Developing tints and shades in cotton fabric using *Rubia tinctorum* root *and Caesalpinia sappan* wood extracts

By

P C POORNIMA

(21PBX005)

A Thesis Submitted to the

Avinashilingam Institute for Home Science and Higher Education for Women Coimbatore -641043

IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF MASTER OF SCIENCE IN BIO-TEXTILES

MAY 2023

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Certified as Bonafide Research Work

Signature of the Head of the Department **Signature of the Supervisor**

CERTIFICATE FROM THE SUPERVISOR

I certify that dissertation entitled **"Developing tints and shades in cotton fabric using *Rubia tinctorum* root and *Caesalpinia sappan* wood extracts"** submitted for the degree of Master of Science (M.Sc.,) Bio-Textiles by P C POORNIMA (21PBX005) is the record of project work carried out by her during the academic year 2022 to 2023 under my guidance and supervision and this work has not formed the basis for the award of any Degree, Diploma, Associate ship, Fellowship, Titles in this University or any other similar institution of higher learning

Signature of the Supervisor with Designation

13/5/23

Signature of the HOD

DECLARATION

I declare that the dissertation entitled "**Developing tints and shades in cotton fabric using *Rubia tinctorum root and Caesalpinia sappan wood* extracts**" submitted by me for the degree of Master of Science (M.Sc.,) is the record of work carried out by me during the period from 2022 to 2023 under the guidance of Dr. G. BAGYALAKSHMI, M.Sc., M.Phil., Ph.D., Associate Professor, Department of Textiles and Clothing, Avinashilingam Institute for Home Science Higher Education for Women, Coimbatore -641043 and has not formed the basis for the award of any Degree, Diploma, Associate ship, Fellowship, Titles in this University or any other similar institution of higher learning.

Signature of the Candidate

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INTRODUCTION

I. INTRODUCTION

Colour has always played a dominant role in human life. It has played an important role in bringing beauty to the world in every civilization from the Stone Age to the 'silicon floating age' (Bhuyan and Saikia, 2004). The dyeing of textiles with various dyes and the use of dyes for fashionable pattern effects is gaining increasing attention (Nagendrappa, 2010). Dyes can be either natural or synthetic, both of which are used to dye fibers, threads and fabrics (Kajla and Srivastava, 2006). The advent of "Mauvein" by Henry Perkin in 1856 and the subsequent commercialization of synthetic dyes replaced natural dyes (Gupta, 2019). Synthetic colourants have become the standard in many applications such as cosmetics, textiles and food colouring, surpassing natural colourants. Although these dyes have many advantages such as colour versatility, low cost and excellent fastness, they also have disadvantages such as allergenicity, toxicity and even carcinogenicity, which are harmful to humans and the environment (Saxena et al., 2014). Natural dyes are making a comeback as people become more conscious of environmental concerns and sustainability (Kodalph, 2008). Nature has gifted lots of dye yielding plant species, animal and mineral which can be usedfor dyeing.

Natural colouring agents are derived from various parts of plants such as leaves, fruits, roots and barks etc (Vankar,2007; Pisitsak et al.,2016). These are biodegradable, eco-friendly and renewable source compatible with environment and less allergic (Baaka et al.,2016; Khan et al., 2016). Natural dyes are well known for producing very uncommon, soothing and soft shades as compared to synthetic dyes. This shift in paradigm in favour of natural dyes is also attributed to the stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes (Grover andPatni,2011).

India has a rich biodiversity, with over 450 plants that provide dyes and pigments (Mahesh et al., 2011). However, many of these plant dyes have not yet been fully investigated for their textile dyeing potential. There is an urgent need to develop effective scientific technology. Most of the plants used for dye extraction are classified as medicinal, some of which have recently been shown to have antibacterial activity (Mahesh et al., 2011; Chengaiah et al., 2010). It also pointed out the need to reconsider the application of natural dyes to textiles and to restructure the traditional natural dyeing process to control each treatment and pre-dyeing process (preparation, ribbing) and dyeing process. I was variables for producing unusual shades with balanced colourfastness and eco-friendly textiles (Alemayehu, 2014; Tripathi et al., 2015; Samanta and Agarwal, 2009; Pierce, 1993).

Natural dyes have a low affinity with fabrics (cotton, wool, silk) and require the use of dyes. The word 'mordant' is derived from the Latin word 'modern', which means 'to chew' (Prabhu and Bhute, 2012). Mordants are generally salts of metals such as aluminum, iron, chromium, and copper to ensure proper colour durability against sunlight and washing (Geelani et al., 2016; Chungkrang et al., 2018). Mordants are known as elements that help bind dyes to fabrics by forming chemical bridges between the dye and the fibre, thus increasing the colouring ability of the dye and increasing its fastness (Padma, 2000). Natural dyes can produce a wide variety of colours when mixed with other substances. Using different stains with the same dye can shift colours across a broad spectrum or create entirely new colours, which is not readily possible with synthetic dyes (Tasneem and Maria, 2016).

Until now, the use of natural dyes to dye textiles was mainly limited to craftsmen. Artisans, small-scale dyers and printers, small-scale exporters and producers engaged in the production and sale of high-quality, environmentally friendly textiles around the world. Recently, many commercial dyers and small textile exporters have started to explore the possibility of using natural dyes for dyeing and printing ordinary textiles (Agarwal, 2009). The global annual demand for natural dyes is estimated at about 10,000 tones, equivalent to 1% of the global consumption of synthetic dyes (Punrattanasin et al., 2013). Therefore, new pigment plants and modern dyeing techniques are being researched to meet the growing demand for natural dyes (Shukla et al., 2013).

Common drawbacks of natural dyes are standardized dyeing methods due to irreproducible and inconsistent hues, moderate colour fastness, and lack of scientific information on extraction and dyeing chemistries (Bains, 2005; Kumaresan et al., 2012). Extraction of colour components from natural sources is an important step in dyeing textile substrates in order to assess dyeing properties and maximize the colour yield of textile fabrics (Mansour, 2009; Bukhari et al.., 2014). Parameters such as pH, time, temperature and dye concentration for dye extraction and staining are optimized conditions in standardized staining procedures. These are called standardization techniques (Chakrabarti et al., 2011). Both extraction process standardization and extraction variable optimization have technical and commercial impacts on colour yield and extraction process costs, as well as staining costs (Mansour et al., 2019).

Traditional aqueous extractions are typically solvent and time consuming and produce less coloured components compared to using water as a co-solvent with acetone, ethanol and/or dimethylformamide. properties (Mansour et al., 2011; Morshed et al., 2016). Standardized recipes and dyeing techniques help preserve natural dyes as reproducible dyes, create new shades, improve the fat liquoring process and dye affinity for all natural fibers. AccepTable To obtain new shades with colour fastness and reproducible colour yields, dyers can routinely use standardized dyeing process parameters (Sinnur et al., 2018). Many reports are available on application of natural dyes on cotton (Kumar et al., 2004; Kumaresan et al., 2010). The present investigation deals with the extraction of natural dye from the extract of Rubia tinctorum root and Caesalpinia sappan wood at different optimized conditions.

Rubia tinctorum belongs to the family of **Rubiaceae** and comprises about 70 species (Eltamany et al., 2020) commonly known as Madder or Indian Madder or Manjistha Common madder is native to West Asia and the Mediterranean region and has been grown and used as a dye across Asia for centuries. Indian madder is a cultivated species of madder that contains high amounts of alizarin, which is the main compound responsible for producing red in most natural dyes. The herb has also been used to treat various skin concerns and also for brightening the complexion. The herb is enriched with a compound called Alizarin which is an effective anti-bacterial. Application of Manjishta is known to soothe dry skin, allergy and any kind of rash or inflammation, (Kalyoncu, *et,al,* 2006)

Caesalpinia sappan belongs to the family Fabaceae It was previously ascribed to the genus **Caesalpinia**. Sappan wood is related to brazilwood (***Paubrasilia echinata***), and was itself called brasil wood in the Middle Ages. This plant has many uses. It has antibacterial and anticoagulant properties. It also produces a valuable reddish dye called brazilin, used for dyeing fabric as well as making red paints and inks. Slivers of heartwood are used for making herbal drinking water in various regions, such as Kerala, Karnataka and Central Java, where it is usually mixed with ginger, cinnamon, and cloves. The heartwood also contains juglone (5-hydroxy-1,4-naphthoquinone), which has antimicrobial activity. Homoisoflavonoids (sappanol, episappanol, 3'-deoxysappanol, 3'-O-methylsappanol, 3'-O-methylepisappanol and sappanone A can also be found in B. sappan, (Mathew, *et.al,* 2007).

