neutral extraction.

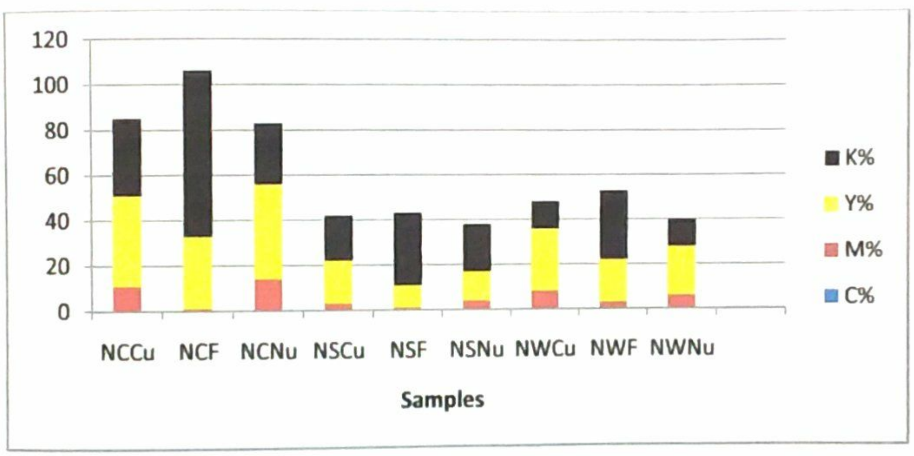
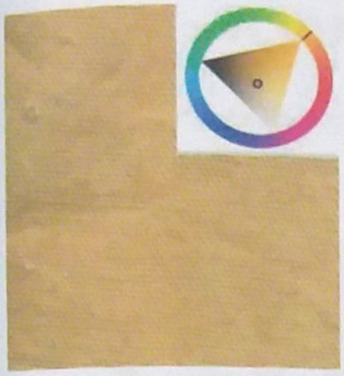
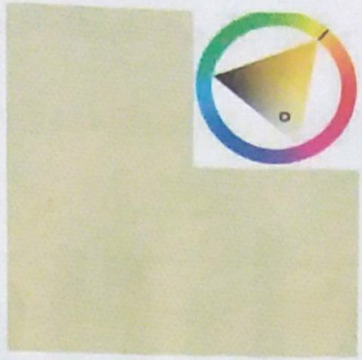


Fig 4.5: CMYK color model for neutral extraction.

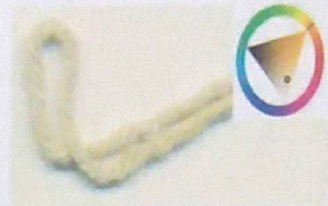
Both RGB and CMYK correspond with each other. For neutral extraction, RGB combination is lower indicating darker color. CMYK proportion (Fig. 4.2) indicates the mixture required while printing to obtain the color found in RGB model. In CMYK model key color (black) is dominant along with yellow. Wherever Ferrus sulphate has been used as post mordant, key color has increased. For both silk and wool fabric, a little darker color is formed only when ferrus sulphate use as a post mordnting agent. Rest of the fabric produces very light color.



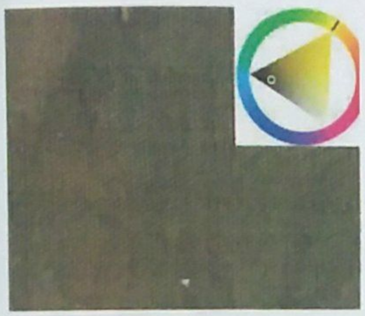
Cloth: Cotton,E.M: Water, P.M: Copper sulphate, Color: Light taupe



Cloth:Silk,E.M: Water, P.M: Copper sulphate, Color: Pale silver



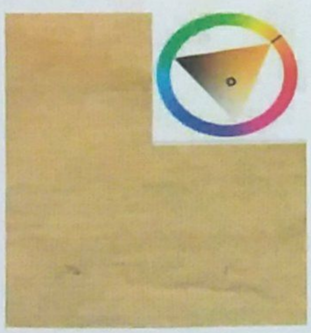
Cloth:Wool ,E.M: Water, P.M: Copper sulphate, Color: Desert sand



