EXTRACTION AND APPLICATION OF SOME PLANT DYES AS COLOURANTS FOR FOOD AND TEXTILES

BY

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(NSU/M.Sc/ICH/0004/17/18)

M.Sc. INDUSTRIAL CHEMISTRY

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A DISSERTATION SUBMITTED TO THE SCHOOL OF POST GRADUATE STUDIES, NASARAWA STATE UNIVERSITY KEFFI, IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF MASTER OF SCIENCE DEGREE IN INDUSTRIAL CHEMISTRY

DEPARTMENT OF CHEMISTRY
FACULTY OF NATURAL AND APPLIED SCIENCES
NASARAWA STATE UNIVERSITY, KEFFI NIGERIA

DECLARATION

I hereby declare that this dissertation has been written by me and is a report of my research work. It has not been presented in any previous application for Master Degree in Industrial Chemistry. All quotations are indicated and sources of information specifically acknowledged by means of references

....   22/07/2021 ....

Idzi, Vincent Danjuma   Date
(NSU/M.Sc/ICH/0004/17/18)

CERTIFICATION

This dissertation title "Extraction and Application of some plant Dyes as Colourants for Food and Textiles" meet the regulation governing the award of Master Degree in Industrial Chemistry, of the School of Postgraduate Studies, Faculty of Natural and Applied Sciences, Nasarawa State University, Keffi, and is approved for its contribution to knowledge.

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DEDICATION

This research work is dedicated to my lovely family: Grace my wife and my children: Pricilla, Collins and Conquer, my mother Talatu and my late father Danjuma, for their unwavering love, care, prayer, support, understanding patience and tolerance during the cause of my study and also my long hour of working on the computer even at odd time.

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CHAPTER ONE

INTRODUCTION

1.1 Background to the Study

World interest in natural dyes has increase, due to the awareness of the environmental and health hazards associated with the synthesis, processing and use of synthetic dyes. Natural dyes are made up of colourants that are obtained from animal or vegetable matter without any chemical processing. Recently, the use of natural dyes obtained from animal or vegetable matter without undergoing chemical processing, has gained momentum as a result of increase in demand for these dyes by the pharmaceutical, cosmetic, food, as well as the textile industry.

Textile processing industry is among the major environmental polluters. In order to process a ton of textile, it is possible 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 (Al-Sehaibani, H. 2000). Over 7×10^5 tonnes and approximately 10,000 different types of dyes and pigments are manufacture world-wide annually. It is estimated that 10-15% of the dye is lost in the flow of the liquid waste during the dyeing process (Iqbal and Ashiq, 2007). Thus, there are two main ways to reduce the environmental impact of textile processing. One is to construct sufficiently large and very effective liquid waste treatment plants, and the other way is to make use of dyes and chemicals that are environment friendly.

Natural dyes are mostly environmentally friendly, biodegradable, less toxic, and less allergenic as compared to synthetic dyes. However, studies have shown that certain natural dyes may have significant mutagenic effects e.g., Elderberry colour 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 (Han and Yang, 2005; Hill, 1997).

However, in spite of the advantages 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 do not have the ability to stick to the fibre 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 fibre and which also combines with dyestuff. A link is formed between the fibre and the dye, which allows certain dyes with no or little affinity for fibre to be fixed (Gulrajni, 1992).

Although natural dyes have several advantages, there are also some limitations as well. These are as follows:

i. Strenuous extraction of colouring component from the raw materials with low colouring component.
ii. Limited number of suitable dyes, allow only dyeing cotton, natural silk, linen and wool
iii. Low fastness properties in comparison with chemical dyes.
iv. Great difficulty in blending natural dyes.
v. Non-standardized methods of extraction (Teli et.al., 2000; Bhuyan et.al., 2004)

Thus by keeping in view all above disadvantages, this research considered the method of improving colour yield of natural dyes using environment friendly extraction procedure (extraction in aqueous alkaline medium), other than the extensive application of organic solvents was adopted. This medium was chosen in view of the acidic chemical nature of colouring components of plants. This will help to make natural dye as co-partner of reactive dyes.

Furthermore, the dyeing and mordanting characteristics of colouring matter on cotton will also be examined. Finally, comparative studies will also be conduct between the dyeing with optimized alkaline extracts of these plants and synthetic dyes and evaluated the optimum conditions as well. The following plants: Henna, Neem, Rossell, Euclayptus and Turmeric, were selected according to their availability and usefulness in the region.

1.2 Statement of the Problem

From the ancient time, the roots, seeds, leaves, bark and flowers of plants and also mineral matter have been used to dye textiles. Natural dyes or colourants are used in the food manufacturing industry with the main motive of making their products very attractive. Synthetic dyes become popular during the late 1800s and 1900s, and were widely used because of their property of better colour fastness. The industry now uses a wide variety of these synthetic dyes. As a result, the natural dyes which are less harmful are no longer used while more chemical dyes are used. These toxic materials pollute the environment and create more hazards to life.

The adoption of synthetic dyes in Nigeria has drastically reduced interest in exploration of plant dyes from our environment. This is because, one can get the synthetic dyes which have better fastness property easily on the market instead of going through the long process of walking through the forest to collect the plants from which dyes are extracted. As though this was not enough, synthetic food colourants are being used to colour food items like fried yam and turkey tail, beef, bread, pig feet, and pastries which are sold in the market. Even though these synthetic dyes come in handy, they are expensive and may be environmentally unfriendly.

Furthermore, educational institutions located in rural communities where commercial dyes are unavailable for lack of suppliers or inaccessible for lack of funds to purchase them, could use the resourcefulness of our natural environment; thus coming out with their own dyes from flora available in their localities. This dissertation is intended to serve as reference material for practical Chemistry work. Also, not paying attention to the extensive possibilities and creative capacity of the Chemistry practitioners to develop technical skills through the use of the material resources in the natural environment will jeopardize the classroom setting, which is expected to be a field with the right kind of natural chemical extract experiences needed to obtain the objective of self expression among students.

It is therefore useful in this regard that, research be carried out into basic ways of the extraction and application of dyes from local plants found in the environment as substitute for synthetic colourants used in the textile and food industries, which will not be harmful to the human system. In addition, this research will do well to alleviate boredom and create versatility in the Chemistry setting by educating urban dwellers and students of Chemistry on the processes involved in obtaining and producing natural dyes from the environment, including the forest reserves.

1.3 Aim and Objectives of the Study

The aim of this research work is to extract and apply some plant dyes as colourants for food and textiles.

The objectives of the study are:

i. To extract and apply some plant dyes to serve as colourants for food and textiles.

ii. To test the properties of the plant dyes and introduce them to food products and textile fabrics.

iii. To study the characteristics of some plants dye (Natural dye) and compare to existing synthetic dyes and their effect on food and textiles.

iv. To consider the method of improving colour yield of natural dyes using environment friendly extraction procedure

1.4 Significance of the Study

This work seeks to investigate and capture some natural plants from which dyes can be extracted and applied to selected foods and textile fabrics. The study also plays a significant role in providing beneficial information to the textile and food industries, science lecturers, students and scholars. In addition, the study would create the awareness of unexplored plant dyes and finally serve as a reference material to other research works.

1.5 Scope of the study

The study was limited to exploring the natural plants from which dyes can be extracted and used as colourants for selected food items and textile fabrics. Although there are different food additives (Nutritional Supplements, Flavours, Colouring agents, Preservatives, Emulsifiers, Stabilizers and Thickeners, Acids and Alkali) that are used to enhance food, the study was limited to colouring agents.

1.6 Definition of Terms

Adhere: The ability for a dye to stick firmly to a substrate (surface of a material).

Affinity for dye: The ability for a fibre or fabric to attract dye and exhaust it.

Bleed: To spread colour from one area of a substrate to another area, bleeding may produce both colour loss and staining.

Crack: To transfer colour as a result of abrasion or rubbing.

Exhaustion: The amount of transfer of dye from the bath to the fibre, either by, adsorption or absorption.

Dye stuff: To change the colour of something by using a special liquid. The substance has the ability to change the appearance of a substrate.

Dye liquor: This is a solution or liquid mixed with colour to enable dyeing.

Fabric: This is formed by assembling yarns and or fibres into one cohesive Structure. The most common fabric structures are woven, knit, and non-woven. Fabric may be referred to as cloth, material or piece.

Fade: A fabric is said to fade when it loses colour or runs out during washing.

Fastness: The resistance of a material to change in colour characteristics.

Fibre: These are fine hair-like substances. They may be natural or manufactured and are the smallest components of a textile product. Cotton is an example of a natural fibre while polyester on the other hand is a manufactured fibre.

Finish: This is any chemical or mechanical treatment or process that modifies the properties of textile products.

Frost: Loss of colour resulting from localized abrasion.

Migrate (Shift colour): this occurs when moisture lifts colour and deposits it in another area. This is most seen in the underwear area.

Textile: It is a broad classification of materials that can be utilized in constructing fabrics, including textile fibres, and yarn. It is also used to designate the constructed fabric including woven, knitted, and non-woven structures as well as lace and crocheted goods.

Yarn: Yarns are groups of natural or manufactured fibres that are combined to form a continuous strand that can be used to produce fabric.

The dyeing property as well as the UV light absorption, antibacterial, antispasmotic and corrosion inhibitor were attributed to the presence of Lawsone, 2 hydroxy-1,4-naphthaquinone. This colouring component has the following structural formula with colour index number 75486 (Al-Sehaibani, 2000; Dweck, 2002).

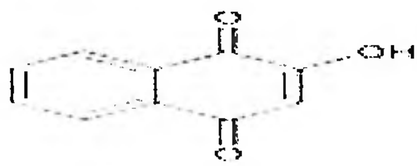
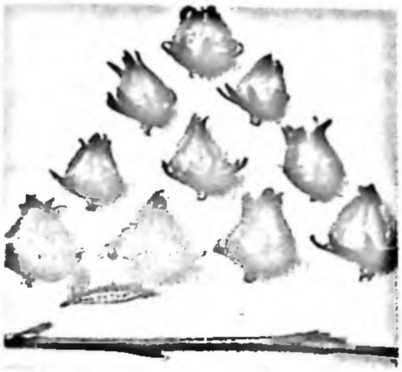


Figure 2.1 2-Hydroxy-1,4-naphthaquinone

2.1.2 Rosselle (*Hibiscus sabdariffa*)

Hibiscus sabdariffa is a species of hibiscus, native to the old world tropics, used for the production of bast fiber and as an infusion (herbal tea). The plant is an annual or perennial herb or woody-based sub-shrub, growing up to 2-2.5 m (7-8 ft) tall, (Falusi, 2007). Hibiscus sabdariffa is cultivated for leaf, seed, fleshy calyx or fiber. It is highly cultivated and distributed in the northern part of Nigeria because of favourable climate. Hibiscus sabdariffa is found in tropical and subtropical regions of the world, it is cultivated for its fiber with characteristics similar to those of hemp or jute which is used to make cloth, rope and in Polynesia, grass skirts. Hibiscus sabdariffa 'Zobo' drink has a shelf of life normally 24 to 48 hours after which it begins to deteriorate. It is best preserved by refrigeration which will control the microorganism reaction on the drink. Due to the ubiquitous nature of micro-organisms, the level of contamination can be reduced if proceeded under standard hygienic conditions and at low temperatures to prevent the multiplication of pathogenic micro organisms that can cause infection to the consumers.



A

B

Plate 2.2 (A&B) Hibiscus sabdariffa

2.1.3 Neem Plant (*Azadirachta indica*)

It is popularly known as the miracle tree. It is known as dogon yaro in northern Nigeria. The Sanskrit name of Neem is Arishtha meaning the reliever of the sickness. It is a tall evergreen tree with the small bright green leaves. It is up to 100 feet tall. It blossoms in spring with the small white flowers. It has a straight trunk. Its bark is hard rough and scaly, fissured even in small trees. The colour of the bark is brown greyish. The leaves are alternate and consist of several leaflets with serrated edges. Its flowers are small and white in colour. The olive like edible fruit is oval, round and thin skinned. The World BookEncyclopedia(2001:212).