Hence the present study "**Developing tints and shades in cotton fabric using Rubia tinctorum root and *Caesalpinia sappan* wood extracts"** was designed with the following objectives.

- To select natural dye source for mordanting and dyeing
- To extract and optimize dye from the selected plant sources.
- To select and prepare the cotton fabric for dyeing with natural surfactants
- To optimize the dyeing conditions to develop the tints and shades of the selected plants sources
- To access the colour strength and evaluate the dyed fabric for performance properties.

REVIEW OF LITERATURE

II.REVIEW OF LITERATURE

The review of available literature pertaining to the present study of **"*Developing tints and shades in cotton fabric using Rubia tinctorum root and Caesalpinia sappan wood extracts*"** has been explained under the following headings

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2.1 Natural fibre – Cotton

Cotton is the most popular natural fibre, accounting for around 90% of all natural fibres. Cotton is one of the most important natural textile fibre crops, both from the agricultural and manufacturing sectors. It is the biggest source of clothing as well as being used to produce apparel, home furnishings, and industrial products. Cotton, the seed hair of plants of the genus Gossypium, is the purest form of cellulose available in nature. After flowering, an elongated capsule or boll is formed on the cotton plant in which the cotton fibres grow. Once the fibres have completed their growth cycle, the capsule bursts and fibres emerge. A cotton capsule contains about 30 seeds. Each seed contains around 2000– 7000 seed hairs (fibres). Depending on the cotton type and growing conditions, the colour of the fibre is usually creamy white or yellowish. Cotton fibre is mostly composed of cellulose. Under 10% of the weight of the raw fibre consists of waxes, protein, pectate and minerals (Dochia et al., 2012).

Based on archaeological evidence, humans utilized cotton fibre from at least more than four to seven thousand years ago, and cotton started to be grown as a fibre crop around three thousand yearsago (Lee et al., 2015). The main countries producing cotton in the world are China, United States, India, Pakistan, Uzbekistan, Turkey, and Brazil are the main countries which producing cotton worldwide together account for over 80% of the world's cotton production. Cotton is also called as "Fibre King" and it also spells as "the White Gold" due to its spin-ability (Priyanka and Jayakumari, 2020). Cotton fibres are may be classified roughly into three large groups based on staple length and appearance.

- **Long-staple cotton**: This type of fibre is fine and lustrous with typical staple length ranging from about 30 to 40mm, and includes types of the highest quality, such as Sea Island, Egyptian,and Pima cottons. Due to the difficulty in its cultivation and its limited production, long-staplecottons are costly and are used mainly for fine fabrics, yarns, and hosiery.

- **Medium-staple cotton**: It is plentiful and standard, such as American Upland, with staple length from about 25 to 33mm. Medium-staple cotton provides about 90% of the current world production of raw cotton fibre. This fibre is widely used for apparel, home furnishings, and industrial products.

- **Short-staple cotton:** This is coarse cotton, ranging from about 10 to 25mm in length, which makes textile processing very difficult and consequently it is not commonly used in textiles, except for very low-grade products. (Yu, 2015).

Table I

Chemical composition of cotton fibre

Per cent (dry basis)			
Constituents	**Typical**	**Low**	**High**
Cellulose	94.0	88.0	96.0
Protein	1.3	1.1	1.9
Pectic substances	0.9	0.7	1.2
Ash	1.2	0.7	1.6
Wax	0.6	0.4	1.0
Malic, citric and other organic acids	0.8		1.0
Total sugars	0.3		
Pigment	Trace		
Other	0.9		

Molecular composition of cellulose

Cellulose is a linear polymer, or long chain molecule, combining several 1000 an hydro glucose units. Although glucose, a simple sugar, is soluble in water, cellulose is not due to its huge polymolecule size. Cellulose is a carbohydrate, composed of carbon (44.4%), hydrogen (6.2%), and oxygen (49.4%); the cellulose molecule with the basic units of cellobiose. Cellobiose is formed by two combined glucose units; many cellobiose units combine to form cellulose. The number of an hydro glucose units in the cellulose molecule is referred to as the degree of polymerization. Each repeating unit equals 2n, or two an hydro glucose units. These units are flipped over as they combine together (Yu, 2015).

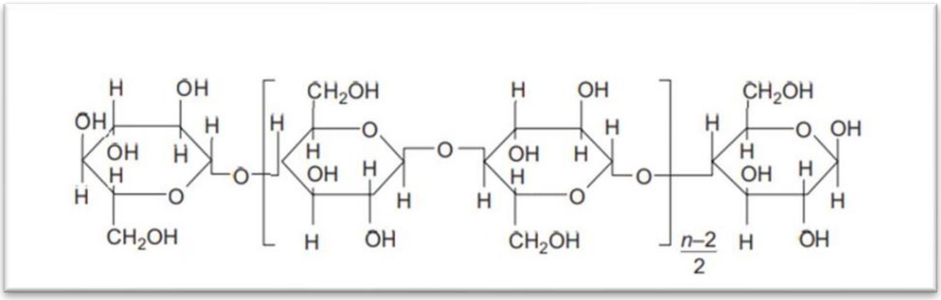


Figure 1 - Cellulose molecule

2.1.1 History of Cotton

Cotton bolls and pieces of cotton cloth was found to be used from 7000 years old in Mexico. In the Indus River Valley in Pakistan, cotton was being grown, spun and woven into cloth 3,000 years B.C. At about the same time, native of Egypt's Nile valley were making and wearing cotton clothes. Arab merchants brought cotton cloth to Europe about 800 A.D. By 1500, cotton was known generally throughout the world. Cotton was first spun by machinery in England in 1730. The industrial revolution in England and the invention of cotton gin in the U.S paved way to the cotton to hold important place in worldwide due to this it holds the world (Gillow, 2004). East India Company introduced cotton to Britain in the 1690s. New inventions in the 1770s—such as the spinning jenny, the water frame, and the spinning mule— and industrial revolution, made the British Midlands into a very profiTable manufacturing centre (Prasad, 2018).

American cotton industry starts growing with invention of cotton gin in 1793 by Eli Whitney. By the early 1830s the United States produced the majority of the world's cotton (Philips and Lakwete, 2004). Current estimates for world production are about 25 million tonnes or 110 million bales annually, accounting for 2.5% of the world's arable land. India is the world's largest producer of cotton. The United States has been the largest exporterfor many years (Deshmukh and Mohanty, 2016).

2.1.2 Characteristics of Cotton

Cotton has a number of distinguishing characteristics that make it such a popularfibre in the textile industry.

- **Softness:** The cotton plant is soft and fluffy and results in a fabric often retains that soft feel.
- **Durability:** The cotton plant's cellular structure is strong, creating a tough andwear-and-tear resistant fabric.
- **Absorbency**: Cotton fabric is very absorbent fabric because there is a lot of space between the cotton fibres.
- **Holds dye well:** Due to its absorbent nature, cotton takes dye very easily and can be made into a wide variety of colours.
- **Breathability**: The fibre structure of cotton makes it more breathable than synthetic fibres.
- **No static cling:** Cotton does not conduct electricity, therefore static is not an issue

with cotton. (Furstenberg, 2021).

2.1.3 Properties of Cotton

The molecular arrangement within the fiber and the conditions of fiber formation, impact the properties that make cotton fiber readily distinguished from all other textile fibers. All significant fibre properties are classified in relevant groups (Steadman, 1997; Hunter,1998).

- Properties according to Length
    - Staple Length Values
    - Span Length Values
    - Uniformity
    - Parameters for Length Distribution
    - Short Fiber Content
- Transverse Dimensions of Cotton
    - Micronaire
    - Fineness
    - Maturity
- Tensile Properties
    - Strength
    - Breaking Elongation.
- Non-Lint Content
    - Average Trash
    - Trash Particle
    - Size Distribution
    - Trash Type
- Dust
    - Level and Size

    - Seed Coat Fragments
    - Foreign Matter and Contaminants
    - Neps
- Cotton Colour
- Miscellaneous Fiber Properties
    - Fiber Friction
    - Cleanability
    - Microbial Attack
    - Cohesiveness
    - Compressibility and Resilience
    - Moisture Content (Rathore, 2022).