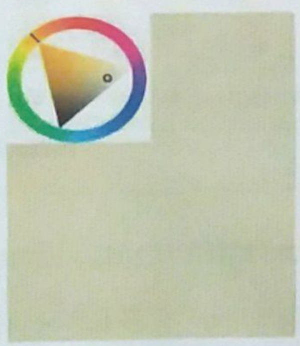
Cloth: Cotton,E.M: Water, P.M: Ferus sulphate, Color: Rifle green

Cloth: Silk,E.M: Water, P.M: Ferus sulphate, ColorDark gray

Cloth:Wool,E.M: Water, P.M: Ferus sulphate, Color:Khaki



Cloth: Cotton, E.M: Water, P.M: No, Color: Camel



Cloth: Silk, E.M: Water, P.M: No, Color: Pale silver

Cloth: Wool, E.M: Water, P.M:No,Color:Desert sand

Fig. 4.6: Color obtained in different fabrics in neutral extraction media (with color wheel)

4.1.2.2RGB and CMYK Color Model for Acidic Extraction

Acetic acid (CH_3COOH) was used as an acidic extraction medium.

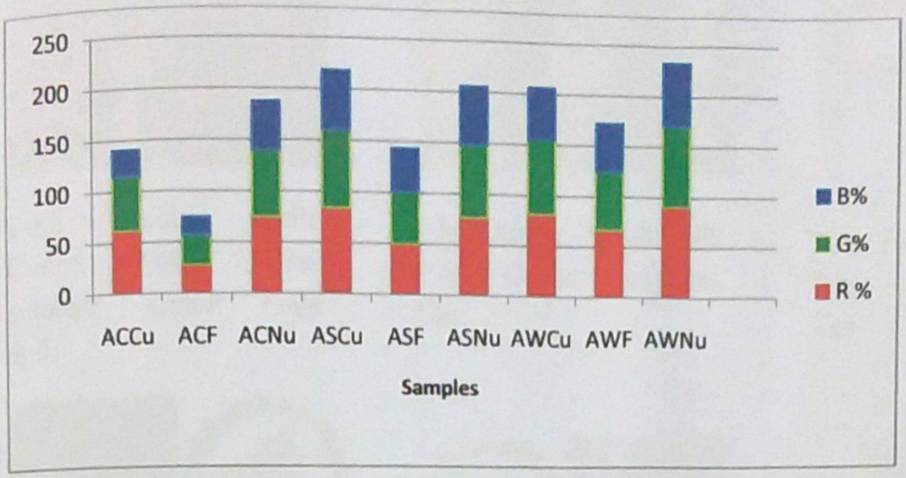


Fig. 4.7: RGB color model for acidic (CH_3COOH) extraction.