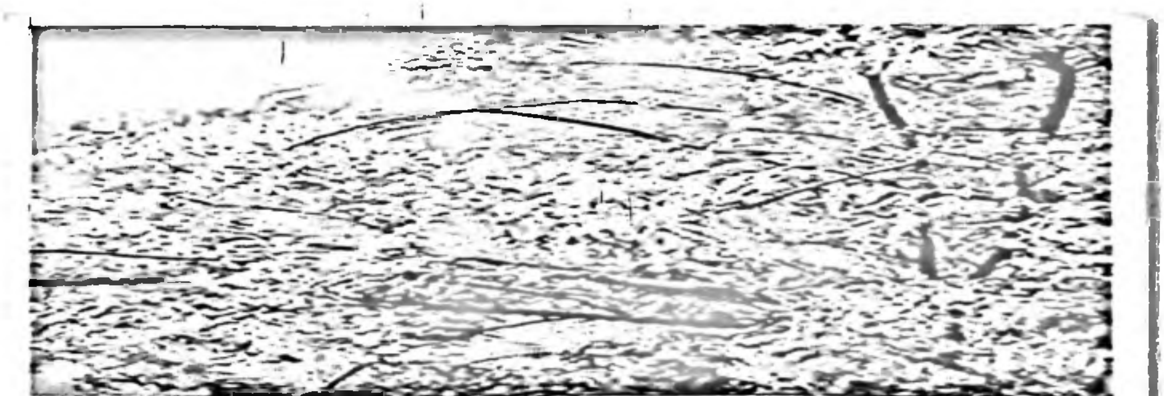
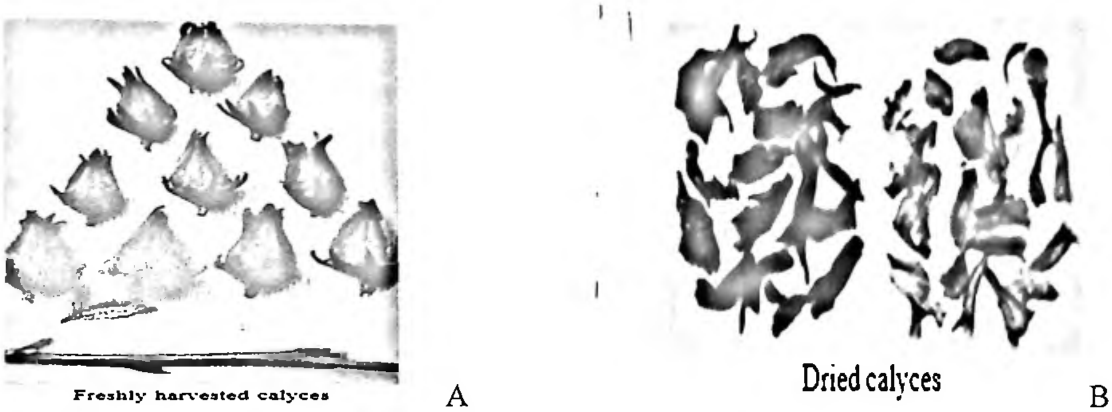


Plate 2.3 Neem plant



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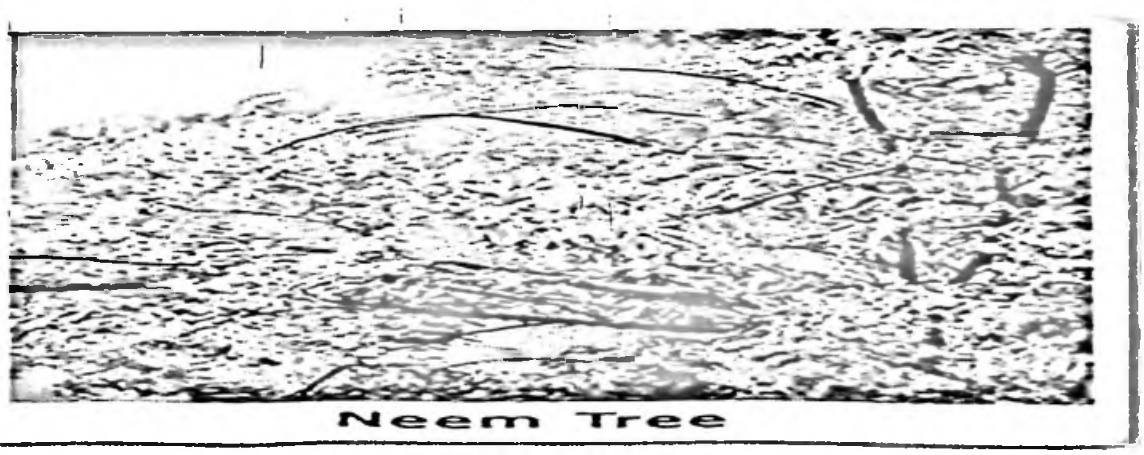

Plate 2.3 Neem plant

2.1.4 Turmeric (*Curcuma longa*)

Turmeric (*Curcuma longa*) is a plant native to south India and Indonasia. It is also cultivated in Nigeria, China and the whole of South East Asia. It is also called "Haldi". Its tuberous rhizomes have been used as a condiment, a colourant and an aromatic stimulant since antiquity. Turmeric consists of various molecular constituents, including three gold colour alkaloidal curcuminoid, curcumidesmethoxy curcumin and bisdemethoxy curcumin. The curcuminoid content responsible for colour, depends upon the turmeric variety and within a variety on the maturity at harvest. It may be present to the extent of 4 to 8 % in turmeric harvesting at the right maturity being an important factor for colour and aroma. (Han and Yang, 2005)

Curcumin has anti-inflammatory, antifungal and anti-tumorous. It is also widly used as food additives. WHO (World Health Organization) and FAO (Food and Agricultural Organization) committees have approved it as food additive, (Han and Yang, 2005). The plant of turmeric is show in Plate 2.4.

Plate 2.4 Turmeric Rhizome

2.1.5 Euclayptus (*Eucalyptus grandis*)

Euclayptus grows on desert swamp places, valleys and mountains. Its trees are characterized by their leathery, whitish leaves with a peculiar aroma (Plate 2.5). Euclayptus bark is one of the most important sources of yellowish brown colourant. The colouring matters of Eucalyptus consist of natural Tannin, Eriodictyol, Narinjemin, Quircetin, Rhamnazin and Rhamnatic. The major colouring component of Euclayptus bark is Quercetin which is also an antioxidant (Vankar, 2002). The Euclayptus trees are show in Plate 2.5 .

Plate 2.5 Eucalyptus Trees

2.2 Colour in textiles

Dyes, also referred to as dyestuffs, are the most common way to add colour to fibres, yarns and fabrics. Manufacturers of food, fur, ink, leather, paper, plastics and wood also use dyes (World Book Encyclopedia, 2001) to colour their products. The appeal of colour is universal. According to Kadolph (2007), colour is one of the most significant factors in the appeal and marketability of textiles products, consumers generally expect two things: aesthetically pleasing colours and prints; and colour permanence, implying that the aesthetic aspects of a textile fabric is the prime consideration for consumers. According to Adu-Akwaboa (1994), colour is often the primary consideration in the selection and purchase of clothing and other household textiles and to a large extent, the most exciting thing about textiles.

Kuehni (2004) asserts that for the normally sighted, colour is everywhere, in the interior of a dwelling, natural and stained wood, wallpapers, upholstery fabrics, pottery, paintings, plants and flowers. Collier and Tortora (2001) share the view that most objects made by human beings are decorated in some way and that the decoration of textile fabrics may be achieved by varying the construction of the fabric and adding colour through dyeing or printing. The World Book Encyclopedia (2001) also reports that besides textiles, manufacturers of food, fur, ink, leather, paper, plastics and wood also use dyes to colour their products.

2.3 Dyes

Over the years, dyes have been used to colour textile fabrics, fibres, yarns and other non-woven items. Dyes are applied to textiles in the form of a solution or paste just as manufacturers add dyes to colour food items to either conceal the original colour or enhance their colours to make them attractive to buyers. With respect to textile products, colour is accepted as a common language that consumers are usually more concerned with than any other characteristic. The desire for colourful textiles led to the discovery of synthetic dyes and the manufacture of dyestuff and chemical agents that produce long lasting colours in materials. Colour for the textiles industry is mostly obtained from dyes and pigments. (Collier & Tortora, 2001)

A dye is an organic compound composed of a chromophore (the coloured portion of the dye molecule) and an auxochrome (which slightly alters the colour). The auxochrome makes the dye soluble and is a site for bonding the fibre. Dyes are molecules that can be dissolved in water or some other carrier so that they will penetrate the fibre. Dyeing processes may be used either to colour fibres and yarns before they are made into cloth or to colour the fabric itself. Dyeing also provides a means to decorate fabrics. Dyes may be applied as a solution or paste to patterns created on fabric. To be usable in colouring fabrics, a dye must be highly coloured; must yield goods that are "colourfast" or resistant to colour change or loss during use and care; and, must be soluble or capable of being made soluble in water or other medium in which they are applied, or they must themselves be molecularly dispersible into the fibres of the fabric. (Collier & Tortora, (2001))

Also referred to as dyestuffs, dyes are the most common way to add colour to fibres, yarns and fabrics. When a textile fabric, fibre or yarn is placed in the dye bath (dye solution), the item absorbs the molecules of the dye and assumes the colour of the dye. Any excess dye that remains on the outside of the fibre can bleed or become sensitive to surface abrasion. Because dyed textiles vary in their ability to hold colour (The World Book Encyclopedia, 2001), chemical additives or mordants such as salt and acid are sometimes used to regulate absorption of the dye into the fibre. This suggests using the proper dye for particular fibres, which also demands knowledge of the affinity of the fibre or substrate for particular dyes, whether it is mineral, vegetable, animal or synthetic. The implication is that all textiles could be made colour fast to an extent so that the fabric can hold its colour under normal use, laundering or exposure to sunlight. (Collier & Tortora, (2001)) Below are structures of some dye:

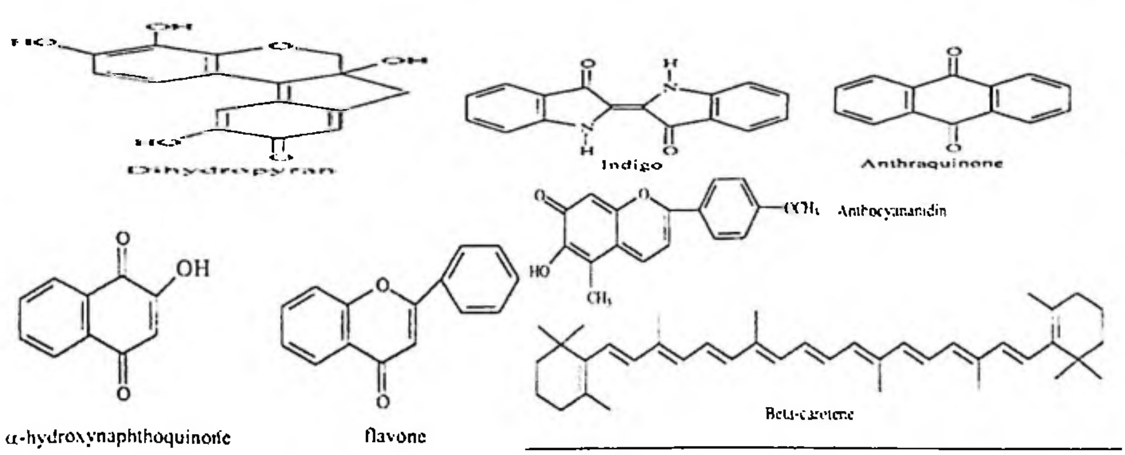

Figure 2.3 Structures of some dyes

2.3.1 Natural dyess

Natural dyes of plant, mineral and animal sources are fascinating, beautiful and sometimes they challenge the wits of researchers and educators. Most of them produce very colourful effects that are so amazing to behold. Natural colours are beautiful to behold Joseph (1977) ' Colouring matter extracted from the roots, stems, leaves or barriers, and flowers of various plants have various exceptions and are also not substantive (have little or no colouring power by themselves) except when used in conjunction with mordants. Joseph (1977) also adds that the beautiful colours that are created from natural dyes would initially appear vivid, but soon fade. Lack of colour fastness resulted in the discovery of mordants - substances which aid in the absorption of dyes.

Dyer (1976) stated that the effectiveness of natural dyes differ with each plant, with distinct differences in the colour obtained at different times of the day. Some may require mordant to improve their fastness but others may be used as direct dyes on fibre. Natural dyes also have various exceptions: they are not substantive, with little or no colouring power in themselves except when used in conjunction with mordants although most of them produce very colourful effects so amazing to behold, that sometimes they challenge the wits of researchers and educators.

2.3.2 Plant dyes

The roots, nuts and flowers of plants that grow in our backyards are all sources of colouring pigments and dyes. The World Book Encyclopedia (2001) notes that most natural dyes come from such parts of plants as the bark, berries, flowers, leaves, and roots while Storey (1997) and Adu-Akwaboah (1994) mention the use of seeds, fruits, and young shoots as other sources of natural dyes. The outer, inner bark and heartwood of trees also produce dyes. Dyer (1976) points out the madder plant that grows in Asia and Europe as a source of bright red dye that is used on fabrics that include linen and silk.

Plant dyes play a major role in the textiles industry. Dyes from a variety of woody plants and herbs are used in dyeing cloth, straw and fishing nets etc. Irvine (1961) mentions over 100 woody plant species found in the forests and grassland across the globe that yield dyes of varying strengths and colour that are widely used for a different purposes. According to Opoku-Asare (2005), natural dyes which have sustained the centuries old indigenous cloth dyeing and printing industry are mostly from trees which yield dark brown and black dyes respectively.

Humans have always used the colour of a food to form judgments about its desirability. The act of eating (and deciding what to eat) is a multi-sensory experience, synthesizing perceptions of sight, taste, smell, and touch. Colour provides visual information about a food's quality and condition, and influences the perception of its flavour. In nature, colour is determined by a food's inherent qualities, indicating types of flavour and degrees of sweetness, ripeness, or decay. The World Book Encyclopedia (2001)

Nonetheless, humans have contrived to add or change the natural colour in foods from very early times and for a variety of reasons such as aesthetic purposes, to increase appetite and appeal, for symbolic effects, to make a less desirable food seem more desirable, and to mask defects (www.Answers.com). From ancient times, wide varieties of food colourants have been derived from plant, animal, and mineral sources. This changed in the middle of the 19th century with the discovery of synthetic dyes that soon found their way into food.

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These synthetics were, in general, less expensive as well as more stable, controllable, and intense in hue than natural colours. Since that time, the safety and acceptable use of food colourants, both natural and synthetic, remain controversial topics, eliciting debate, continual scientific study, and periodic legislative action (www.answers .com/library/food & cultures Encyclopedia). The visual appearance of a food is a major factor in determining its acceptance by the consumer, who has a number of inbuilt expectations regarding the proper colour for a particular type of food, such as accepting peas to be green and strawberry jam to be red. Conducting sensory evaluation of foods is often necessary in order not to bias the result with the consumer's expectations (Gardner, 1993).

According to Lugutuah (2004), the stalk of the sorghum plant is a colour additive that is used in the preparation of "waakye" to make it appear reddish. It is also used as an additive for ham (whole cut of meat and mechanical formed), in cosmetics such as lipstick and shampoo, in medicine, as a colouring agent of sugar coated pills and in candies (www.fao.org).

Colour, whether natural or synthetic, are added to foods for a number of reasons such as:

i. To replace natural colour where they have been destroyed by heat or by preservatives.
ii. To ensure uniformity of colour from batch to batch, where raw materials of varying colour intensity have to be used.
iii. Where the colours of the natural ingredient are too weak and it is believed that the public prefers stronger colours.
iv. Where natural colours are affected by light.

v. To give an attractive appearance to foods which otherwise look unattractive or unappetizing. It is believed that the enjoyment of food depends on "eye appeal" among many other factors.