Fibre elongation is an important property that has received little attention so far. The role of elongation has not been fully defined yet but there is evidence that it strongly influences processing efficiency (Kechagia, 1996).

Physical properties of cotton:

High-quality cotton lint can produce high-quality yarn, fabric, and therefore high-quality end-products. High-quality cotton lint has a number of physical properties, some of which are measurable, whereas others are not.

- **Colour:** The colour of cotton fibre varies from almost pure white to a dirty grey. However, the standard cotton grades classify colour as white, light spotted, spotted, tinged, yellow stained, and light grey. High quality cotton is usually very light or almost white.
- **Tensile Strength:** Cotton is moderately strong fibre; tenacity is 26.5-44. Tensile strength 2800-8400 Kg/cm^2. The strength is greatly affected by moisture, the wet strength of cotton is 20%, which is higher than dry strength.
- **Elongation at break:** Cotton does not stress easily. It has an elongation break of 5-1%.
- **Specific Gravity:** Specific gravity is 1.54

- **Moisture regains**: Cotton has a moisture regain of 8.5%. At 100% humidity, cottonhas an absorbency of 25-27%.
- **Elasticity, stiffness and rigidity:** Elasticity, stiffness resilience, and rigidity Compared to other fibres the elasticity of cotton is relatively low with elongation of only 3–7%. Like other vegeTable fibres, cotton fibre has low resilience so products made from pure cotton crease easily and do not recover well from wrinkling Cotton fibre being finer than most other vegeTable fibres is soft with low rigidity. (Yu, 2015)
- **Tenacity:** The strength of cotton is increased by its long polymers, the countless, regular, hydrogen bond formation between adjacent polymers and the spiraling fibrils in the primary and secondary cell walls. It is one of the few fibres which gains strength on wetting which occurs due to improved arrangement in the amorphous region of the fibre (Asaduzzaman et al., 2016).
- Elastic – plastic nature: Cotton fibre is inelastic in nature because of its crystalline polymer system and hence for this reason cotton fibres wrinkle and crease easily. The polymer molecules can slide past each other only under considerable strain as they are prevented from doing so by their long lengths and countless hydrogen bond between them to hold the polymer molecules in position. Strong strain forces cause polymer fracture as the crystallinity of the polymers makes it difficult to bend and crush. These polymer fracture forms weak points on the polymer system and hence forming weak areas in the fibre structure. Therefore, cotton fibres get creased and wrinkled on the application of force. (Kozłowski, 2012)
- **Hygroscopic nature**: The cotton molecule is very moisture absorbent due to its abundant polar –OH group which attracts water molecules towards it. But the OH molecule can only enter the cotton molecule through the amorphous region as the intermolecular spaces in the crystalline region are very small. The swelling up of cotton fibre in presence of water is mainly due to the separation of polymers by water molecules in the amorphous region. Cotton fibres usually feel crisp on touching as it absorbs moisture from the skin of the finger. The hygroscopic nature of the fibre prevents cotton materials to develop static electricity. The polarity of the water molecule is attracted by the hydroxyl group of the polymer and hence static charges are dissipated (Prasad, 2018).
- **Thermal properties:** Cotton fibre has the ability to conduct heat energy, minimizing any destructive heat accumulation. Hence cotton fibres can withstand hot ironing

temperatures. But excessive heating on cotton fibre chars and burns them indicates that cotton fibre is not thermoplastic. This prevents the fibre from assuming the new position of the polymers when heat is applied. The polymers begin to vibrate on heating and gradually disintegrate (Chatterjee, 2021).

Chemical properties of Cotton

- **Effects of alkalis –** These fibres are resistant to alkalis and are comparatively unaffected by normal laundering. The resistance is because of the lack of attraction between the cottonpolymers and alkalis. Abidi, N. (2018).
- **Effect of Acids –** Cotton fibres are weakened and destroyed by acids. Acids hydrolyze thecotton polymer at the glycosidic oxygen atom which connects the two glucose units to form the cellobiose unit. Mineral acids being stronger than organic acids will hydrolyse the cotton polymer more quickly (Gordon and Hsieh, 2007).
- **Effect of Bleaches** – The most common bleaches used on cotton textile materials are sodium hypochlorite and sodium perborate. They are: oxidizing bleaches and bleach because of the oxygen liberated from them, (Dumitrescub et al. 2012)
- **Effect of Sunlight and weather –** The ultra-violet rays of sunlight provide photo chemical energy whilst the infra-red rays provide heat energy essential to degrade the cotton polymers in the pressure of atmospheric oxygen, moisture and air pollutants. The breakdown of polymers takes place through diverse hydrolysis reactions. The beginning degradation is noticed as a slight fibre discolouration. Fading of coloured cotton textile is partially because the breakdown of the dye molecules in the fibre's polymer system. (Sinclair, 2014)
- **Colour Fastness** – Cotton is easy to dye and print. The classes of dye which may be used to colour cotton are azoic, direct, reactive, sulphur and vat dyes. The polar polymer system easily attracts any polar dye molecules into the polar system. Therefore, dye molecules which can be dispersed in water will be absorbed by the polymer system of cotton. However, the dye molecules can enter solely the amorphous regions of the polymer systemof cotton. The small inter polymer spaces in the crystalline regions of the polymer systemprohibit the entry of the crystalline molecules Sahoo et al. (2014).

- **Mildew** – Cotton is damaged by fungi. Heat and dampness support the growth of mildew.The fungi produce a chemical compound which has the power of changing cellulose to glucose. The fungi feed on the molecules of sugar: Cotton treated with acrylo nitrite is resistant to mildew (Adanacioglu and Olgun, 2012)

- **Insects –** Moths and beetles do not change cotton. Silver fish will eat cotton cellulose especially if heavily starched.

2.1.4 Structure of Cotton fibre

Cotton fibres have a multi layered structure that has been studied for nearly a century. The structure of the primary cell wall of the cotton fibre, and particularly the outer surface layer (the cuticle), has a major influence on fibre properties, processing and use (Degani et al., 2004). Cotton fibre has a fibrillar structure which consists of a primary wall, a secondary wall and a lumen. Most of the non-cellulosic materials are present in the outer layers of cotton fibre (Agarwal, 2005; Mangat, 2009). Under a microscope a cotton fibre looks like a twisted ribbonor a collapsed and twisted tube (Heikinheimo, 2002).

These twists are called convolutions: there are about 60 convolutions per centimetre. The convolutions give cotton an uneven fibre surface, which increases inter-fibre friction and enables fi ne cotton yarns of adequate strength to be spun. The cross-section of a cotton fibre is often described as being kidney-shaped. The outer most layer, the cuticle is a thin film of mostly fats and waxes. The waxy layer forms a thin sheet over the primary wall that forms grooves on the cotton surface. The primary wall comprises non-cellulosic materials and amorphous cellulose in which the fibrils are arranged in a criss-cross pattern. Owing to the non-structured orientation of cellulose and non-cellulosic materials, the primary wall surface is unorganized and open. This gives flexibility to the primary wall, which is required during cell growth. The basic ingredients responsible for the complicated interconnections in the primary wall are cellulose, hemicelluloses, pectin's, proteins and ions. The secondary wall, in which only crystalline cellulose is present, is highly ordered and has a compact structure with the cellulose fibrils lying parallel to one another (Dochia et al., 2012).