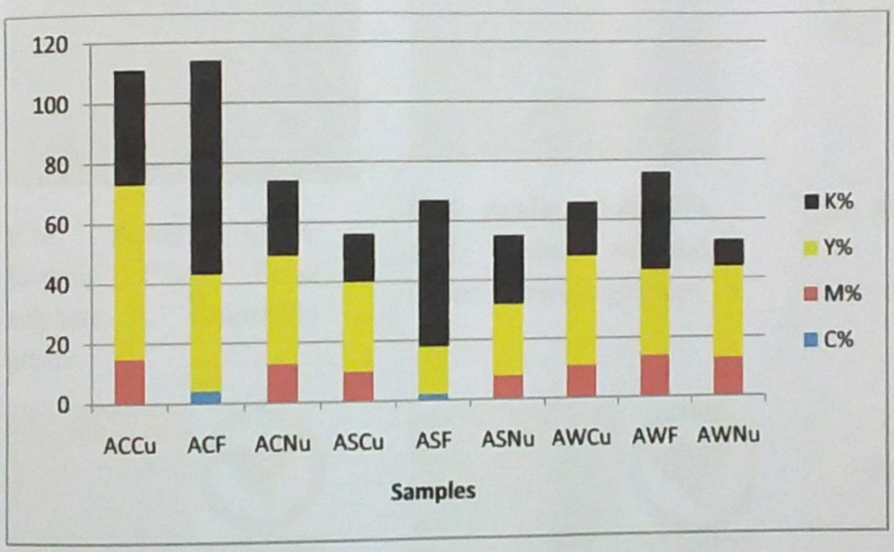
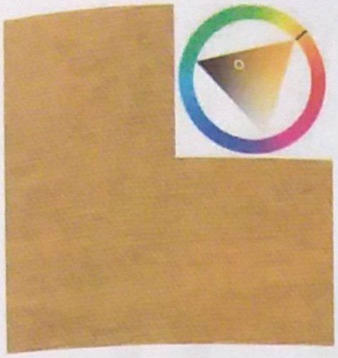
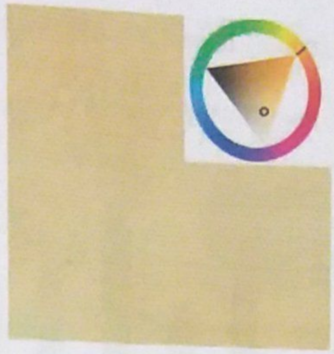


Fig. 4.8: CMYK color model for acidic (CH_3COOH) extraction

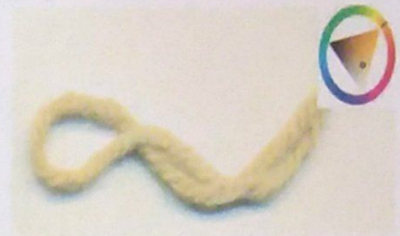
In case of acidic extraction, it is seen that RGB combination is lower in cotton fabrics (Fig 4.7). In CMYK color model, the presence of high value of key color indicates ferrus sulphate as mordant. The darkest color we found at the cotton fabrics. Some fabrics produced lighter color like silk in ferus sulphate, wool in copper sulphate and wool using no post mordanting agent.



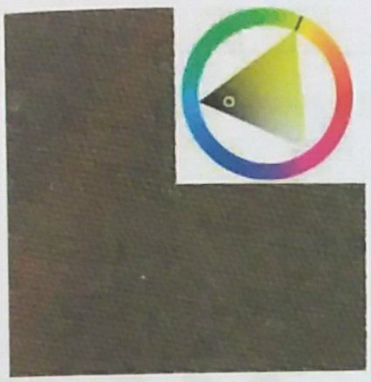
E.M: Acidic, Cloth: Cotton P.M: Copper sulphate, Color: Field drab



E.M: Acidic, Cloth: Silk P.M: Copper sulphate, Tan



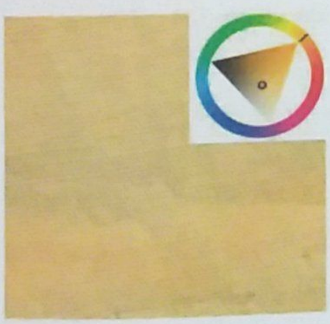
E.M: Acidic, Cloth: Wool P.M: Copper sulphate, Tan



E.M: Acidic, Cloth: Cotton, P.M: Ferus sulphate, ColorRifle green

E.M: Acidic, Cloth: Silk P.M: Ferus sulphate, Color: Camouflage green

E.M: Acidic, Cloth: Wool P.M: Ferus sulphate, Color: Grullo



E.M: Acidic, Cloth: Cotton P.M: No, Color:Pale taupe

E.M: Acidic, Cloth: Silk P.M: No, Color: Kh

E.M: Acidic, Cloth: Wool P.M: No, Color: Desert sand

Fig. 4.9: Color obtained in different fabrics in basic (NaOH) extraction media (with color wheel)

4.1.2.3RGB and CMYK Color Model for Basic extraction

Sodium hydroxide (NaOH) was used as a basic extraction medium.