Because of the above considerations, it is believed that the use of added colours, whether natural or synthetic has a direct bearing on the acceptance of the food.

2.3.3 Animal dyes

Red animal dyes derived from certain species of tiny scale insects known as cochineal (Dactylopus coccus costa) that fed on red cactus berries. These insects were gathered by hand and ground into pigment, requiring 70,000 carcasses to make a pound of dye. By 1600, approximately 500,000 pounds of cochineal were shipped annually to Spain (www.answers.com/library/food&cultures encyclopedia). The dyes were highly valued from ancient times and right through the Middle Ages. Aimson (1999) states that the purple robes of royalty in Ancient Rome were dyed using a substance extracted from a rare crustacean called Trumpet Shell (Purple Fish) which was found near Tyre on the Mediterranean coast. An estimated 8,500 shellfish were crushed to produce one gram of the dye, which made it so expensive that only kings could afford to use it. Major sources of animal dyes are: Cochineal (red) - from bodies of cochineal insects, Tyrian purple or crimson - from the bodies of some types of marine snails, Sepia (brown) - from secretions of several types of cuttlefish.

2.3.4 Mineral dyes: (Derived from coloured clays and earth oxides)

Chrome Green - from a compound of chromium and oxygen, Chrome Red - from a compound of chromium and lead, Chrome Yellow - from a compound of chromic acid and lead, Prussian blue - from a compound of iron and cyanide. The World Book Encyclopedia (2001)

An ochre dye made from iron ore is one of the oldest pigments that have been in use since pre-historic times.

2.3.5 Synthetic dyes (These are made by chemical synthesis)

Since loss of colour in use is a major source of consumer dissatisfaction, the use of the appropriate dye for a particular fibre is crucial to colour fastness hence reputable manufacturers should choose dyes carefully. William Perkin, an English chemist working in London, made an accidental discovery of a purple dye while trying to synthesize quinine by reacting aniline sulphate with an oxidizing agent. This purple dye, which Perkin called mauve, was found to dye silk and although it was not particularly fast, it became popular, Encarta, 2003. Encarta 2003 also indicates that this first synthetic dye influenced the development of the synthetic dye industry. Today, industry uses synthetic dyes almost entirely because they hold their colour better and cost less to produce than natural dyes. Pre-packaged dyes are also readily obtainable in almost every colour, and are available to anyone who can purchase the end product. Perkin's discovery showed chemists that dyes and pigments could be produced synthetically in a laboratory and it was no longer necessary to search out natural products for use as colourants. Besides industrial use, professional and home dyeing of apparel and household decorations is practiced widely based on the different types of dyes and their ability to resist fading.

2.4 Mordants

Natural dyes initially appear vivid but they soon fade; very few of them prove to be colourfast. It is this fastness problem that led to the discovery of mordants (Joseph, 1977) which are natural acids and oxides that react both with the dyestuff and the fibres to form an insoluble compound that "fixes" the colour firmly in the fibres and prevents the dye from dissolving easily. Some natural dyes require mordants to make them colourfast.

According to Aimson (1999), mordants play an essential part of the dyeing process. Mordanting is very necessary, except for plants which contain a lot of tannin and do not necessarily require mordants. Mordants "bite" into the fibre, and make the dye stick to the fibres and also have the ability of changing the colours of dyes. The most commonly used mordants are alum (Potassium Aluminium Sulphate), Chrome (Dichromate of Potash), and sulphate (Ferrous Sulphate). Others include Iron sulphate, Tin crystals and stannous chloride, Tannic acid, Aluminum, chromium, Copper and iron. Chrome and Tin are poisonous, so Alum is recommended for first attempts at mordanting. All yarns and fabrics must be well washed and rinsed before a mordant is applied

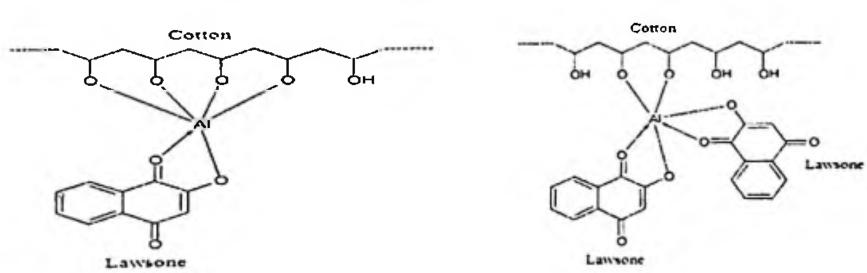

Figure 2.4 Structures showing mordant reaction with cotton and dye

2.5 Medicinal and Nutritional Values of the Plants Extract

Henna (Lawsonia inermis) leaves are mashed and applied to the nails to prevent inflammation and promote healthy growth, as well as to the eyelids to prevent eye strain. Lime juice is added to the prepared fresh leaves and used for inflamed bruises and swellings in man and animals for skin diseases and leprosy, or painted on the body for fever, especially for children. Henna health benefit include reduce inflammation, detoxify, protect the skin, use as antibacterial, and antifungal (The World Book Encyclopedia, 2001)

Nim tree (*Azadirachta indica*) leaves are relatively rich in potash and phosphate and are used in South India as green manure and cattle feed. The Leaves are placed in books in India sometimes to keep away book mites. The leaves are used in Ghana to repel locusts and have been successful to protect cocoa beans from Ephestia cantella and also used for its anti-malarial properties (Irvine, 1961). Neem is also used in treating malaria, stomach upset, body pain, skin diseases, fever, loss of appetite etc. Neem is rich in ammino acid.

Rosselle is used as antioxidant, lower blood pressure, prevent cancer, boost liver health and also fight against bacteria among others. Roselle is rich in vitamins. Turmeric prevents heart diseases and cancer, also as anti-inflammatory and antioxidant, use for depression and arthritis, it contains high calories, carbohydrate low protein and fat while Eucalyptus is use to treat asthma, wounds and burns, ulcer, bronchitis, blocked nose, head lice, toe nail fungus and fever, it contains protein, starch and lipid. (The World Book Encyclopedia, 2001)

2.6 Some Empirical Review

Zhou *el al.* (2006) described the preparation process of dye which comprised of soaking 100 parts of madder or sappan wood dust with water for 3-5 hours, adding 5-7 parts of 3% wood alcohol, filtering, removing the supernatant from the filtrate, heating to 18-22°C, sealing, fermenting for 65-79 hours, concentrating into 50-75% bright red pigment liquid for dyeing fibers. The fibre dyeing process comprised of mixing the pigment liquid 1, water 25-30 and 5% tannic acid 0.03-0.05 parts, dipping 5 parts of animal fiber or 6 parts of natural silk in the solution, boiling for 20-30 minutes, holding at 70-90°C for 60-75 minutes and drying. The bright red pigment liquid can be used together with other plant pigments.

Ansari and Thakur (2000) extracted the natural dye from pomegranate and optimised the conditions of extraction. Optimization of conditions for extraction of Natural Yellow dye from pomegranate rind has been carried out by studying the effect of pH of extraction media, time and temperature of soaking/extraction and mass to liquor ratio on quality and yield of the dye. The extracted dye has been characterized by its physico-chemical properties, viz. solubility, micro chemical analysis, thin layer chromatography and UV/Visible spectrophotometry. Dyeing experiments and analysis of red listed chemicals have also been carried out to see the efficiency and eco friendliness of the dye and to explore the possibility of its commercial use as a substitute for synthetic dyes based on forbidden aryl amines.

Bhattacharya and Shah (2000) dyed wool fabric with Catechu by two different process sequences using various metal sulphates as mordant. The dyeing behaviour has been assessed by measuring different fastness properties. The effect of different metal ions has been studied with respect to their influence on colour and fastness properties. The mechanism of mordant interaction with the fibre has been briefly considered.

Kharbade and Agrawal (1988) identified the natural dyes in historic textiles from the mid nineteenth century using thin layer chromatography (TLC) and microchemical tests. Yellow, brown and red dyes were analyzed by TLC and blue by microchemical tests. Seventy samples which were from museum textiles were compared with reference materials prepared in the Laboratory as well as with chemically pure major dye components of natural dyes. One yellow, two brown, one blue and two red natural dyes were identified in different primary and secondary coloured samples. It is also seen from the results that mixtures of two dyes have been used to obtain desired shades.

Frigerio (1992) compared characteristics of natural dyes with synthetic dyes to minimize environmental pollution. Logwoad, tropical legume dyes, yellow woad of Cuba, dyes extracted from insects, indigo, mollusks extraction and extraction from Sandalwood, saffron, curcuma, nuts, henna and lichens are described.

Tsatsaroni and Eleftheriadis (1994) dyed cotton and wool fabrics with the aqueous extract of saffron containing α-crocin as the main colourant species. The dyeing was carried out with and without metal salts as mordants. The wash and light fastness of the dyed fabrics were studied. The colour of the fabrics was investigated in terms of fastness values.

Teli *et al.* (1994) successfully applied the natural dye extracted from turmeric on the cotton material. They described that if fabric is treated with tannic acid and/or metal salts and then dyed, the dyeing show improvement in depth and performance properties such as fastness to light, washing, rubbing (dry as well as wet) etc. They used $CuSO_4$ and $FeSO_4$ and got variation in tones, improvement in light fastness and properties otherwise inferior. The influence of concentrations of tannic acid and metal salts on cotton dyeing was also studied.

Gallotti (1995) discussed the feasibility of using plant-based dyes for textile application with reference to the use of set-aside land for non-food crops. Then the processes used to extract dyestuffs from the plant materials are described: traditional methods; ultra-filtration and inverse osmosis; extraction with fluids in a supercritical state. Finally, the analysis of natural dyes is discussed with reference to analysing the dye and its precursor, checking the purity of the extract and identifying the dyestuffs on textile materials.

Deo and Desai (1999) dyed cotton and jute fabrics with an aqueous extract of tea, containing tannins as the main colourant species. The dyeing was carried out with and without metal salts as mordants, using three different dyeing methods: pre-mordanting, meta-mordanting and post mordanting. The resulting wash and light fastnesses of the dyed fabrics were good to excellent.

Bansal and Sood (2001) developed a dye from eupatorium leaves, a common weed in Himachal Pradesh. The preparations of dye material and yarn samples were specified. Four mordants were used (listed). Optimising dyeing variables covered dye extraction, concentration of harda, pH for dyeing, dyeing time, concentration of mordants and colour fastness tests. Results, revealed the best dye extraction medium was alkaline, optimum cotton yarn dyeing time in eupatorium material was 45 minutes and optimum harda concentration 30%. Mordant data is listed and the four best colours obtained were greenish raw umber, light greenish raw umber, light umber sienna and olive green. Colour fastness tests are reported with light umber sienna giving the best outcome.

Gulrajani (2001) evaluated the cotton dyeing by using various natural dyes alone and in combination to yield six basic shades: blue, yellow, red, black, green and fawn. These dyed fibres were then blended in various proportions along with undyed cotton fibres and spun on a rotor-spinning machine to produce 204 coloured yarns. The fastness properties of the six basic shades were determined. The yarns having 50% dyed fibre and 50% undyed cotton fibre was also determined. The values were plotted to obtain the colour gamut of natural dyes on cotton yarns.

Gulrajani (2001) presented status of natural dyes with reference to the stake holders of natural dyes. He estimated the dye requirements, availability of natural dyes, technology for production and some important natural dyes and mordants are critically discussed. Application techniques and fastness properties of natural dyes are also briefly discussed. It was suggested that natural dyes are not substitutes for synthetic dyes. Some of the limitation of natural dyes such as use of banned metal salts as mordants, poor fastness properties and use of agricultural land for growing natural plants could overcome through research and development.

Gulrajani (2001) demonstrated the possibilities of nylon dyeing by using vegetable dyes like Annatto, Ratanjot and Berberine. Furthermore evaluation of sample fastness and colour value was undertaken. Dye origins were explained and the fabric sample specified. Dyeing solutions were specified and testing methods and equipment described. The results noted the Anlab Colour Space plots for each dye prepared. Wash fastness was good for Rajantot, very good for Annatto and poor for Berberine. Light fastness values were included. Nylon could be dyed with these vegetable dyes at PH-4 (Rajanjot), pH- 6 (Annatto) and pH-9 (Berberine). Good to moderate light and wash fastness resulted.

Choo and Lee (2002) extracted and analyzed natural dyes by using traditional Korean methods of dyeing cloths. Nine plants were used, either singly or in combination, to produce a wide range of colours. Some of the fabrics were then analysed for chemical identification of their dye components and mordants. The results of microchemical tests, visible spectroscopy, thin layer chromatography and high performance liquid chromatography are compiled as reference data for later comparison with the test results of antique samples that are increasingly becoming available from a number of excavations. This body of reference data will permit a more scientific understanding of traditional dyeing crafts, essential for authentic restoration and proper conservation.

Dweck (2002) examined some of the existing methods for colouring the hair and skin using natural material (such as henna) and proposes a parallel technology that exists in the dyeing of wool and fabrics to extend the colour range. Many of the listed plants and their derivatives are not found in Annex IV of the Cosmetic Directive and may not be used as colours; however, they do have other properties which may justify their inclusion into a product, for example, as astringent or anti inflammatory agents. The paper concludes with some reported anti-greying and hair styling preparations cited in the literature.

Bechtold *et al.* (2003) investigated Plant materials which are available from farming regions in the moderate Austrian climate to serve as sources for natural dyes in textile dyeing operations. The extraction of the dye components from the plant materials was performed with boiling water without addition of chemicals or solvents. Based upon a rigorous selection of possible plant sources, a selection of natural dyestuffs applicable in a one-bath dyeing step was established. A broad variation in shade and colour depth can be achieved by applying mixtures of natural dyestuffs in various combinations of iron-and alum-mordants. More than 60% of tested dyeings achieved acceptable fastness properties. On the basis of the developed natural dyestuff-based dyeing procedures, a comparison was made between the effluents from processes based upon them and those based upon the current 'state-of-the-art' techniques utilizing synthetic dyes. The comparison revealed that lowering of the chemical load released with waste water can be expected by shifting to the plant-based dyes.