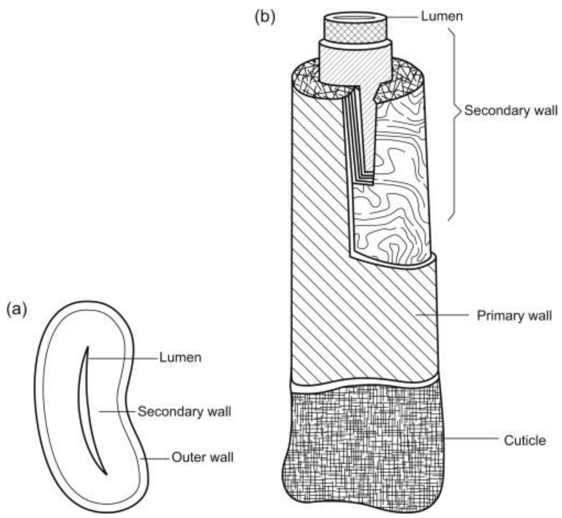


Figure 2 - Cross sectional view and vertical view of cotton fibre
(Miraftab, M. 2009)

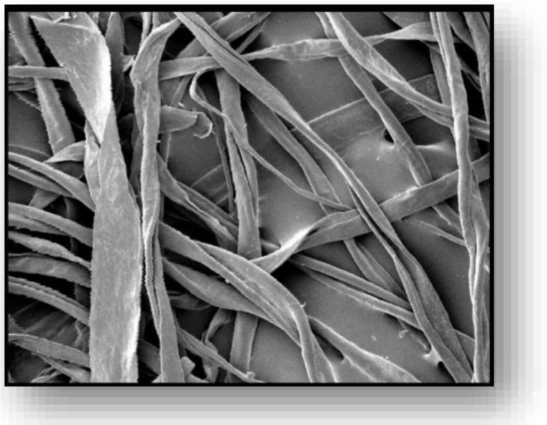


Figure 3 - Microscopic view of cotton fibre (Miraftab, M. 2009)

2.1.5 Advantages of cotton

- Cotton is inexpensive fabrics.
- It has absorbent properties.
- Cotton has high breathable property
- It is easier to wash.
- ComforTable to wear and soft against silk
- Cotton fabric can withstand to wear and tear
- Cotton fibre has greater resistance to alkali
- It has good heat resistance.
- Those are easily wash and care able.
- It does not irritate skin

2.2 Natural dye

2.2.1 Introduction

Natural dyes are colourants obtained from plants, invertebrates, insects, fungi or minerals. Most natural dyes are vegeTable dyes, the main sources of which are various parts ofplants such as roots, stems, seeds, barks, leaves and wood. There are also other biological sources such as fungi, snails, insects, etc. Natural sources were the main source of textile dyes before chemically dyeing (Hossain, 2021; Shanmathi et al., 2016). Since prehistoric time natural dyes is used for colouring of food substrate, leather as well as fibres like wool, silk and cotton. The use of non-allergic, non-toxic and eco-friendly natural dyes on textiles have become a matter of significant importance due to the increased environmental awareness in order to avoid some hazardous synthetic dyes (Agarwal, 2009).

2.2.2 Classification based on Origin

Based on origin natural dyes fall into three categories: plant, animal and mineral.

Plant dyes

The different parts of plants for example roots, nuts and flowers are sources of colouring pigments and dyes. Henna (orange-red) -from leave of henna plants, Fustic (yellow)-from the wood of the fustic tree, Indigo (C16H10N2O2(blue)-from leaves and stems of the indigo plant, Logwood (black)-from the core (heart) of the log wood tree, Turmeric (violet) - from the roots the turmeric plant and Saffron (yellow) -from stigmas of the common crocus are the common ones (korankye,2010). Plants are the major sources of natural colourants and almost all their parts such as stem, leave, fruits, seeds and pills are used for extracting natural colour and they have antimicrobial, antifungal, insect repellent, deodorant, disinfectant and other medicinal values. There were as many as 500 plant species identified as sources for dyes (Jihad, 2014)

Animal dyes

These colours are obtained from animals and insects. Red mouthed rock shell was one of the main sources of Tyrian purple. Phoenician purples and biblical blues are the most royal and sacred of all ancient dyeing were produced from Levantine Sea snails of the family Muricide. These molluscs may have been in use for the production of the royal purple pigment. Secretion of insects and dried insect bodies are the major source of natural dyes. For example, shell-fish provides the colouring matter, Lac and cochineal is obtained from insect carmine and it gives similar colours. It is gathered by spreading cloth on the ground under infested trees, during the season of red rain.

Mineral dyes

Some mineral pigments found in nature such as cinnabar, redocher, yellowocher, raw sienna, malachite, ultramarine blue, azurite, gypsum, talc, charcoal black, and so on, have been used for colouration purposes. Apart from the redocher that was used by the monks for colourationof their robes, these were mainly used in paintings and murals along with gum as binder (Adityaet al., 2021).

2.2.3 Classification based on Application

According to this classification, natural dyes can be classified as either direct dyes or mordant dyes (Agarwal et al., 2000). Direct dyes may be further subdivided into:

- Direct dyes for cotton, eg. turmeric, pomegranate, annatto, safflower, etc.
- Direct dyes for wool and silk, eg. turmeric, pomegranate, annatto, safflower, etc.
- Acid dyes, eg. saffron
- Basic dyes, eg. Berberine.

Direct dyes for cotton can be applied to all natural textile fibres; acid dyes can generally only be applied to wool and silk; basic dyes can be used for wool and silk as well as tannic acid treated cotton. On the other hand, mordant dyes are equally suiTable for both animal and vegeTable fibres. Important mordant dyes include madder, logwood and cochineal. There is a further class of dyes known as vat dyes, which are insoluble in water. These include indigo, woad and Tyrian purple (Patel,2011).

Vat dyes: Indigo is a water-insoluble dye, and before application it is solubilised in water. The solubilisation of natural indigo is done with the help of sodium hydrosulphite and sodium hydroxide. After solubilisation, it is applied on cellulosic fibre, and after dyeing the development of colour is done by oxidation with hydrogen peroxide. Indigo dye is the representative of indigoid class of vat dyes

Direct dyes: The natural dyes which are water soluble and have a long and planar molecular structure and presence of conjugated (single and double bonds) bonds can be applied by direct dyeing method. The dye molecules may contain amino, hydroxyl and sulphonic groups. Turmeric, Harda, pomegranate rind and annatto can be applied by direct dyeing method. Common salt is used to get better exhaustion of dyes. The dyeing temperature is kept at 100°C.

Acid dyes: The dye molecules possess sulphonic or carboxylic groups in their structure, which produce affinity for wool and silk fibre. The dyeing is done at acidic pH of 4.5–5.5. After dyeing the fastness improvement is done with tannic acid. The dyeing of wool and silk with saffron is done by acid dyeing method. The presence of common salt in dye bath produces levelling effect.

Basic dyes: The dye molecules produce coloured cation after dissolution in the water at acidic pH. The dye molecules contain –NH2 groups and react with –COOH groups of wool and silk. The dye bath pH is kept 4–5 by adding acetic acid (Gupta, 2019).

Classification based on colour

Natural dyes are frequently categorised on the basis of the colour that they impart to the fibre substrate.

Natural yellow dyes:

Yellow symbolises growth and happiness and is perhaps the most abundant hue in nature. The number of plants that yield yellow dyes is much higher than the number that yield other colours, and the Colour Index lists a total of 28 natural yellow dyes. Yellow is of particular significance in India, where it is still considered an auspicious colour with great religious significance (Patel,2011)

Natural red dyes

The Colour Index lists 32 red natural dyes. Most of the red colourants are found in the barks or in roots of the plant or camouflaged in the bodies of the dull grey insects. Few prominent members are madder (Rubia tinctorum), manjistha (Rubia tinctorum), Brazil wood/sappan wood (Caesalpinia sappan), Al or morinda (Morinda citrifolia), cochineal (Coccus cacti) and lac dye (Coccus lacca) (Yusaf et al., 2017; Vankar, 2000).

Natural blue dyes

The Colour Index lists only four blue natural dyes, viz. natural indigo, sulphonated natural indigo, Kumbh (Manipur) and the flowers of Japanese 'Tsuykusa' used mainly for making awobana paper (Sinha et al., 2012). The most brilliant and the fastest blue shades are obtained from indigo on all fibres. The principal colouring matter is indigotin, whose main sources are Indigo (Indigofera tinctoria) and woad (Isatis tinctoria) (Patel,2011). These dyes exhibit excellent fastness to wash on cotton Photo-stabilisation of gardenia blue may be induced using functional UV absorbers (Oda, 2012; Oda et al., 2013).

Natural black dyes

One important black natural dye is Logwood Haematoxylum campechianum) which is also known as Cam peachy wood because it was discovered by the Spaniards on the bay of Campeche in Mexico. It is still used today for dyeing silk in deep shades on an iron tannate mordant. It also gives excellent depth and fastness on most natural and synthetic fibres. Tannins are further important sources of black dyes. Pomegranate rind contains the hydrolysable tannic flavogallol, which combines with iron salts to give deep blacks

Natural brown dyes

The majority of natural brown dyes are obtained from quinone-based dyes, naphthoquinones and anthraquinones. Generally, copper and iron salts are used as mordants and they tend to turn the colour to dull and deep shades, particularly browns. There is one other natural dye, cutch, which produces rich brown shades with copper and chromium salts. This dye is a tannin-based flavonoid, and is derived from Acacia catechu and wild Acacia arabica.