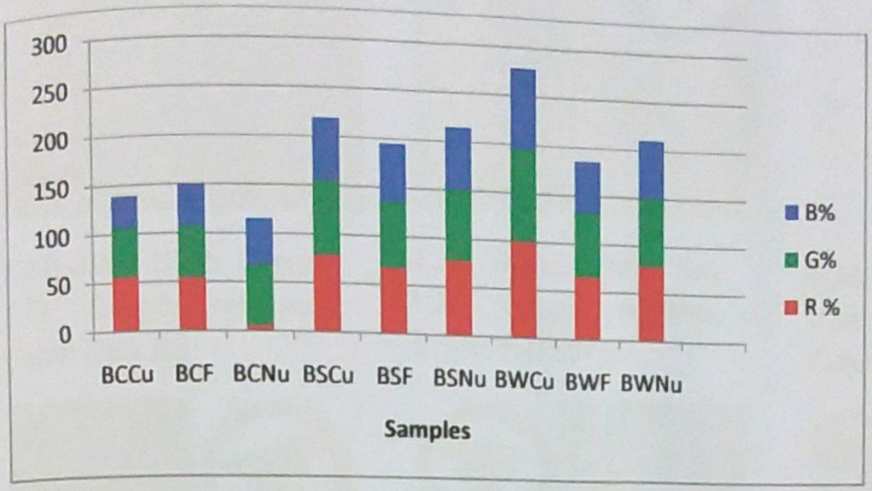


Fig. 4.10: RGB combination for basic (NaOH) extraction.

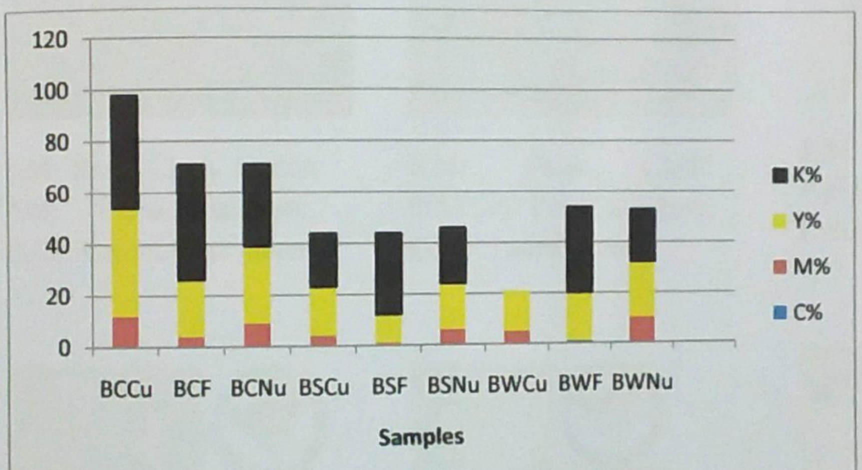
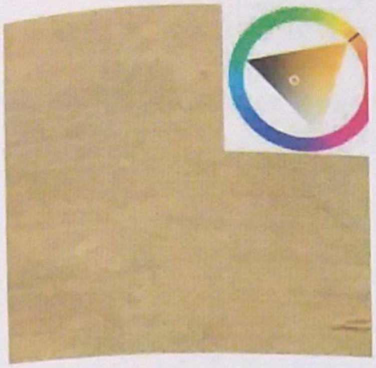
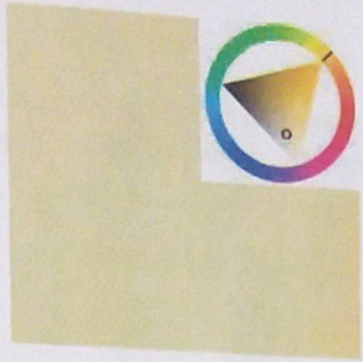


Fig. 4.11: CMYK combination for basic (NaOH) extraction

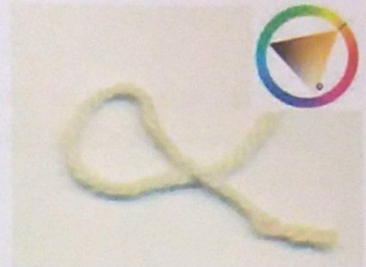
In case of basic media RGB combination is lower and CMYK percentage is higher for cotton fabrics. That produces darker color. In case of wool and silk, key color of CMYK was increased where ferus sulphate is used in post mordanting agent. The rest of the fabrics produce lighter color. It is seen that, RGB combination is higher and the key color of CMYK is absent in wool with copper sulphate post mordanting that makes almost no color.