Cevallos-Casals and Cisneros-Zevallos (2003) evaluated anthocyanin-based aqueous Andean red sweet potato and purple corn extracts under different pH, temperature, and light conditions, and compared to commercial colorants (purple carrot, red grape, red 40, and red 3). Red sweet potato and purple carrot colorants, rich in acylated anthocyanins, showed higher stability than purple corn and red grape colorants, rich in non-acylated anthocyanins. After storage at 20°C for 138 days, the order of stability in the pH range 0.9-4 was: red sweet potato≥purple carrot>purple corn>red grape. After this storage time, red sweet potato pH 4 extracts maintained a red-violet hue. Half-lives for pH 3 extracts at 98°C were 4.6, 4.6, 2.4, and 2.0 h for red sweet potato, purple carrot, red grape, and purple corn, respectively. The hues for purple corn pH 3 extracts were similar to those of red 40. Parameters measured included degradation index, polymeric colour, colour retention and spectral data.

Mathur and Gupta (2003) obtained natural dyes from concentrating the aqueous solution extract of banana flower petaloide under reduced pressure & evaporating it to dryness. Bharat merino sheep wool yarn dyed with turmeric (Curcuma longa) was subjected to mordanting separating with natural mordant and chromium under the identical condition. Out of the different concentration of the mordants used 3.5% natural mordant and 1.55 % chromium on the weight of yarn show similar colour fastness, reflectance, colour shape and values. The chemistry of wool dyeing and the physio chemical properties of dyed wool yarns are also discussed.

Paul *et al.* (2003) described dyeing procedure with walnut bark, a good source of brown colour and relatively eco friendly with ease in application for rural population especially. Dyeing variables in using walnut dye to dye wool were tested, and the colourfastness of selected dye on woollen yarns was also investigated. The advantages of natural dyes are detailed, particularly in comparison to synthetic dyes which can cause harmful effects. The selection of fibre, bleaching of woollen yarns, selection of dye and selection of mordants were described. Experiments were conducted to determine the optimisation of dyeing variables - medium of dye extraction, and optimisation of dye extraction time, dye concentration and dyeing time. Preparation of final samples and colourfastness testing were covered. Walnut bark was found to be a good source of brown colour, with a variety of fast shades using different mordants. Further studies are recommended to promote rural entrepreneurship and the rural economy.

Paul *et al.* (2003) used the roots of Berberis vulgaris, a common shrub in India to prepare a dye in order to optimise various variables for its use as dye. Four synthetic mordants were used for the study: alum, chrome, copper sulphate and ferrous sulphate. The medium of dye extraction, extraction time, and dye concentration were investigated, the woollen yarns were dyed according to the results, and colourfastness was then tested. Four tables present data on the colourfastness results. Overall, Berberis was found to be a good source for producing a number of fast shades ranging from yellow to black on woollen yarns by using different mordants.

Phukan and Phukan (2006) used the bark of the Tapar tree for dyeing mulberry silk yarn. Both alkaline and acidic methods of dye extraction were used, together with pre-mordanting, simultaneous mordanting and post-mordanting with alum, chrome, copper sulphate and ferrous sulphate. Fastness was also assessed. The results show that alum and the pre-mordanting method gave the best result, with good colourfastness. The alkaline medium method of dye extraction gave the best dye absorption.

Zhou *et al.* (2003) listed typical examples of bio-dyestuffs including insect and tree secretions, and vegetable dyes. The plants providing red, yellow, blue, green and black colours are also listed and the method of extraction detailed. The problems faced by bio-dyeing are noted. Bio-materials can be used to replace harmful, energy or material-expensive chemical treatments for pretreatment and finishing. These include wool decrement by enzyme, shrink-proofing of wool by enzyme, wool and fabric washing by enzyme and other bio-reagents. Chinese textile exports are meeting more environmentally based barriers in international trade and biomaterials instead of harmful reagents would help to resolve these problems.

Agarwal and Gupta (2003) discussed the conditions for dyeing of wool with a vegetable dye from the roots of the herb Madder (Rubia cordifolia). The optimized conditions included the concentration of the dye, the extraction time, the dyeing time, the concentration of the mordants, and the method of mordanting for wool fibers. The dyed samples were subjected to tests for fastness to light and washing. From optical density data, the optimum concentration of the dye was found to be 5 grams per 100 ml of water, while the optimum extraction and dyeing time were found to be 120 minutes and 90 minutes, respectively. The simultaneous method of mordanting was observed to give the best results in terms of lustre, depth of shade, evenness of the dye, and the overall appearance.

Bhuyan *et al.* (2004) described natural dyes as important alternatives to synthetic dyes. A study was initiated in the year 2000 at the RRL (CSIR), Jorhat to extract dyes from parts of five different plant species indigenous to northeastern India. The colour components responsible for dyeing were isolated and their chemical constituents were established based on chemical and spectroscopic investigations. The principal colour components from the species Morinda angustifolia Roxb., Rubia cordifolia Linn. and Tectona grandis Linn, were found to contain mainly anthraquinone moieties in their molecules. Those from the species Mimusops elengi Linn and Terminalia arjuna (Roxb.) Wight & Arn. contained flavonoid moieties in their molecules. The absorption of dye (%) on fibres increased with increasing concentrations of dye in the dye-bath. Maximum absorption of dyes on fibres was obtained at 3% concentration of dyes obtained from R. cordfolia (35.350%), M. angustifolia (31.580%) and T. grandis (25.888%) and at 4% concentration of the dyes from M. elengi (31.917%) and T. arjuna (12.246%). The dyes obtained from the native plants may be alternative sources to synthetic dyes for the dyeing of natural silk and cotton.

Ferreira *et al.* (2004) described the sources and structures of dyes used to colour Western historical textiles as described in this tutorial review. Most blue and purple colours were derived from indigo, obtained either from woad or from the indigo plant though some other sources (e.g. shellfish and lichens) were used. Reds were often anthraquinone derivatives obtained from plants or insects. Yellows were almost always flavonoid derivatives obtained from a variety of plant species. Most other colours were produced by over-dyeing e.g. greens were obtained by over-dyeing a blue with a yellow dye. Direct analysis of dyes isolated from artefacts allows comparison with the historical record.

Garima *et al.* (2004) worked on wool dyeing using reinwardtia flowers and poplar leaves in the ratio of 50:50 each as natural dye. Different variables viz. wave length, dye material combination, dye extraction time, dye material concentration, dyeing time, pH and mordants were standardized. 7% dye material, extracted and dyed for 45 minutes each using 1 and 4% of copper sulphate, chrome and ferrous sulphate as mordants gave excellent colours ranging from yellow ochre to military green. The fastness grades in terms of washing as colour change were 3-4/5 and colour staining from 3-5, light fastness from 3/4 to 4/5, rubbing fastness 4-5 and perspiration from 3/4 to 4/5. Hence the source explored was found suitable for dyeing of wool.

Paul *et al.* (2003) studied the applications of natural dyes in textile dyeing that are being worldwide. The dye exhaustion, fixation and levelness of dyeing depend on several factors such as the properties of fibres, the molecular structure of dyes, and the medium of the dye bath. The solubilisation of insoluble natural dyes in colloidal systems like micro-emulsions and its influence on dye exhaustion and fixation are becoming important in this connection. In the present investigation, the phase behaviour of water/non-ionic surfactant/polar oil system was studied and the micro-emulsion regions were identified. A water insoluble natural dye, C.I. Natural Orange 2, was then solubilised in selected micro-emulsions and its ability to dye wool was studied. The mechanism involved in the dye migration from the oil-swollen micelles to the fibre surface was also investing.

Phukan and Phukan (2004) standardized the condition of dyeing mulberry silk yarn with the bark of Arjun tree, Terminalia Arjuna. Mordants such as alum, chrome, copper sulfate, and ferrous sulphate were used for the study for the fixation of the dye molecule with the fiber. To remove the sericin, degumming was done before dying, with washing soda, alkaline and acidic methods were employed for dye extraction. Yarns were mordanted in the first stage and dyed in the second stage in the pre mordanting method. In simultaneous mordanting, mordants and dyes were applied simultaneously in the same bath. In the post-mordanting method, however, the yarns were first dyed and then mordanted. The alum mordant and pre-mordanting method showed the best results in both alkaline and acidic medium for the Arjun tree dye. Yarns dyed with Arjun dyes showed colour fastness to washing, rubbing, light, and perspiration.

Rose *et al.* (2004) performed efficient dyeing of cotton yarns with a plant dye, Ornamental Mustard (Brassica juncea) with certain optimum variables. Experiments were therefore conducted to standardize the medium of dye extraction, wave length, extraction time, dye material concentration, dyeing temperature, dyeing time, and dyeing pH. The ornamental mustard leaves were extracted in aqueous, alkaline, and alcoholic mediums, and the best color was obtained in the alkaline medium. The results showed that the optical density increased with increased extraction time up to 30 minutes, and further decreased with increase in extraction time. The maximum dye absorption was observed at seven per cent dye material concentration, and increased with increase in dyeing temperature. The dye absorption also increased with increasing pH, and thus the optimum pH selected for dyeing was 10. Thus, dye extraction in an alkaline medium with optimum wave length of 360nm, extraction time 30 minutes, dye material concentration 7%, dyeing temperature 100°C, dyeing time 45 minutes, and dyeing pH 10, gave excellent results for dyeing cotton yarns.

El-nagar *et al.* (2006) described that synthetic dyes are more available than natural dyes, because of lower prices and wider ranges of bright shades with considerably improved colour fastness properties. In current years, concern for the environment has created an increasing demand for natural dyes, which are friendlier to the environment than are synthetic dyes. The aim of this work is to study the effect of dyeing cotton fabrics with both a natural dye (henna) and a synthetic dye (Remazol blue) on some mechanical properties and those of stability to light exposure. The undyed and dyed cotton fabrics were tested for their mechanical behaviours expressed as tenacity (N), elongation %, and work breaking (N•m). They were also tested for shrinkage and crease recovery angle. The stability to light before and after 100 h exposure was examined by investigating the microstructure [using x-ray diffraction (XRD)] and macrostructure [using a scanning electron microscope (SEM)] and the reflection spectra. The results proved that the cotton samples dyed with Henna dye have higher mechanical properties than those dyed with "Remazol" reactive dye. Moreover, the light fading behaviors of both synthetic and natural dyes were studied in terms of the reflection spectra (400-800 nm), microstructure, and macrostructure of the sample's fibers.

Clementi *et al.* (2005) spectrally characterized a naturally occurring dye, orcein, which was widely used in antiquity for textile dyeing, in both solution and powder. Laboratory samples of wool and silk orcein-dyed threads were analysed before and after ageing. An original fragment of Renaissance tapestry was also analysed. The textile (wool) and the colourant (orcein) were recognised by comparison with the data from the laboratory samples.

Cristea and Vilarem (2005) evaluated the light fastness of selected natural dyes (madder, weld and woad) and the effect of some commonly used antioxidants and UV absorbers on the light fastness of these dyes. The photofading rate curves of madder and weld fixed on cotton correspond to type II fading rate curves described by Giles. These results are in concordance with those of Cox-Crews. The woad presents a type III fading rate curve, similar to the indigo fading rate curve presented by Cox Crews. A poor light fastness of the three natural dyes in comparison with synthetic ones is established beyond question. Nevertheless, the use of some additives can improve this default of natural dyes. In all the cases, the use of UV absorbers or antioxidants improved the light fastness of dyed fabrics. The most effectives were the vitamin C and the gallic acid.

Grover *et al.* (2005) stated that eco-friendly dyes have gained much importance in dyeing of textiles, especially silk fabrics. The possibilities to extract dyes from plants and to optimize various dyeing variables of these dyes for dyeing of silk have been studied. The natural dyes which were selected for the study included Jatropha, Lantana, Hamelia, Euphorbia, Kilmora, and Walnut. Silk was initially degummed prior to dyeing, to make the fabric free from sericin, which obstructs the penetration of dyestuffs into the fiber. A series of experiments were conducted to determine the dye extraction medium, optimum concentration of dye, extraction time, dyeing time, mordant concentration, and methods of mordanting. The acidic media exhibited maximum percent absorption for Jatropha, Lantana, Hamelia, and Euphorbia dye, while Kilmora and Walnut dye showed good results in alkaline medium. The results obtained from different experiments lead to the optimization of a standard recipe for dyeing of silk with each dye source.

Singh *et al.* (2005) explored the study to test of some natural dyes as inherent antimicrobial activity with a view to develop protective clothing from these. Four natural dyes Acacia catechu, Kerria lacca, Quercus infectoria, Rubia cordifolia and

Rumex maritimus were tested against common pathogens Escherichia coli, Bacillus subtilis, Klebsiella pneumoniae, Proteus vulgaris and Pseudomonas aeruginosa. Quercus infectoria dye was most effective and showed maximum zone of inhibition thereby indicating best antimicrobial activity against all the microbes tested. Minimum inhibitory concentration was found to be varying from 5 to 40 μg. The textile material impregnated with these natural dyes, however, showed less antimicrobial activity, as uptake of these dyes in textile material is below MIC.

Vankar *et al.* (2005) studied the dyeing of cotton fabric using Eclipta as natural dye in both conventional and sonicator methods. The effects of dyeing show higher colour strength values obtained by the latter. Dyeing kinetics of cotton fabrics were compared for both the methods. The time/dye uptake reveals the enhanced dye uptake showing sonicator efficiency. The results of fastness properties of the dyed fabrics were fair to good. CIELAB values have also been evaluated.