Green dyes

Plants that yield green dyes are rare. Both woad (Isatistinctoria) and indigo have been used since ancient times in combination with yellow dyes to produce green shades. Woollen cloth mordanted with alum and dyed yellow with dyer's green weed was over dyed with woad and, later with indigo, to produce the once-famous Kendal green. Soft olive greens are also obtained when textiles dyed yellow are treated with iron mordant (Mansour, 2018).

Orange dyes

Orange Dyes that create reds and yellows can also yield oranges. The sources for a natural orange dye are barberry, annatto, sweet pepper blood roots etc (Tayade et al., 2013; Yusaf et al., 2017).

2.2.4 Classification based on Chemical constitution

Natural organic dyes and pigments belong to a wide range of chemical classes, such as indigoid, anthraquinonoids, naphthoquinones, polymethines, ketones, imines, quinones, flavones, flavanols, flavanones and chlorophyll (Rungruangkitkrai and Mongkholrattanasit, 2012)

2.2.5 Advantages of natural dyes

- They have few side effects and a high safety factor.
- They are biodegradable and environmentally friendly.
- Some natural dyes have certain therapeutic and health effects also.
- Natural dyes are generally extracted from minerals, plants, and animals, as well as natural pigments.
- The tone is harmonious and natural, with a soft and beautiful feeling.
- Effluents of natural dye processing are biodegradable.
- Many natural dyes have the potential to absorb a significant amount of ultraviolet lightNatural dye poses functional properties, such as anti-bacterial, antifungal, and antimicrobial properties.
- Since some natural dyes have antioxidants in their structure, they enhance antioxidant properties of the treated materials.
- Some of the natural dyes have inherent insect repellent properties (Chattopadhyayet al., 2013).

2.2.6 Disadvantages of Natural dyes

- They are sensitive to metal ions and pH, not resistant to light, and have poor heat stability.
- Affected by the change of natural conditions due to their complex physical and chemical structure.
- The extraction process is complex and expensive.
- Limited sources for extraction.

- Mordant is needed to improve fastness which is sometimes not ecological.
- Reproducibility is a major concern.
- The availability of natural dyes is seasonal (Bechtold et al., 2003).

2.3 Natural dye extraction methods

Natural dyes have plant and animal constituents such as water-insoluble fibres, carbohydrates, protein, chlorophyll, and tannins, among others, extraction is an essential step for preparing purified natural dyes. Extraction of natural dyes is a complex process. There aredifferent methods for extraction of colouring materials are:

- Aqueous extraction
- Alkali or acid extraction
- Microwave and ultrasonic assisted extraction
- Fermentation
- Enzymatic extraction
- Solvent extraction
- Super critical fluid extraction

2.3.1Aqueous Extraction

Aqueous extraction was traditionally used to extract dyes from plants and other materials. In this method, the dye-containing material is first broken into small pieces or powdered and seived to improve extraction efficiency. It is then soaked with water in earthen, wooden, or metal vessels (preferably copper or stainless steel) for a long time usually overnight to loosen the cell structure and then boiled to get the dye solution. For getting effective dye solution in water requires different temperatures and time changes. As most of the dyeing operations are carried out in aqueous media, the extract obtained by this method can be easily applied to the textile materials (Saxena et al., 2014).

2.3.2 Microwave and ultrasonic assisted extraction

Actually microwave- and ultrasound-assisted extraction processes where extraction efficiency is increased by the use of ultrasound or microwaves, thus reducing the quantity of required solvent, time, and temperature of extraction. When the natural dye containing plant materials is treated with water or any other solvent in the presence of ultrasound, very small bubbles or cavitations are formed in the liquid. These increase in size but upon reaching a certain size, they cannot retain their shape. When this happens, the cavity collapses or the bubbles burst creating high temperature and pressure. Millions of these bubbles form and collapse every second. The creation of very high temperature and pressure during extraction increases the extraction efficiency within a short time. Also the process can be performed at lower temperature and therefore extraction of heat-sensitive dye molecules is better. As exploration of new dye sources and attempts to optimize the dye extraction process is continuing, use of this extraction technique has been recently reported by many researchers.

In microwave extraction, the natural sources are treated with a minimum amount of solvent in the presence of microwave energy sources. Microwave increases the rate of the processes so the extraction can be completed in a shorter time with better yield. Sinha et al. have reported extraction of annatto colourant with microwave energy. Earlier their group had reported microwave-assisted extraction of blue pigment from the butterfly pea. Microwave and ultrasound extractions can be considered as green processes due to reduction of extraction temperature, solvent usage, and time which results in lower consumption of energy (Mansour, 2018.

2.3.3 Alkali or Acid extraction

Extraction of dye is done under acidic or alkaline medium. For this dilute acidic or alkaline can also be used which helps in hydrolysis of glycosides resulting in better extraction. Acidic method is used in extraction of tesu natural dye from tesu flower. Alkaline medium is used for those colour extraction which contain phenolic groups. Extraction of colour from lac insect, annatto seeds, safflower petals are extracted by this method (Chungkarg et al., 2021).

2.3.4 Enzymatic and fermentation extraction

Recent developments in biotechnology for the extraction of effective components from natural plants are becoming popular. There is a selection of appropriate enzymes that can decompose plant tissues mildly, accelerate the release of effective components, and improve the extraction rate. For example, cellulase can degrade cellulose, hemicellulose, and other substances, and can cause localized loose and swelling changes of the cell wall and cytoplasmic structure, thus increasing the diffusion of effective components in the cell to the extraction medium and promoting the efficiency of pigment extraction. Temperature and pH are the main factors affecting the effect of the enzyme. The enzyme extraction method has the advantages of milder extraction conditions and sTable physical and chemical properties of active components. The structure of Geniposide in natural gardenia yellow pigment can be changed by an enzymatic reaction to produce gardenia red and blue pigment. The extraction of anthocyanins by the enzymatic method is about 72% higher than that by the solvent method. This method is suiTable for the dyes extracted from hard plant materials like the bark and roots (Saxena and Raja, 2014)

2.3.5 Solvent extraction

Natural colouring matters depending upon their nature can also be extracted by using organic solvents such as acetone, petroleum ether, chloroform, ethanol, methanol, or a mixture of solvents such as mixture of ethanol and methanol, mixture of water with alcohol, and so on. The water/alcohol extraction method is able to extract both water-soluble and water-insoluble substances from the plant resources. The extraction yield is thus higher as compared to the aqueous method as larger number of chemicals and colouring materials can be extracted. Acid or alkali can also be added to alcoholic solvents to facilitate hydrolysis of glycosides and release of colouring matter. Purification of extracted colour is easier as solvents can be easily removed by distillation and reused. Extraction is performed at a lower temperature thus chances of degradation are fewer. The disadvantages of the method are the presence of toxic residual solvents and their greenhouse effect. Another disadvantage of this method is that the extracted material is not readily soluble in water and the subsequent dyeing process has to be carried out in an aqueous medium (Mansour, 2018).

2.3.6 Super critical extraction

Supercritical fluid is the most complex process but has the advantages of both liquids and gasses, high density and viscosity, lower surface tension and higher solubility, which enhance rapidly with the increase in pressure. It can penetrate the matrix of extraction materials and be a very effective extraction mechanism (Prabhu and Bhute, 2012). Extraction separation and solvent removal are combined into one unit that identifies the process flow and improves the production efficiency. The extraction diagram is depicted in Figure 5. In addition, it also has -a few advantages, such as fast extraction speed, good selectivity, extraction and segregation can be carried out at room temperature or a low temperature. There is no residual solvent pollution, no environmental pollution, e.g., some natural products are sensitive to heat emitted during the process, or the chemically unsTable components are easily destroyed, which can preserve the original flavour and nutritional components of natural products (Haiyee et al., 2016).