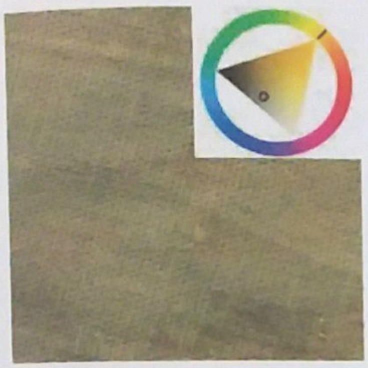
E.M: Basic, Cloth: Cotton P.M: Copper sulphate, Color: Dark tan



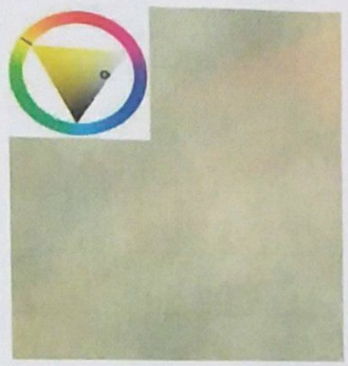
E.M: Basic, Cloth: SIlk P.M: Copper sulphate, Color: Pale silver



E.M: Basic, Cloth: Wool P.M: Copper sulphate, Color: Papaya whip

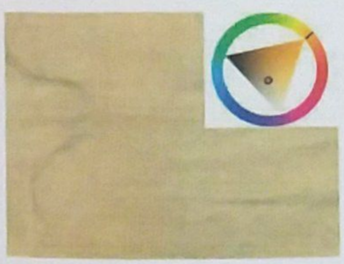


E.M: Basic Cloth: Cotton P.M: Ferus sulphate, Color: Camouflage green

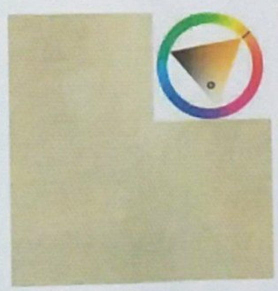


E.M: Basic Cloth: SilkP.M: Ferus sulphate, Color: Laurel green

E.M: Basic Cloth: Wool, P.M: Ferus sulphate, Color: Grullo



E.M: Basic, Cloth: Cotton P.M:No, Color: Grullo



E.M: Basic, Cloth: Silk, P.M:No, Color: Khaki

E.M: Basic, Cloth: Wool, P.M:No, Color: Khak

Fig. 4.12: Color obtained in different fabrics in basic (NaOH) extraction media (with color wheel)

4.2 Wash Fastness test

All cloths ware washed with soft detergent to test the fastness property of the dye on cloth. From this test it was observed that cotton retained more color in acidic media than neural and basic media.

Chapter Five

Conclusion and Recommendations

5.1 Conclusion

From this test it is seen that dye extracted in every media and post mordanting treatment gives orange color. This research shows that, bark of Ora (*Sonneratia caseolaris)* is capable of producing dye in all extraction media. Neutral (distilled water) and acidic media has almost the same effect of dye extraction. But, acid gave the best result as extraction media. This media produced shade in all fabrics. Cotton gave good darker color in all dye extraction media. Silk and wool give darker color when it is treated with ferus sulphate as a post mordanting agent. The dye has good scope in the commercial dyeing of cotton with acidic extraction media. Different color shade can be achieved using different post mordanting agents.

5.2 Recommendations

There is a great opportunity of using Ora bark for natural dye extraction. However, the following issues need to be investigated

- Natural pre mordanting and post mordanting agents should be tried to make the production chain natural.
- Amount of extraction medium and dyeing materials along with cooking time should vary and optimized for better production.

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