Bechtold *et al.* (2006) reported the introduction of natural dyes into modern textile dye houses requires the classification of products of standardised quality with regard to colour depth and shade of the dyeings. Canadian golden red was chosen as a representative example to test the methods that are available to assess the quality of different crops of plant material which had been collected over a period of five years. Aqueous solutions containing the extracted flavonoid dyes were characterised by means of direct photometry, measurement of absorbance after addition of $FeCl_2$, analysis of total phenolics (TPH) in the extract and dyeings on wool yarn. TPH calculated as gallic acid varied from 62 g/kg to 97 g/kg of plant material; only one sample exceeded this range with a value for TPH of 142 g/kg. Correlation among TPH, photometry in the presence of $FeCl_2$ and lightness of the dyeings can be used to characterize samples.

However, correlation between the photometric results and colour depth of dyeings was not sufficient to permit characterisation of the plant material with regard to the final dyeing. At present, a combination of laboratory dyeings and CIELab coordinates was found to be suitable to establish an experimental basis for standardisation of plant material.

Bechtold *et al.* (2006) described that food and beverage industry releases considerable amounts of wastes which contain natural dyes. Such wastes could serve as a source for the extraction of natural dyes for textile-dyeing operations. The extraction of brilliant yellow and red colours from fruits and vegetables is of particular interest. Wastes, e.g. pressed berries, pressed grapes, distillation residues from strong liquor production, and wastes and peels from vegetable processing, have been extracted with boiling water and test dyeing on wool yarn were performed. Colour strength, shade and fastness properties of the dyeing have been tested. The extracts were applied as direct dyes and in the presence of iron (II) or alum mordants. The results prove the potential of such wastes as a source for natural dyestuff extraction. To obtain textile dyeings with acceptable fastness properties, however, rigorous selection of dyes and development of suited processes is required. A considerable number of red natural dyes need further research to optimise the low level of fastness to light.

Colombini *et al.* (2007) described an analytical procedure based on GC-MS to identify in textiles the most common flavonoid yellow dyes used in Europe since ancient times, extracted from weld, young fustic, dyer's broom, sawwort and the berries of some species of Rhamnus. Later on, old fustic and quercitron bark were introduced as sources of yellow colours. The method is based on the solvent extraction of flavonoids from raw plant materials (weld, dyer's broom and old fustic), aged and not aged alum-mordanted wool dyed specimens; subsequently, flavonoids are derivatised with N,O bis(trimethylsilyl) trifluoroacetamide and analysed by GC-MS. The method easily allows the identification of a dyestuff by the detection of the molecular markers apigenin, luteolin, genistein, morin, maclurin, together with 4-hydroxybenzoic acid, 3,4-dihydroxybenzoic acid, 2,4 dihydroxybenzoic acid, 2,4,6- trihydroxybenzoic acid, which survive in aged textiles. Two photo oxidative degradation pathways for colour fading, one involving the mordant metallic ion and the other the light as a catalyst, are suggested.

Das *et al.* (2006) studied the application of dye obtained from Punica granatum fruit rind on wool and silk fabric in the presence and absence of environment-friendly mordanting agents. The dyeing of silk and wool with pomegranate solution is found to be effectively accomplished at pH 4.0. Pre- and post-mordanting employing ferrous sulphate and aluminium sulphate improve the colour uptake, light fastness and colour retention repeated washing. The use of such mordants, however, does not improve wash fastness property of dyed substrates.

Kamel *et al.* (2007) studied the dyeing of cationised cotton fabrics with lac natural dye by using both conventional and ultrasonic techniques. The effects of dye bath pH, salt concentration, ultrasonic power, dyeing time and temperature were studied and the resulting shades obtained by dyeing with ultrasonic and conventional techniques were compared. Colour strength values obtained were found to be higher with ultrasonic than with conventional heating. The results of fastness properties of the dyed fabrics were fair to good. Dyeing kinetics of cationised cotton fibre with lac dye using conventional and ultrasonic conditions were compared. The values of dyeing rate constant, half-time of dyeing and standard affinity and ultrasonic efficiency have been calculated and discussed.

Mahale *et al.* (2006) selected mahogany leaves for dye extraction and optimization of dyeing conditions by using different textile materials including cotton, silk and wool. A neutral non-bitter principle, Swietenine and a bitter hygroscopic component, Sweitenoide were isolated from the seeds. Melianone was found to be present in the dried leaves of the tree. In addition to optimizing the dyeing conditions, an attempt was made to assess the colorfastness properties of the textile materials. It was observed that cotton dyed with ferrous sulphate, post-mordanted, has the highest dye absorption. Silk and wool showed the highest dye absorption for Stannous chloride under the post-mordanting method.

Mashaly (2006) studied the dyeing of nylon fabrics using lac as a natural dye in both conventional and ultrasonic techniques. The extractability of lac dye from natural origin using power ultrasonic was also evaluated in comparison with conventional heating. The results of dye extraction indicate that power ultrasonic is rather effective than conventional heating at low temperature and short time. The effect of dye bath pH, salt concentration, ultrasonic power, dyeing time and temperature were studied and the resulting shades obtained by dyeing with ultrasonic and conventional techniques were compared. Colour strength values obtained were found to be higher with ultrasonic than with conventional heating. The results of fastness properties of the dyed fabrics were good to very good. Dyeing kinetics of nylon fibre with lac dye using conventional and ultrasonic conditions was compared. The time/dye-uptake isotherms are revealing the enhanced dye-uptake in the second phase of dyeing (diffusion phase). The values of dyeing rate constant, half-time of dyeing and standard affinity and ultrasonic efficiency have been calculated and discussed.

Mukherjee (2006) standardized the strength of dyes and dose of mordants, which are interrelated to each other, are extremely necessary for shade reproducibility as well as for the prevention of serious water pollution. Natural dyes are mostly obtained from vegetable sources which yield dyes according to their maturity, climate and soil. Hence the fact cannot be denied that a natural dye manufacturer will feel the difficulties in controlling the dye strength as well as tone apart from the brightness which is inherent to natural dyes in most of the colourants. But strength approximation after dye manufacture for each batch can be done in terms of a standard metallic mordant from which the doses of other mordanting salts can be correlated. The dyers in small and big sectors can be able to follow the routine of application in an easy method which will help minimize the water pollution to some permissible level.

Murthy (2006) featured an audiovisual representation of antique natural dyes obtained from nature, and modern Laser dyes made by academia at an event held on 9 December, 2005. The topics discussed highlighted the developments and applications of natural dyes such as Ajrakh, and synthesis and structure of laser dyes. Mrs. Hundekar, a student from textiles division of UICT highlighted techniques of Ajrakh printing, which mainly focused on indigo blue dyeing from nature. Dr. Sekar, a co-investigator of project entitled Development of Dye Dope Polymeric Sol Gel Laser Materials, gave a presentation on dye lasers, focusing on the functioning, evolution, properties, and dyes used for these lasers. It is suggested that both laser dyes and synthetic dyes should not be compared with natural dyes as both target different designs for unique niche marketing.

Nagia and Mohamedy (2006) described two anthraquinone compounds which were produced by liquid cultures of Fusarium oxysporum (isolate no. 4), isolated from the roots of citrus trees affected with root rot disease. These anthraquinone compounds are 2-acetyl-3, 8-dihydroxy-6-methoxy anthraquinone or 3-acetyl-2,8-dihydroxy-6-methoxy anthraquinone. Dyeing of wool fabrics with these new anthraquinone compounds as natural dyes has been studied. The values of dyeing rate constant, half-time of dyeing and standard affinity have been calculated and discussed. The effect of dye bath pH, salt concentration, dyeing time and temperature were studied. Colour strength values and the dye uptake were high. The results of fastness properties of the dyed fabric were good.

Rawat *et al.* (2006) studied the application of poinsettia leaf dye, an environmental friendly natural dye, on silk fabric. The fastness properties were found satisfactory. A silk fabric, which was degummed using a solution of genteel and water, was dyed with poinsettia leaves and the dyed fabric was subjected to colour fastness testing. During the colourfastness test, the samples were washed in a Laundrometer for 45 minutes at the constant temperature for 50° C. The colourfastness of the dyed samples to acidic solution was found much better than the alkaline solution. The dye may be useful in imparting number of fast shades on silk using common mordants such as FeSO4 and $CuSO_4$ with good fastness properties except alkaline perspiration.

Sarkar *et al.* (2006) applied the portion of extracted natural dye on hydrophilic substrate like bast fiber. The hydrophilic textile substrate like 100% flax were chosen and prepared for the application of dye to obtain true shades of natural dye. Four chosen flowers were Marigold, Butterfly pea, China rose and Balsam. Use of acid dye bath mechanisms among gardenia, mordant and cotton fibers were analyzed and the processing factors affecting the mordant dyeing were discussed. As a result, gardenia showed favourable dyeing performance on cotton. The soaping color fastness and crocking fastness were improved by 0.5-1 grade after fixation process with the selected dye-fixing agent and optimized process conditions. The dyeing process and fixation process were optimized.

Bechtold *et al.* (2007) used the aqueous extract of ash-tree bark (Fraxinus excelsior L.) as a model to study the shade reproducibility of dyeing on wool. A meta-mordanting process using $FeSO_2 \cdot 7H_2O$ mordant was chosen as a system with particular potential for industrial application. The exhaust dyeing process with immediate use of the extracts as a dye bath and direct addition of $FeSO_4 \cdot 7H_2O$ stock solution as a meta-mordant process showed good shade reproducibility and satisfying levelness of the dyed material. An increase of Fe (ii)-mordant above a dyebath concentration of 2–3 g l 1 did not result in further colour depth. Extraction of 1–2 g of bark was found sufficient to dye 1 g wool yarn to the darkest colour possible; use of higher amounts of bark did not yield substantially higher colour depth. The quality of bark and the extraction step were found to be of significant importance for the colour depth; thus, in an optimised process, conditions of extract formation have to be well controlled.

Clementi *et al.* (2007) investigated the light fastness of wool textile samples, using two complementary experimental techniques: absorption and emission UV-vis spectroscopy and chromatography (HPLC-PDA).dyed with madder and its principal components alizarin and purpurin,.

Spectroscopic techniques were used to follow the time course of ageing, whereas chromatography was applied to determine relative compositional changes that occurred after exposure of wool dyed samples to natural and artificial ageing. The results from the two techniques integrate well each other and provide complementary and useful indications about the sensitivity of the dyed textiles to ageing, showing that purpurin is the principal component responsible for the spectral and chromatic properties of madder as well as for its degradation. The fading of both the fibre and dye is reduced in the presence of alum and in the absence of oxygen. The multi-analytical approach used highlights the potential of the UV-vis spectroscopy for the investigation of dyes on textiles. The great sensitivity of the spectrofluorimetry makes this technique particularly promising for a non-destructive study of dyes on works of art.

Feng (2007) investigated the ultraviolet protective properties of the fabrics dyed by Rheum and Lithospermum erythrorhizon were investigated. Experimental results revealed that the fabrics dyed with natural dyes had good ultraviolet protective properties. They could absorb about 80% of the ultraviolet rays. It was demonstrated that the UV-protective effect was strongly dependent on the absorption characteristics of natural dyes for UVR. Natural dyes such as Rheum and L. erythrorhizon had comparable UV-absorption performance to the common UV-absorber, benzophenone.

Guinot *et al.* (2007) extracted and investigated flavonoids from marigold flowers for their dyeingpotential. Patulitrin (1) and patuletin (2) were isolated and their structures established using NMR and HPLC-MS. These compounds were identified as the main flavonoids present in the dyeing bath. Following the dyeing process, it was demonstrated that aglycone 2 bound more strongly to wool fibres than its glucoside 1. Moreover, analysis focused on 1 and 2 dynamics during plant growth revealed that these components were only found in flowers during and after flowering. The influence of growing location was also investigated and it appeared that cultivation under after exposure of wool dyed samples to natural and artificial ageing. The results from the two techniques integrate well each other and provide complementary and useful indications about the sensitivity of the dyed textiles to ageing, showing that purpurin is the principal component responsible for the spectral and chromatic properties of madder as well as for its degradation. The fading of both the fibre and dye is reduced in the presence of alum and in the absence of oxygen. The multi-analytical approach used highlights the potential of the UV-vis spectroscopy for the investigation of dyes on textiles. The great sensitivity of the spectrofluorimetry makes this technique particularly promising for a non-destructive study of dyes on works of art.

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Mediterranean conditions enhanced biosynthesis of 1 and 2. Finally, several solvents were tested for their potential to extract the flavonoids: the use of a water-ethanol mixture gave high extraction efficiency and allowed selective extraction of 1 and 2. The implications of these results are discussed in relation to the development of marigold as a potential dyeing plant.

Guinot *et al.* (2007) investigated aqueous extracts of plant by-products (carrot, onion, black carrot, sage, spinach and thyme) for dyeing capacity on fibres and for both colorant and antioxidant potential using colorimetric and chromatographic tools, and FTC assay, respectively. Regarding fibres, classical correlations between measured colours and phytochemical patterns of dyeing extracts were verified. Light fastness of onion, sage and thyme samples, evaluated following a normalised test, was very promising considering industrial restrictions; moreover, antioxidative activities of those aqueous plant extracts were very attractive when compared to the three others and to α-tocopherol used as standard. Our results were of great interest underlining new complementary valorisations for plant by products, becoming in this way new and inexpensive natural resources for various industries.

Kale *et al.* (2007) analyzed that synthetic dyes bring better performance but it harms the environment and ecology as it creates pollution. That is why the use of natural dyes has been brought back because these do not bring harm to ecology as they are biodegradable, non-toxic and eco friendly. Natural dyes are soft in color, cool to eyes and good to skin. Several methodologies as the preparation of yarn, selection of dye material and the medium of dye extraction is presented. The optimization of dye extraction time, dye material concentration and mordant concentration were also

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Koyuncu (2007) studied the dyeing of wool yarn using Rheum ribes roots as natural dye in conventional method. The effects of dyeing show higher colour strength values obtained by the latter. Dyeing with Rheum ribes roots has been shown to give good dyeing results. The results of washing fastness properties of the dyed wool yarn were fair to good.