2.4 *Rubia tinctorum*

2.4.1 Introduction

Rubia tinctorum L., also known as madder, has a long history of use as a red dye (Bechtold and Mussak, 2009; Blackburn, 2017; Schweppe, 1993). The genus Rubia belongs to the family Rubiaceae and comprises about 70 species (Eltamany et al., 2020). The herbaceous plant madder consists of small leaves and grows by clinging into each other like bedstraw. The plant is cultivated for the extensive long red coloured roots, which are harvested in the second or third year of growth (Derksen and van Beek, 2002). The earliest evidence to date of the use of madder lake is in a 4000 years old ancient Egyptian quiver (Leona, 2009).

Madder extract is a complex mixture of compounds like anthraquinones (e.g., alizarin 7, the main dyeing compound), sugars (e.g., sucrose), enzymes (e.g., primeverosidase), carboxylic acids, coumarins, flavonoids, iridoids and others (Blackburn, 2017; Derksen and van Beek, 2002; Duval et al., 2016; Eichinger et al., 1999).(Plate 1)

2.4.2 Botanical description

Family: Rubiaceae

Genus: Rubia

Species: tinctorum

Scientific name: ***Rubia tinctorum***

Common names: the rose madder or common madder or dyer's madder

2.4.3 Properties and Medicinal uses

Food: Madder is a food plant for the larvae of some Lepidoptera species.

Fodder: Madder can be used as fodder for animal

Dyestuff: Madder root contains the anthraquinone pigment alizarin and has been since ancient times a popular fine red dye plant. The long fleshy root, when dried and milled, yields a variety of brilliant colours: red, pink, brown, orange, black, lilac, and purple, depending upon the mordant used, (Recep, 2014).

Medicine: Madder root is useful for treating urinary gravel, bladder and urinary tube cleaning, dropsy, amenorrhea (weak or no bleeding) and jaundice. Madder is only good to treat kidney stones the size of grain or sand. As a dye, Madder root colours milk, urine and bones, so that experiments in the growth of bones can be conducted with its help, (De Santis and Moresi, 2007).

2.5 *Caesalpinia sappan*

2.5.1 Introduction

The plant *Caesalpinia sappan* is a small leguminous tree upto 10m in height belonging to the Caesalpiniaceae family,with an orange-red hard Heartwood with spiny trunk ,which besides being useful in turnery gives a red dye. It is commonly known as Brazil or Sappan wood, native to tropical Asia, also grown as a hedge plant. The plant is being used worldwide for a large number of traditional medicinal purposes including anti-tumour, antiinflammatory, immunosuppressive, anti-diabetic, anti-allergic, cardioactive, neurotoxicity, anti-bacterial, anti- acne, anemia, tuberculosis and some other activities. The main active constituent is Brazilin, and together triterpenoids, flavanoids, lipids, steroids, aminoacids etc are found. Leaves yield volatile oil, 0.16 to 0.25%; pods contain 40% tannin; seeds yield 32.1% and 34.4% mucilage and straw – yellow , edible oil(7.5%) having a characteristic smell; roots contains caesalpin-type diterpenoids along with sitosterol.

In folklore medicine it is used as a herbal drinking water for its blood purifying, anti- thirst , anti-tumour and to improve complexion in Kerela. It has the potential to hit the market as a safe natural colouring agent with good medicinal value for pharmaceuticals, food products and beverages. The plant is one of the ingredients of an indigenous drug 'Lukol'which is administered orally for the treatment of non-specific leucorrhoe, (Kusumawati, 2019).(Plate 2)

2.5.2Botanical description

- **Family:** Caesalpiniaceae/Fabaceae
- **Genus:** Caesalpinia
- **Species**: sappan
- **Scientific name:** *Caesalpinia sappan*
- **Common names:** commonly known as Brazil or Sappan wood, Pathimugam in tamil.

2.5.3 Properties and Medicinal uses

Heartwood is traditionally used to treat bleeding gums, anemia, diabetes, cardiac problem and blood stasis and as a post-partum tonic to reduce uterine bleeding; it is also known for its antidiarrheal, sedative, and diuretic properties (Badami et al., 2004; Mekala and Radha, 2015;)

The wood is used as a blood purifier. The small core of heartwood producing a dark red solution in water is being used in herbal drinking water in Kerala, since time immemorial owing to its anti-thirst, blood purifying, anti-diabetic, complexion enhancer and several other properties. It is used as infusion or decoction in diarrhoea, dysentery etc. Also employed in some forms of skin diseases especially lichen. The wood is a component of Vicco Vajradanti, a famous tooth paste and tooth powder of India. The wood is used in carpentry. The timber, which has straight grains, is of great value under the name of Pernambuco for making violin bows (Li et al., 2020).

2.6 Mordant

Mordant comes from the Latin word “mordere”, meaning “to bite”. Mordant is a chemical which can itself be fixed on the fibre and also forms a chemical bond with the natural colourants. It helps in absorption and fixation of natural dyes and also prevents bleeding and fading of colours i.e., improves the fastness properties of the dyed fabrics. (Prabhu et al., 2012). Mordants are metal salts which produce an affinity between the fabric and the dye (Vankar et al., 2009; Samanta et al., 2009).

2.6.1 Types

Mordants are basically three types -metallic mordants such as metal salts of aluminum, copper, iron etc., (ii) tannic mordant such as tannic acid, e.g., myrobalan and sumach and (iii) oil mordant which forms complex with main metal mordant. Myrobalan and sumach are most important mordants of tannins and Oils or oil-mordants (Gulrajani, 1993).

Generally, on the basis of origin mordants are two types- Synthetic and Naturalmordants. Synthetic mordants are obtained from acid dyes, they are called as acid chrome dyes.Natural mordants obtained from natural sources plants and fruits. Cow dung, aloe vera, lemonpeel, pomegranate rind used as natural mordants (Kareem and Omar, 2012).

2.6.2 Application of mordants

The three methods used for mordanting are:

- **Pre-mordanting:** The substrate is treated with the mordant and then the dye. Thecomplex between the mordant and dye is formed on the fibre.
- **Meta/simultaneous-mordanting:** The mordant is added in the dye bath itself. The process issimpler than pre- or post-mordanting, but is applicable to only a few dyes.
- **Post-mordanting:** The dyed material is treated with a mordant. The

complexbetween the mordant and dye is formed on the fibre.

Alum

Potassium aluminum sulfate is the mordant most frequently used by dyers for protein (animal) and cellulose (plant) fibres and fabrics. It improves light and washfastness of all natural dyes and keeps colours clear. It is inexpensive and safe to use (see our safety notes). This form of alum is refined from bauxite, the raw state of aluminum ore, and is free from the impurities (such as iron) some other alums can contain, (Mozaffari and Maleki, 2018).

Iron water

Iron water for natural dyeing has been a game changer for me since it has allowed me to create a variety of shades and tones from one single plant dye. Iron is very commonly used as a mordant which means that you must apply to the fabric before its dyed.When you add iron water to a dye bath you will immediately see a change in colour. You can transition pale and bright colours into darker ones. Golds and yellows can turn into greys and blacks, pinks can turn into purples. It's a fantastic way to expand your colour palette and to discover nature's amazing variety of greys. Note that no natural dye will produce a grey colour by itself. (https://lacreativemama.com/how-to-make-iron-water-natural-dyeing/)(Plate-3)

2.6.3 Properties of mordants

- Mordant dyes have no affinity for textile fibres.
- They are attached to the fibres with the help of mordants.
- Mordant have affinity both for the dye and fibre.
- Mordant dyes are capable of combining with metallic oxides to form insoluble colour lakes.
- Mordant dyes may be natural or synthetic.
- Mordant dyes are mostly applied on natural protein fibres, nylon and acrylic fibres.
- Good light fastness rating about 4-5.
- Most mordant dye are soluble in cold water.
- Wide range of hues can be produced from mordant dyes (http://textileengg.blogspot.com/2015)

Plate 1- *Rubia tinctorum*

Leaves and Flower **Heartwood** **Spinny Trunk**

Plate 2- *Caesalpinia sappan*

Alum Water **Iron Water**

Plate 3- Mordants

METHODOLOGY

III. METHODOLOGY

Experimental procedure pertaining to the study on "***Developing tints and shades in cotton fabric using Rubia tinctorum root and Caesalpinia sappan wood extracts***" is discussed under the following headings:

3.1. Selection of fabric

3.2. Pre-treatment of fabric

3.2.1 Desizing

3.3. Selection of dye source

3.4. Extraction of dye source

3.5. Selection of mordant

3.6. Extraction of mordant Iron water and alum

3.7 Optimization of dye extraction conditions

3.7.1 Solvent

3.7.2 Dye source concentration

3.7.3 Time

3.7.4 Temperature

3.8 Optimization of dyeing conditions

3.8.1 Time

3.8.2 Temperature

3.8.3 Material liquor ratio

3.8.4 Dye Concentration

3.9. Dyeing

3.10. Methods of mordanting

3.10.1 Pre-mordanting

3.10.2 Post-mordanting

3.10.3. Simultaneous mordanting

3.11. Dyeing under optimized conditions

3.12. Phytochemical analysis

3.13. FT-IR

3.14. UV- VIS analysis

3.15. Evaluation of dyed fabric

3.15.1 Fabric weight

3.15.2 Fabric thickness

3.15.3 Fabric stiffness

3.15.4 Colour fastness

3.15.4.1 Fastness to washing

3.15.4.2 Fastness to crocking

3.15.4.3 Fastness to pressing

3.15.5 Colour strength and Colour coordinates

3.16. Absorbency and Wettability

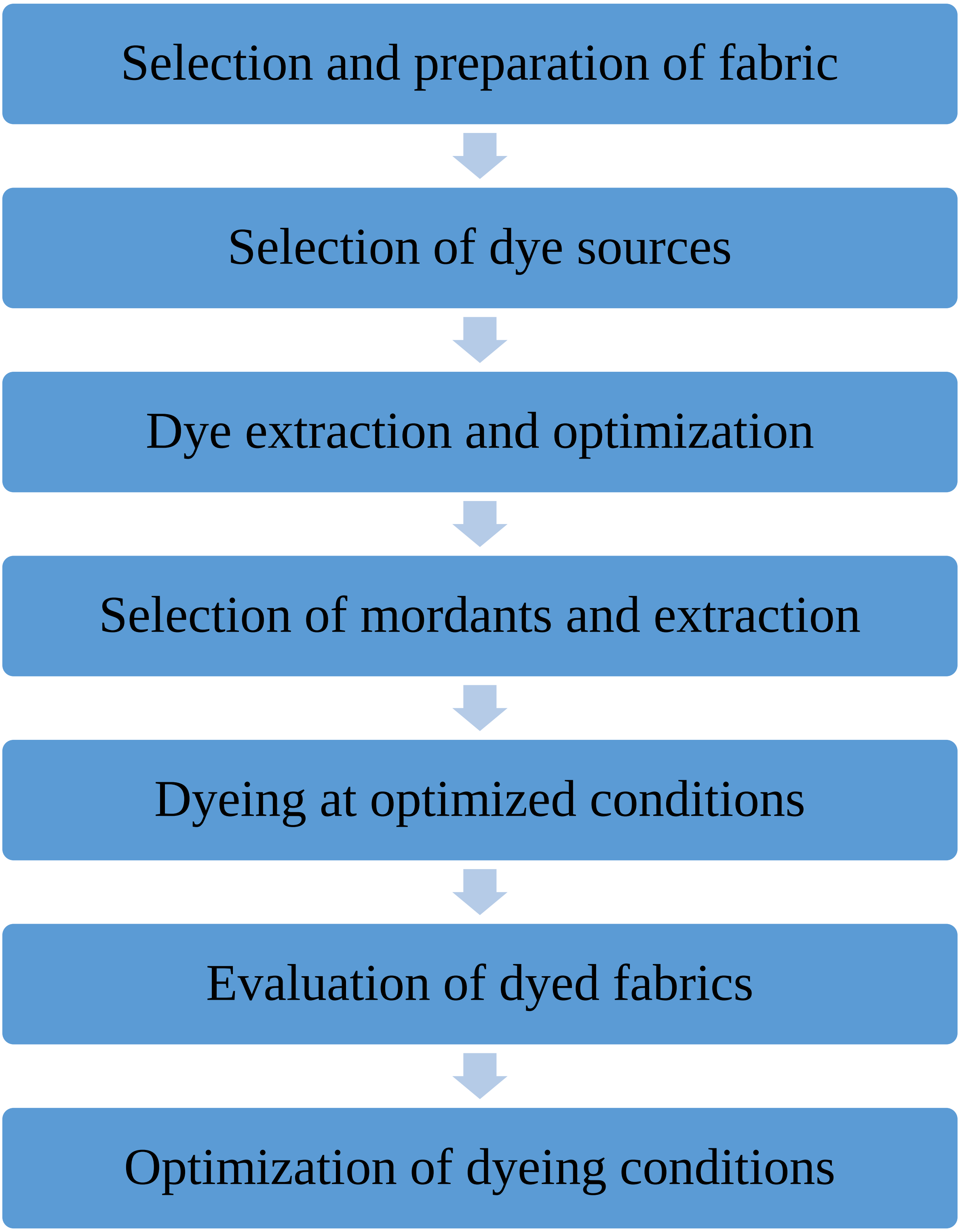
3.16.1 Sinking

3.16.2 Wicking

3.16.3 Spray test

3.17. Nomenclature of the samples

Flowchart depicting the methodology



3.1 Selection of fabric

Cotton is the most important textile fiber because of its adaptability, visibility, performance and other properties, especially natural comfort. Cotton fibers are used in everything from baby wears to space suits for astronauts. Hence cotton fabric was selected for study and was sourced from local manufacturer of cotton fabrics, Salem. Fabric details are given in Annexure I.

3.2 Fabric pre-treatment

The fabric preparation process is essential. By consistently eliminating all impurities, it enhances the aesthetic appearance and processability of fibre to fabric and prepares the fabric for dyeing and finishing procedures. (Vigo,1994)

3.2.1. Desizing

Desizing is an essential step in the cotton manufacturing process. Sizes are merely the starch that must be removed before colouring is applied to woven fabrics made of warp yarns in order to help them resist the weaving process. The process of "desizing" involves taking the starch out of the fabric. Sizing material must be removed from cotton warp yarns and fabric. The size of these strands is designed to prevent them from breaking under the pressure and tension of weaving. The fabric contains both natural and synthetic fibres, and other foreign materials are eliminated during the process because starch prevents dye from penetrating fibre. To remove starch from the fabric, a variety of processes are utilised. To remove the sizing material from the chosen cloth, it was boiled at 100°C for one hour. (Wadje, 2009). Selected cotton fabric was desized using the recipe given below. (Plate -4)

Recipe:

ML Ratio: 1:30

Soap nut extract -10%

Extract of ash water – 10%

Temperature – 100° C

Time – 1hr

3.3 Selection of dye source

3.3.1 Madder roots (*Rubia tinctorum)*

The dye source selected for the study was *Rubia tinctorum* root, which was commonly known as Indian Madder, belonging to plant species family of Rubiaceae.. This tree has wide range of properties including 'medicinal' and each part of the tree has used to cure health issues

(Kumaresan et al., 2018; Varghese et al.,2018). For the present study the dried roots were taken as natural dye source. The roots were bought from the local herbal medicinal shops near Saibaba colony, Coimbatore, Tamil Nadu.

3.3.2 Sappan wood / brazilwood (*Caesalpinia sappan*)

The dye source selected for the study was Caesalpinia sappan heartwood, which is commonly known as sappan wood or brazilwood, belonging to the tree species family Fabaceae. In Tamil it is called "Pathimugam".. The wood was traditionally used to treat tuberculosis, diarrhoea, dysentery, skin infection and anaemia among people (Badami et al.,2003). For the present study heartwood was taken as natural dye source. It was commercially purchased in the nearby herbal medicine store, Saibaba colony, Coimbatore, Tamil Nadu.

3.4 Extraction of dye source

For the extraction of dye from the collected Rubia tinctorum root and Caesalpinia sappan heartwood, known quantity of dried source (5%) were taken and added to distilled water and heated in water bath for 30min at 100^{o} C. After cooling the extract was filtered and used for dyeing as suggested by Kannathasan &Kokila, 2021. (Plate-5)

3.5 Selection of mordant

Mordants are metal salts which produce an affinity between the fabric and dye. For the present study two mordants such as alum & iron water (metal mordant) were used.

3.6 Extraction of mordant

The extraction process was carried out using aqueous method.