Lee (2007) extracted natural colorant from Coffea arabica L., using water as extradant at 90°Cfor 90 min. Studies have been made on the dyeing, colour fastness, and deodorization properties of cotton, silk, and wool fabrics dyed with Coffea arabica L. extract solutions, The best mordants were found to be Fe_2O_4, $CuSO_4$, and $SnSO_4$ for improving the colour strength of cotton, silk, and wool fabrics. Mordants $MnSO_2$, $ZnSO_4$, and $NiSO_4$ for cotton (Rating 3), and all mordants except $MnSO_4$ for silk (Rating 3), mordants $CuSO_4$, $FeSO_4$, $CoSO_4$, $Al_2(SO_4)_3$, and $MnSO_4$ for wool (Rating 4) were the best mordants to improve the light fastness. It was found that $FeSO_4$ and $CuSO_4$ were the best mordants for the improvements of colour strength and light fastness for silk and wool fabrics. In addition, it was found that cotton, silk, and wool fabrics dyed with the Coffea Arabica L. extract showed good deodorization performance.

Lu *et al.* (2007) performed experiment of wool fabrics dyeing with sorghum red as natural dye by mordant dyeing method. The process conditions of premordant and post-mordant dyeing were determined in quadrature experiments. The experimental results were as follows: the consistency of the dye was the key factor on dyeing depth in pre-mordant process, the dyeing depth enhanced with consistency of the dye solution increasing; the pH value was the second factor that affected the depth, the depth improved with the increase of pH value. The cinsistency of Fe2+ played an important role in post-mordant dyeing process. The depth enhanced with the increase of consistency of Fe2+. Temperature was the less important factor in the process. The depth improved with the temperature rising. The rubbing and washing colour fastness of dyed wool fabric were all 4 or up to 4. It indicated that sorghum red dye was suitable to dye wool fabric..

Sharma *et al.* (2007) explored the herbaceous plant Eupatorium adenophorum as a very good green colour source for dyeing of silk yarn with excellent fastness properties. Leaves of the Eupatorium plants were collected and shade dried, crushed and packed. Sericin was removed so as not to interfere with luster and dye absorption. The dye material was entered into the dye liquor and boiled. Four mordants were used with three methods of mordanting. A mordant ranged from 1-5% was selected for the study. The concentration of dye material was optimized by taking seven concentration prepared by boiling. Yarn was dyed in the dye bath for varying time, the time at which the absorption was high and selected as optimum dyeing time, and then evaluated for color fastness.

Results show that 10% alum with post mordanting method has yielded yellowish green shade while with 4% chrome in pre mordanting sap green color has been obtained. Light army green and dark army green shades on silk were obtained with 4% $CuSO_4$ and 4% $FeSO_4$ using post-mordanting method. Excellent fastness to light and outstanding fastness to washing was shown by dyed silk yarn using 4% chrome. Little noticeable staining and color change was found for dry and wet crocking samples.

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Tiwari and Vankar (2007) carried out standardisation and optimisation of dye extraction of Terminalia arjuna bark. The dyeability of aqueous extract was evaluated for dyeing cotton fabric. Dyed cotton fabric shows good fastness properties and evaluated as commercially viable natural dye source.

Vankar and Shanker (2007) used Bischofia javanica Bl. (Local name Maub) belongs to family Euphorbiaceae for natural dye production for textile dyeing. In the present study innovative sonicator dyeing wit Bischofia has been shown to give good dyeing results. Pretreatment with 1-2% metal mordant and using 5% of plant extract (owf) is found to be optimum and shows very good fastness properties for cotton, wool and silk dyed fabrics.

Vankar *et al.* (2007) studied the production of anthraquinone reddish orange dyes in roots stem and leaves, which has been used for dyeing textiles since ancient times from Rubia cordifolia (Tamin, local name). Commercial sonicator dyeing with Rubia showed that pretreatment with biomordant, Eurya acuminata DC var euprista Karth. (Theaceae family) [local name, Nausankhee (Apatani tribe), Turku (Nyishi tribe) in 2%] showed very good fastness properties for dyed cotton using dry powder as 10% of the weight of the fabric is optimum. Use of biomordant replaces metal mordants making natural dyeing ecofriendly. Indigotin and indirubin was eliminated. For acidic extraction of dyes from fibres, ethanol was used. Due to its higher boiling point than methanol it evaporates slower from the extraction solution enabling a more efficient extraction of dyes.

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CHAPTER THREE

MATERIALS AND METHODS

3.1. Materials/Reagents and Equipment

Plant source, Cotton cloth, Bowls, Beakers, Conical flask, Mordants, Knife, mortar, Filter paper, Tripod stand, Mesh, Sodium hydroxide: For making the extraction medium alkaline, Sodium sulphate: For boosting dye exhaustion during dyeing, Alum and Ferrous sulphate: as mordant, Reactive dyes, Red for making comparative study with natural dyes, Water: for extraction of dye and also for dyeing of fabric, Reflux apparatus/pressure cooker: For dye extraction, Dyeing-bath: for dyeing process, ferric chloride, chloroform, concentrated sulphuric acid, concentrated hydrochloric acid for the plant extract phytochemical screening, Colour imminochromatographic strip for colour test

3.2 Processing of Sample

Roselle, Turmeric, Hena, Neem and Eucalyptus samples (10kg) of each were harvested from various farms at Fadan-Karshi, Sanga L.G.A area of Kaduna state. They were washed and allowed to dried in air, and were powdered in a mortar to smaller pieces and sieved to obtain a uniform particles size of the material. These smaller pieces were used to extract the corresponding dyes.

Plate 3:1 Processed samples

3.3 Optimization of Extraction Conditions

The extraction of the dye from Roselle, Turmeric, Hena, Neem and Eucalyptus were carried out in distilled water as well as in aqueous solutions of sodium hydroxide. The processed materials were divided into four parts for each plant and extracted separately one after the other, in four different temperatures, time, and three NaOH concentration/pH and the results were compared to conclude the best extraction method.

The processed material were measured, 10g of each sample were added separately to 500cm^3 of distilled water at a neutral pH as while as in aqueous solutions of sodium hydroxide with varying pH: pH=12 (0.01mol/dm^{-3} NaOH), pH=13 (0.1 mol/dm^{-3} NaOH), pH=13.5 (0.35mol/dm^{-3} NaOH). The extraction process was carried out for four experiments separately for each plant sample at a temperature range: for neutral sample 90-100°C for 30min, aqueous solutions sample A 60-65°C for 1hr, for sample B 70-

75°C for 45min and C 90-100°C for 30min. The dyes extracted from the plant samples were used for colouring food and dying of the fabric.

3.4 Phytochemical Test

i. Test for phenols: The extract ($2cm^{3)}$ was taken into a test tube and a few drops of ethanol and few drops of dilute ferric chloride were added. It was shaken well till the appearance of greenish- yellow colour which indicates the presence of phenol.

ii. Test for tannins: The extract ($2cm^3$) was taken into a test tube and then few drops of 0.1% ferric chloride added. It was mixed well till the appearance of brownish-green colour which indicates the presence of tannin.

iii. Test for flavonoids: The extract ($2cm^{3)}$ was taken into a test tube and mixed with 10% sodium hydroxide which results in greenish-brown colour which indicates the presence of flavonoids.

iv. Test for saponins: The extract ($2cm^{3)}$ was taken into a test tube and shaken with a small amount of distilled water. Appearance of frothing indicates the presence of saponin.

v. Test for steroids: The extract ($2cm^{3)}$ was taken into a test tube and mixed well with a few drops of chloroform. Then, a drop of acetic acid added and the mixture heated for few minutes after which few drops of concentrated sulphuric acid was added. Appearance of orange colour indicates the presence of steroids.

vi. Test for quinines: The extract ($2cm^{3)}$ was taken into a test tube and mixed with few drops of concentrated hydrochloric acid, appearance of green colour indicates the presence of quinine.

vii. Test for sesquiterpenoid lactones: The extract ($2cm^{3)}$ was taken into a test tube and mixed with 2ml of chloroform and then a few drops of conc. sulphuric acid added. Appearance of orange colour indicates the presence of sesquiterpenoid lactone.

viii. Test for terpenoids: The extract ($2cm^{3)}$ was taken into a test tube and mixed with 2ml of chloroform and then few drops of concentrated sulphuric acid added. Light orange colour appearance confirms the presence of terpenoids.

ix. Test for alkanoids: The extract ($2cm^{3)}$ was in attest tube, $1cm^3$ HCL was added and the mixture heated gently for 20min, cool and filter, $1cm^3$ of the filtrate was treated with wagner reagent, formation of brown reddish precipitate indicates the presence of alkaloid.

x. Test for glycosides: The extract ($2cm^{3)}$ was taken into a test tube and mixed with $2cm^3$ of glacial acetic acid and one drop of ferric chloride is added followed by $1cm^3$ of conc. sulphuric acid and is mixed well. Appearance of brown colour indicates the presence of glycosides.

xi. Test for anthocyanins: The extract ($2cm^{3)}$ was taken into a test tube and $2cm^3$ of 2% HCl and NH_3, the appearance of pink red turns blue violet indicates the presence of anthocyanin.

3.5 Optimization of Dyeing Conditions

The dyeing processing was optimized by varying dyeing temperature, dying time, and mordant concentration. The dyeing temperature was varied from 60^0C, 80^0C and 90^0. Dying time varied between 1hr, 45min, and 30min.With mordant (Alum) concentrations of 2%, 5%, and 7%.

In another pot, the alum solution was prepared for pre-mordanting. It was heated until it started to boil, then the fabric was added. The fabric was simmered in the fixative for at least an hour, then carefully pulled out from the simmering fixative and then squeeze out completely. The fixed fabric was immersed in the extracted dye, boiled and simmered until the fabric takes up the dye at least for an hour. The fabric was then removed and placed in air to dry.

Another deferent procedure conducted for co-mordating. In this procedure the mordant was added to the extract and the fabric was immersed into the extract, boiled and simmered until the fabric takes up the dye at least for an hour, while the time, temperature as while as pH were varied.

3.5.1. Dyeing Method for Reactive Dyes

Reactive dyeing was carried out by mixing the dyes to liquor ratio 1:15, at 65°C dyeing temperature for 60 minutes. The comparative studies of reactive dyes with natural dyes were carried out by using visual eye test. After thorough rinsing and soaping process, the colour coordinates of dyed samples were determined by using visual Colour Tool and fastness properties were also conducted according to the quality assurance tests of; Washing fastness, Rubbing fastness, and Light fastness.

3.6. Quality Assurance Tests of Dyed Fabric

Natural destructive agencies are light, weather, oxygen and other atmospheric gases which can fade and destroy certain dyes. In addition to natural agencies, there are many chemicals and finishing treatments used in wet processing textiles which may influence fastness of colours to some degree. Most dyes are organic compounds and are, therefore, vulnerable in varying degree to the action of destructive agents. Several tests for the assessments of fastness of dyes are available. A number of tests are necessary to cover all the important properties of any one dye, because good fastness to one inference is not necessarily accompanied by equal fastness to other conditions. Tests may be divided into those of customer's significance such as light, washing, rubbing, least an hour, then carefully pulled out from the simmering fixative and then squeeze out completely. The fixed fabric was immersed in the extracted dye, boiled and simmered until the fabric takes up the dye at least for an hour. The fabric was then removed and placed in air to dry.

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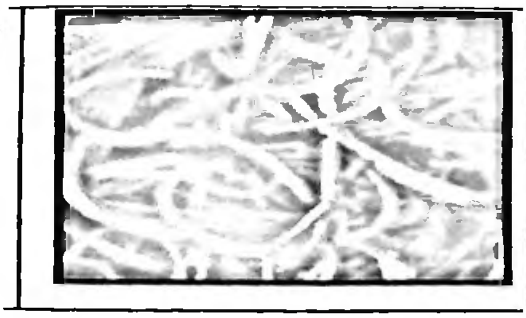
3.7 Application of Some Selected Dyes to Food

Roselle, Turmeric and Neem dyes were choosen for the preparation of Rice, Indomie noodles and Spaghetti, after the photochemical test. Hena and Eucalyptus were not used because of the presence of sesquiterpene lactones (SLs) which is known to cause allergy reactions in human. The extract at neutral pH of the above mentioned plants were used for the preparation of the food using separate pots.



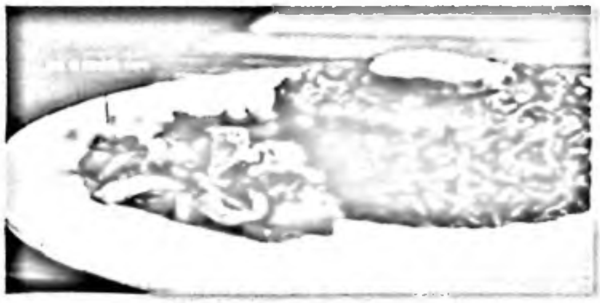
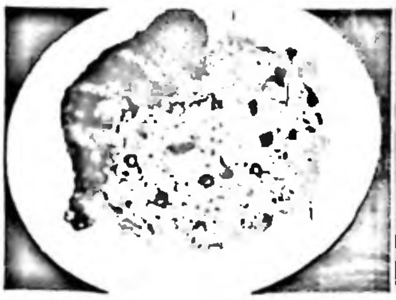


Plate A: Spaghetti

Plate B: Rice

Plate 3.2 Food items coloured with Hibiscus Sabdariffa dye (A&B)



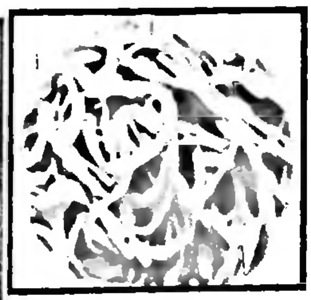
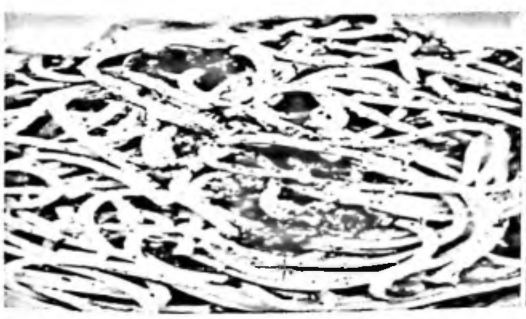


Plate **A:** Rice

Plate B: Indomie

Plate **C:** Spaghetti

Plate 3.3 Food coloured with Turmeric (A,B&C)



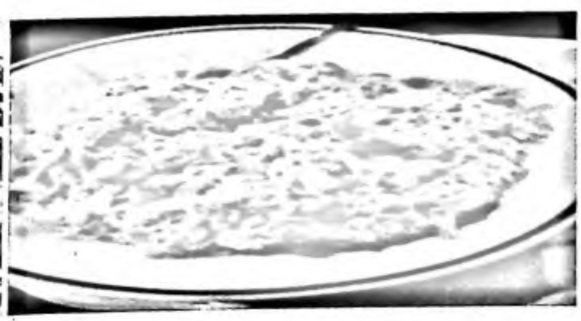


Plate A: Spaghetti

Plate B: Rice

Plate 3.4 Food coloured with Neem (A&B)

CHAPTER FOUR

DATA PRESENTATION AND ANALYSIS

4.1 Data Presentation

Table 4.1: Colour Intensity of Extracted Dyc Samples

NaOH	Temperature	Time	Roselle	Turmeric	Hena	Neem	Eucalyptus
Neutral	90-100°C	30min	Good	Good	Good	Good	Good
0.01 mol/dm^{-3}	60-65°C	1hr	Good	Good	Good	Good	Good
0.1 mol/dm^{-3}	80-85°C	45min	Excellent	Excellent	Excellent	ExceL.	Excellent
0.35 mol/dm^{-3}	90-100°C	30min	Good	Good	Good	Good	Good

Colour code grading (concentration) using improved Muscll colour value scalc:

Less than 40 = very weak, 40 – 59 = weak, 60 – 79 = Good, 80 – 100 = Excellent.

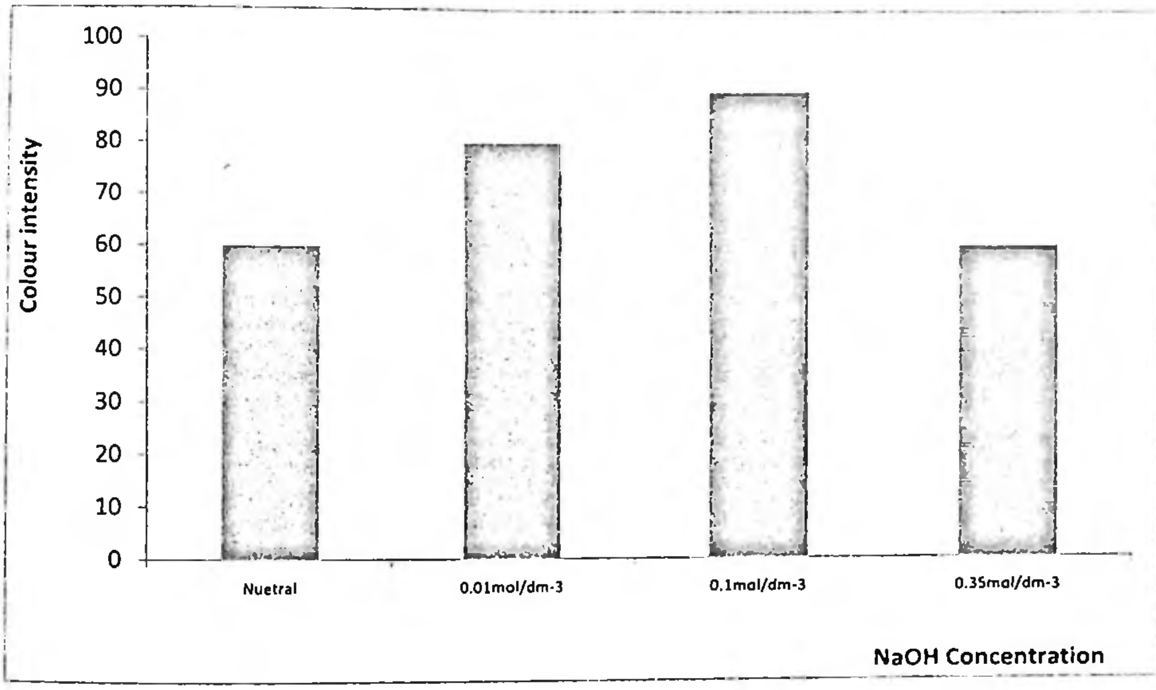


Figure 4.1 Concentration of NaOH against colour intensity

Table 4.2: Phytochemical Screening of Plants

Phytochemicals	Roselle	Turmeric	Henna	Neem	Eucalyptus
Phenols	+	+	+	+	+
Tannins	+	+	+	-	+
Flavonoids	+	+	+	+	-
Saponins	+	+	+	-	+
Steroids	-	-	+	+	+
Quinones	-	-	+	-	-
Anthocyanins	+	-	-	-	+
Terpenoids	-	+	+	+	-
Glycosides	-	+	+	+	+
Alkaloids	+	+	+	+	-
Sesquiterpene L	-	-	+	-	+

(+) = present (-) = Absent

4.3 Colour Intensity of Dyed Fabric

Alum%	Time	Roselle	Turmeric	Henna	Neem	Eucalyptus
Nil	1hr	Good	Good	Good	Good	Good
7	1hr	Good	Good	Good	Good	Good
5	1hr	Good	Good	Good	Good	Good
2	1hr	Excellent	Excellent	Excellent	Exce.	Excelle

Colour code grading (concentration) using improved Musell colour value scale:

Less than 40 = very weak, 40 – 59 = weak, 60 – 79 = Good, 80 – 100 = Excellent.

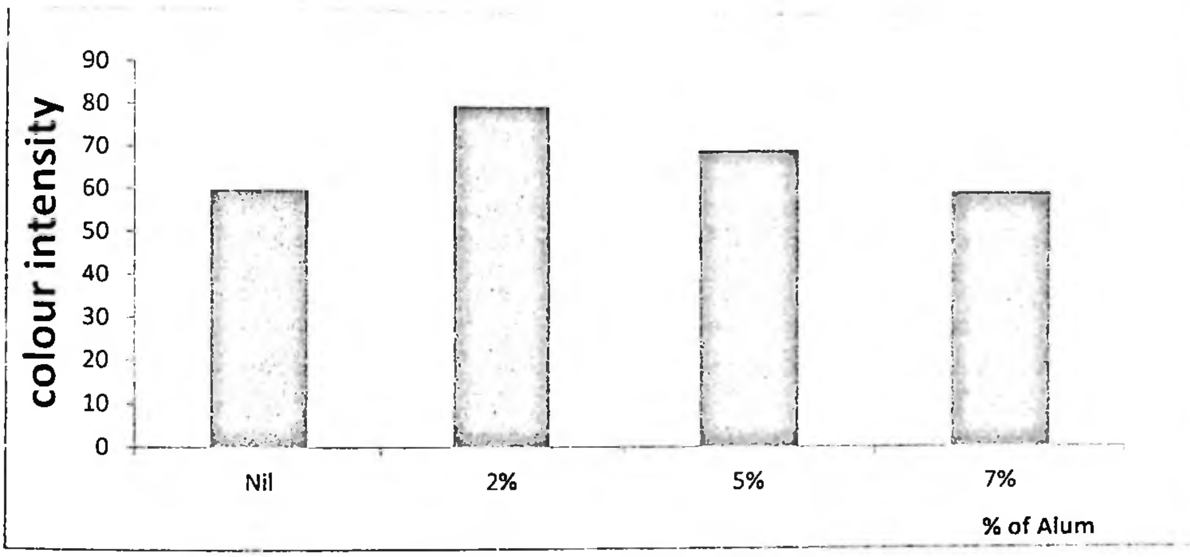


Figure 4.2 percentage of Alum against colour intensity in dyed fabric

Table 4.4: Colour Fastness Properties for Pre-mordating

Nature of test	Roselle	Turmeric	Hena	Neem	Eucalyptus
Washing	Good	Good	Good	Good	Good
Light	Good	Good	Good	Good	Good
Rubbing	Good	Good	Good	Good	Good

Colour code grading (concentration) using improved Musell colour value scale:

less than 40 = very weak, 40 – 59 = weak, 60 – 79 = Good, 80 – 100 = Excellent.

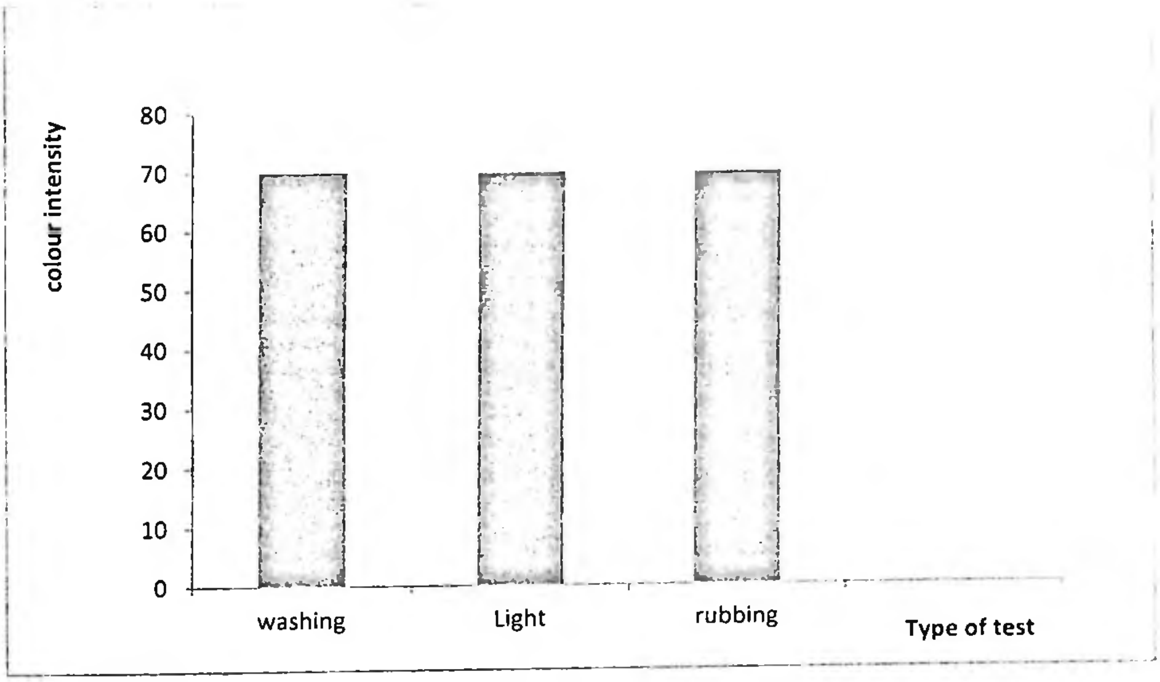


Figure 4.3 Colour fastness tests against colour intensity for pre-mordating

Table 4.5 Colour Fastness Properties for Co-mordating

Nature of test	Roselle	Turmeric	Hena	Neem	Eucalyptus
Washing	Week	Week	Week	Week	Week
Light	Good	Good	Good	Good	Good
Rubbing	Good	Good	Good	Good	Good

Colour code grading (concentration) using improved Musell colour value scale:

less than 40 = very weak, 40 – 59 = weak, 60 – 79 = Good, 80 – 100 = Excellent.

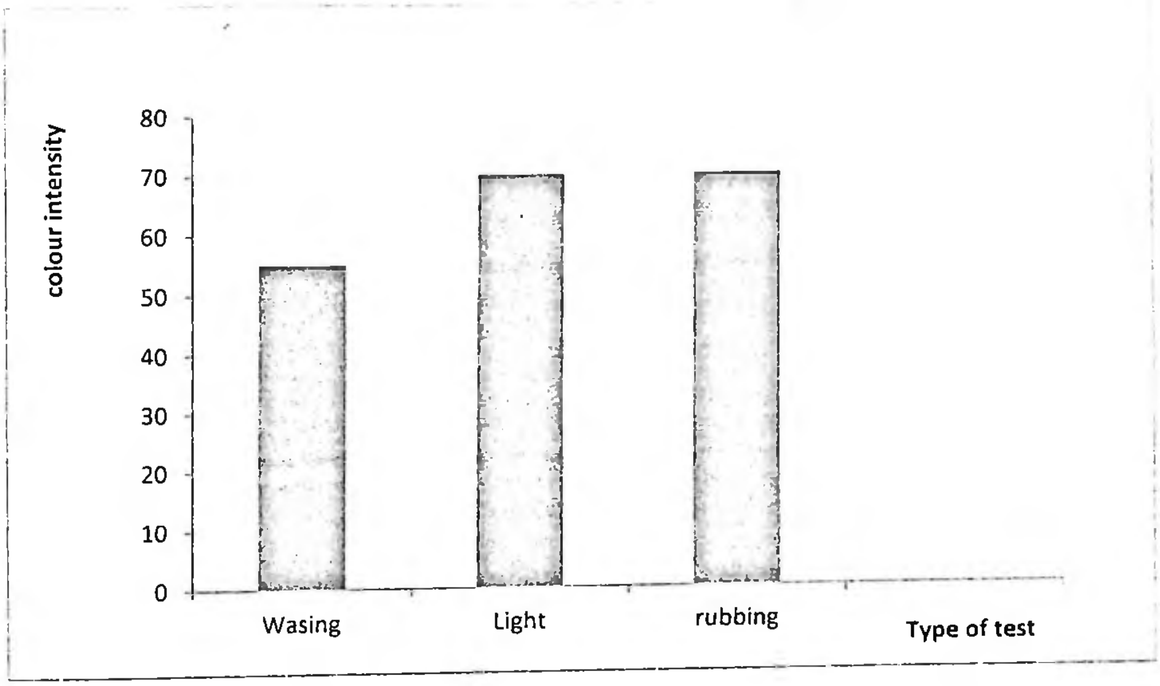


Figure 4.4 Colour fastness tests against colour intensity for co-mordating

4.2 Data Analysis

4.2.1 Effect of Extraction Medium on Relative Colour Strength:

The effect of extraction medium on the extraction of colouring component was conducted with different aqueous mediums (distilled water and NaOH solution). The colour strength value with alkaline medium is more than double in comparison with extraction in distilled water. The reason can be attributed to acidic hydroxyl group in naphthaquinone (ElNagar et.al., 2005), which reacts with alkali and form naphthaquinone salt which is more soluble in water. Thus the extraction of colouring component becomes more in alkaline medium. As cell wall is made up of cellulose material, this gains anionic charge in alkaline medium. Due to these anionic repulsive forces among cell wall, it loses its strength and ruptured easily in alkaline medium. This gives more colouring component into alkaline medium.

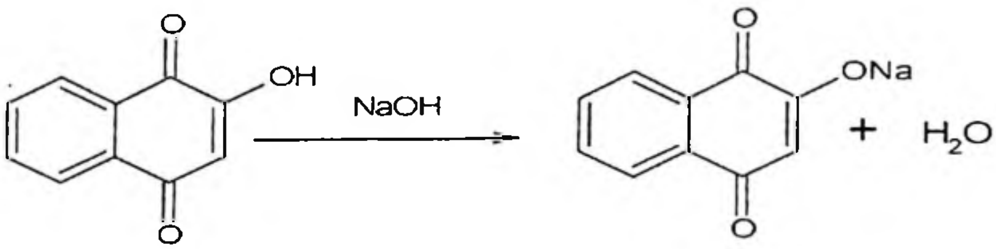


Figure 4.5 Reaction of Naphthaquinone + NaOH = naphthaquinone sal and water

The colour strength value of sample dyed with alkaline extract of Henna's leave is much higher in comparison with sample dyed with distilled water extract of Henna.

4.2.2 Effect of Extraction Temperature on Relative Colour Strength

The colour strength value increased with increase of temperature; thus the best selected temperature was 80-85°C for alkaline extraction and 90 - 100°C neutral. The reason of higher colour strength value at boiling is attributed to increase in solubility of colour component and rupturing of cell wall as well.

4.2.3. Effect of Extraction Time on Relative Colour Strength

There are significant indications that the colour strength increased with increase in extraction time and reached maximum value at 60 minutes. The effect of time for extraction can be correlated with the higher contact time of solvent with powder which grasped more colouring component into alkaline and further increase resulted in decrease in colour strength. But it might be due to decomposition of colouring component at higher temperature with more contact time (Nagia and Mohamedy, 2006).

4.2.4 Effect of Dyeing Temperature on Colour Strength

The effect of temperature on the dye ability of cotton with alkaline extract was conducted at various temperatures (40-90°C), it is clear that the colour strength increased with increase in dyeing temperature and reached a maximum value within 60-70°C then it decreased. Furthermore high temperatures caused lower colour strength and unevenness as well. This increase in dye uptake can be explained by fabric swelling and disaggregation of dye molecules into single molecule at higher temperatures (Shenhnai, 1997; Kamal et.al., 2006).

4.2.5 Effect of Dyeing Time on Colour Strength

The longer the dyeing time, the higher is the colour strength until dye exhaustion attains equilibrium and there is decrease in the colour strength after further increase in time from 100 minutes. This effect was attributed to shift in equilibrium of colourant from fabric to dye bath (Kamel et.al., 2006).

4.2.6 Effect of Dyeing Salt on Colour Strength

A salt effect on the dye ability for textiles is well established phenomenon and leads to increase in the dye exhaustion on the fabric. Increase in salt concentration increases the colour strength. The explanation could be that when cellulosic fabric is immersed in water it acquires a negative electrical charge (due to Zeta Potential). This negatively charged fabric surface repells negative charge dye anions. But Glauber's salt tries to reduce or neutralize negative charge on fabric. Thus it creates facilitation for the approach of the dye within the range of bond formation. The other reason for high colour strength value by increasing salt concentration is that more repulsive forces developed in aqueous extraction in the presence of more salt, which forces the dye molecules towards fabric (Shehnai, 1997).

4.2.7 Effect of Dyeing pH on Colour Strength

pH value of the dye bath has considerable effect on the dye ability on cotton fabrics while using alkaline aqueous extract. The effect of dye bath's pH can be attributed to the correlation between dye structure and cotton. Since in hena, the α-hydroxy-naphthaquinone has slight negative charge on higher pH, which could form H-bond with cellulosic hydroxyl groups. But further rise in pH made the dye and fabric more anionic which repelled each other and caused lesser dye ability on higher pH.

4.2.8 Effect of Pre-Mordanting With Alum On Relative Colour Strength

Among the 3 different concentrations (2, 5 & 7%) of mordant (alum) at two temperatures 60°C and 80°C for mordanting, the maximum colour strength was obtained with 2% at 80°C. It is indicated that colour strength decreased with increasing mordant concentration at both temperatures (60 & 80°C) of mordanting. All other samples except the sample dyed after 2% mordent have almost similar colour strength values. This low colour strength value can be attributed to the fact that alum forms quite strong bonds with dye but not with the fabric (Bhattacharya, 2000). Thus it bleeds during dyeing from treated cotton fabric, forming insoluble coloured complexes into dye bath. Furthermore, the colour strength value for samples dyed after pre-mordants at 80°C have higher than the samples dyed after premordant at 60°C, it might be due to more penetration of mordant into dilated fabric molecular at higher temperature (80°C). Thus the optimum sample with respect to relative % colour strength is sample dyed after premordant with 2% alum mordent at 80°C.

4.2.9 Effect of Co-Mordanting With Alum On Relative Colour Strength

The effect of co-mordanting on the colour strength (%) values at different concentration (2, 5 & 7 %), and at temperature (60 and 80°C). showed a correlation between the concentration of mordant and colour strength values. It can also be viewed that the sample without co-mordanting has least colour strength value in comprison with all other samples co-mordanted. This observation can be because the mordant increased the affinity of colourant to the fabric, even thought it has poor washing fastness which may be due to that mordant extracted the colour from the dyed fabric because of the affinity of colourant with mordant in case of alum Thus pre-mordanting appears to be the more favourable method in comparison to co-mordanting method in case of alum. These observations can also be correlated with the results given by Gupta et.al. (2004)

4.2.10 Comparison of Colour Coordinates For Sample Dyed With & Without Alum Mordanting

Mordant effect enhanced the uptake of colourant during dyeing. The colour strength value of sample dyed after mordanting (Alum) was higher.

4.3 Fastness properties

4.3.1Washing Fastness:

The washing fastness test was conducted for samples dyed with and without mordant which assessed in the grey scale rating for the staining of adjacent cotton material and alteration in colour on dyed fabric. It is clearly seen that the results of washing fastness for all the samples show moderate to good fastness. It is again observed that pre-mordanting in case of alum mordant were the better methods for the shade depth and fastness properties as well. This could be attributed to the formation of insoluble colour complexes. The better fastness to washing was due to the affinity of colouring component through H-bonding & vander waals forces for the sample dyed without mordant.

4.3.2 Light Fastness

The light fastness values obtained for samples without mordanting and samples with mordanting have no difference. The light fastness of samples dyed without mordanting show fairly good results due to the chemical structure of 2-hydroxy-1,4-naphthaquinone in henna. The 2-hydroxy group develops the hydrogen bonding with carbonyl group of naphthaquinone which protect the chromophoric group from fading (Christie, 2001). The similar values of all samples including mordanted and nonmordanted could be correlated that coloured complexes have shown similar stability as indicated by the 2-hydroxy-1,4-naphthaquinone. The moderate light fastness was due to the smaller microscopic particles during mordanting.

4.4 Comparison of shades of natural dyes extract with synthetic dyes

4.4.1 Washing fastness comparison

The washing fastness of fabric dyed without mordant, with optimum mordant concentrations for natural dyes (alkaline extract) and sample dyed with reactive dyes show the usual way in terms of the grey scale values for the staining of adjacent cotton material & alteration in shade. It is indicated that sample with natural dye extract & mordanted gave rating good to very good washing fastness in comparison to the very good to excellent rating for shade developed with reactive dyes. The very good to excellent washing fastness of sample dyed with reactive dyes was due to the strong covalent bonds between the reactive dye molecules and the fabric. But it can be seen that sample dyed with natural dye extract are also comparable to sample dyed with reactive dyes.

4.4.2 Light fastness comparison

The light fastness values of fabric dyed with alkaline extract with and without mordant and with reactive show results for reactive dyes were good to better according to grey scale reading. The higher light fastness properties of reactive dyes can be attributed to the strong intramolecular H bonding which exists in the form of six member rings (Christie, 2001). This enhances the stability of the compound by a reduction in electron density at the chromophore. As a result sensitivity of dye towards photochemical oxidation becomes reduced. But such strong interaction is not strongly present in the α-hydroxy naphthaquinone. Here it is noted that sample dyed without mordant has same and even better fastness to light in comparison with sample dyed with mordant. It can be attributed to the catalytic effect in reducing colouring component of mordant.

4.4.3 Rubbing fastness comparison

The rubbing fastness values of sample dyed with alkaline extract in the presence & absence of mordant and dyed with reactive dyes shows that both types of dyes have almost similar behaviour towards rubbing fastness. This observation can be correlated to the fact that both dyes equally penetrated into the cotton fabric.

5.5 Dyes applied to food

The Hibiscus sabdariffa dye presented deep pink attractive colours for spaghetti and rice. Also, the drink produced from it looked very pleasing and mouth watering just as normal wine produced from berries. Also the rich golden yellow colour obtained from turmeric give rice, indomie and spaghetti an appealing and aesthetically pleasing, attractive and highly decorative colour, for parties. Neem give a greenish to yellow colour that is pleasant to the eye just like turmeric

CHAPTER FIVE

SUMMARY, CONCLUSION AND RECOMMENDATIONS

5.1 Summary

The extraction process focused on leaves, calyces, and rhizomes sample of 5 different plants, each of which has known medicinal properties and traditional uses for various ailments and diseases. Both plants yielded strong dyes that coloured cotton fabric with and without application of mordant. However, application of mordants to these dyes caused slight changes to the colour of dyes obtained, depend on the concentration.

When rice, indomie and spaghetti were cooked in selected dye solutions, Turmeric dye yielded golden colour on food while Neem dye turned the cooked food colours yellowish green. Tests with the Hibiscus sabdariffa, which found use as a natural drink, results suggest that aesthetically pleasing colours of natural dye and food additives can be derived from the local environment. These can be adapted for health and appetite benefit. The results also indicate the feasibility of local plants dye extracts as suitable organic replacements for the chemical colourants used in the textiles and food industry. It is important for chemistry practitioners to explore and adapt resources in the local natural reserves for promoting natural products. The success of this researched have potential for textile dyers and personnel in food industry, such as those engaged in cake and confectionery food who must explore the potential of plant products for their decoration so that, they can create pleasing, healthy and attractive consumable products that result from imaginative thinking.

These natural raw materials can help improve the appeal of many local foods the same way as margarine manufacturers add yellow colouring to make their product look like butter. The beautiful colours can give various textile and food products the appeal that consumers cannot overlook in making purchasing decisions. Besides, these natural dyes can ensure the safety and wholesomeness of processed food and promote healthy living just as parts of the plants are used for healing purposes.

5.2 Conclusion

The result of the experiment with different parts of the identified plants was a remarkable range of colourful direct dyes of various shades and tints. The dyes were colourfast on cotton fabric and could be used directly without mordants to teach basic skills in tie-dye. Most of these natural dyes could be used to colour textile products i.e. cotton thread for teaching macramé, crocheting and food. The coloured dye extracts on textile fabric and food cannot be over emphasized. For instance the yellow dye obtained from Turmeric gave a brilliant colour to a cotton fabric whiles at the same time, serving as a yellow food colourant for cooked rice, spaghetti and indomie.

The results also show the potentials of natural dye extracts that can be used as colourants for food and textiles. It is however, imperative for the food and textiles industry to develop effective strategies for incorporating this experiment into their core research and business development so that they can explore the other plants and come up with more scientific ways of producing and preserving these plant dyes and food colourants for extensive use.

This will also offer opportunity for students to learn skills in tie-dye, batik and printing, dyeing of yarns for macrame, and crocheting at little or no cost as compared to synthetic dyes.

5.3 Recommendations

The benefits of exploring plant dyes and their application on food and textiles will improve natural chemistry practice, which will as while contribute to national development.

In view of the results of the researched, the following are recommended for consideration.

i. It is important for the food and textiles industry to develop effective strategies for incorporating this experiment into their core research and business development so that they can explore the other plants and come up with more scientific ways of producing and preserving these plant dyes and food colourants for extensive use.
ii. Chemistry students should be mandated in all level to use plant dye during their colour chemistry and dye practical period to encourage the exploration of the natural dye from our natural reserved, which apart from health and environmental effect could enhance national development. It is also an opportunity for students to learn skills in tie-dye, batik, printing, dyeing of yarns for macramé, and crocheting at little or no cost as compared to synthetic dyes.
iii. Slightly acidic pH conditions should be used for the extraction and the result compared to neutral and alkaline pH extraction
iv. Sample harvesting should be carried out during the dry season and the results compare with the raining season to determine the effect of weather on the yield of dye
v. Vocational institutions, Non-Governmental Organizations, public and private agencies involved in skills training and youth employment can engage their

trainees in the cultivation of herbal plants for sustainable extraction of dyes that could further develop and make available to educational institutions, the textiles and food industry, as well as for the pharmaceutical and beverages industry.

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