- Alum is dissolved in 100ml of water in the 5% concentration.
- Iron water was prepared following ancient fermentation method. 1kg of rust iron particles were added in 10litres of water with 1kg of jaggery and stirred well and kept closed in an air tight container for about 15 days. It should be stirred occasionally. (C.E. Pellew,1998, Dyes And Dyeing)

Plate- 4 Desizing

Plate – 5 Madder Root and Sappan Wood Dye Extraction

Plate – 6 Pre-Mordanting with Iron Water and Alum

Plate – 7 Dyeing of Pre Mordanted-Fabrics with Madder and Sappan Dye

3.7 Optimization of dye extraction conditions

3.7.1 Solvent

Dye was extracted with 5% dye source concentration at 100°C for 30min individually in water. The dye extracts were used to dye the fabric separately. The dyed fabric was assessed for colour strength**.**

3.7.2 Dye source concentration

To determine the optimum dye source concentration, different dye source concentrations were used for dye extraction such as 3%, 5% and 7 %. The concentration at which the colour yield was maximum was selected as optimum concentration individually for dye extraction from ***Rubia tinctorum*** **root and Caesalpinia sappan heartwood** based on the colour strength**.**

3.7.3 Time

To find out the optimum dye extraction time, the extraction of dye was done at different time intervals such as 15 mins, 30mins and 45mins. The extracted dye was used to dye the fabric and the colour strength was assessed. Based on the colour strength extraction time was fixed for further experiments.

3.7.4 Temperature

To analyse the optimum temperature for dye extraction, dye extraction was carried out at different temperature such as 60°C ,80°C and 100ºC. The extracted dye was used to dye the fabric and the colour strength was assessed. Based on the colour strength extraction temperature was fixed for further experiments.

3.8 Optimization of dyeing conditions

3.8.1. Time

To obtain optimum dyeing time, the dyeing was carried out at different intervals of time such as 15 mins, 30mins and 45mins. With the help of Colour lab Spectrophotometer, the colour strength of the dyed fabric was analysed. The time which showed maximum colour strength was selected as optimum dyeing time.

3.8.2 Temperature

To analyse the optimum temperature, dyeing was done at different temperatures such as 60°C,80°C and 100°C. Colour lab spectrophotometer was used to measure the colour strength of the dyed fabrics were analysed. The temperature which showed maximum colour strength was selected as optimum dyeing temperature.

3.8.3 Material liquor ratio

To analyse the material liquor ratio for dyeing, dye extractions ratio was taken at different intervals like 1:10, 1:20, 1:30, 1:40 and 1:50. With the help of colour lab spectrophotometer the colour strength of the dyed fabrics was analysed. The M.L. Ratio which showed maximum colour strength was selected as optimum dyeing material liquor ratio.

3.8.4 Dye concentration

To analyse the optimum dye concentration ratio for dyeing, different dye concentrations were used for dyeing such as 100,75:25,50:50 and 25:75. The concentration at which the colour yield was maximum was selected as optimum concentration for dye extraction from *Rubia tinctorum* root and *Caesalpinia sappan* heartwood based on the colour strength.

3.9. Dyeing

Dyeing was carried out using dye extracted from *Rubia tinctorum* root and *Caesalpinia sappan* heartwood at optimized conditions such as time, temperature, M.L.Ratio and dye concentration. The dyed fabric was assessed for colour coordinates and colour strength using premier Colour lab spectrophotometer**.**

3.10. Methods of mordanting

Mordants are commonly used for dyeing process using natural dyes. Mordant is a substance that binds the dye to fabric fibre. It helps to make colour resistance to washing and sunlight.

Mordanting process was done in three ways pre, post and simultaneous mordanting.

Preparation of mordant solution

- Alum is dissolved in 100ml of water in the 5% concentration.
- Iron water was prepared following ancient fermentation method. 1kg of rust iron particles were added in 10litres of water with 1kg of jaggery and stirred well and kept closed in an air tight container for about 15 days. It should be stirred occasionally.

3.10.1 Pre-mordanting

Pre mordanting was done before dyeing the fabric. Cotton fabric was first treated with mordant solution (alum and iron water) with M.L ratio 1:30 for 30mins at temperature 80°C. After pre mordanting, the samples were dyed with ***Rubia tinctorum*** root and ***Caesalpinia sappan*** heartwood extracts separately. (Plate- 6)

3.10.2 Post mordanting

Post mordanting was performed after dyeing. The selected fabric was dyed with extracted dye from ***Rubia tinctorum*** root and ***Caesalpinia sappan*** heartwood separately. After dyeing the fabrics were removed from respective dye bath , then the samples were treated with selected mordant (alum and iron water) with M.L ratio of 1:30 for 30 min at 80°C separately.

3.10.3 Simultaneous mordanting

Mordanting method, both dyeing and mordanting is performed at same time. The selected fabric was dyed with extracted dye from ***Rubia tinctorum*** root and ***Caesalpinia sappan*** heartwood along with the mordants (alum and iron water simultaneously) with M.L ratio 1:30 for 30mins at temperature 80°C separately**.**

3.11. Dyeing under optimized conditions

Dyeing was done using optimized extraction and dyeing conditions such as solvent, dye concentration, time, temperature and M.L.Ratio. The dyed sample was assessed for colour strength and colour coordinates using Colour lab spectrophotometer**.** (Plate-7)

3.12. Phytochemical analyis

Phytochemicals occur naturally in plants,that possess defense mechanisms, they are used as defenses against various diseases. Important phytochemical groups such as alkaloids, steroids, flavonoids, phenolic compounds, anthraquinones and tannins found in various plant extracts which are responsible for their unique medicinal properties. (Chandrashekar et al., 2012; Anand et al., 2011; Kalaiarasan and John, 2010). Phytochemical analysis is done to analyze the chemical composition present in plant dyes using precipitation and staining reactions and classify them into different chemical groups (Tiwari and Srivatsava, 2019). The extracted pigments were tested using standard methods to identify components present in *Rubia tinctorum* root and *Caesalpinia sappan* heartwood extracts.

Twenty ML of extracts in separate glass containers were submitted to Bharat Ratna Prof. C.N.R.RAO Research Centre, Avinashilingam Institute of Home Science and Higher Education for Women, Coimbatore-43, for carrying out Phytochemical analysis.

3.13 FT-IR

Infrared spectroscopy is the analysis of infrared light interacting with a molecule. This can be analysed in three ways by measuring absorption, emission and reflection. The main use of this technique is in organic and inorganic chemistry. It is used by chemists to determine functional groups in molecules (Chahande et al., 2019). Therefore to analyse the functional groups present in the extracts of *Rubia tinctorum root* and C*aesalpinia sappan* heartwood FT-IR test was carried out in Bharat Ratna Prof. C.N.R.RAO Research Centre, Avinashilingam Institute of Home Science and Higher Education for Women, Coimbatore-43, for carrying out FT-IR analysis. (Plate – 8)

3.14 UV- VIS Analysis

Ultraviolet-Visible (UV-vis) Spectroscopy is a popular, versatile technique used to detect molecules in a compound. The UV light is passed through the sample under study and transmitted of light is measured. At a particular wave length, the amount of absorbance is a factor of the chemical structure of the molecules present in the sample (Amutha et al., 2020). For the present investigation the dyed samples were submitted for UV-vis spectroscopy analysis at Bharat Ratna Prof. C.N.R.RAO Research Centre, Avinashilingam Institute of Home Science and Higher Education for Women, Coimbatore-43, for carrying out UV- VIS analysis. (Plate - 9)

3.15 Evaluation of dyed fabric

3.15.1 Fabric strength

Tensile strength is the force required to break a fabric when it is under tension resistance of the fabric to a tensile load or stress in either the warp or filling direction. Elongation is the extent to which the fabric under tension extends, till it cut off. Ten percent strength loss and per cent change in elongation at break were determined by the tensile strength, according to standard procedures. Eureka Brand Pendulum Tensile Strength Tester **plate()** was used to determine the breaking strength and elongation of the increased samples processed. The capacity of the machine and the rate of transverse curve 90 kg and 40 cm per minute respectively. The sample was cut to a width of and 12 inches length. The yarns raveled from both the edges until the width measured to under the optimum conditions, the sample were mounted centrally, gripped along the full width prevent slippage of the sample. The pendulum of the tester was set vertically and the pointer at zero on the scale. When the load was applied to the sample it ruptured the sample, mechanism was stopped and the dial reading was recorded in kilogram for breaking strength and elongation in ems were noted.

Five readings of warp and weft specimen were recorded. The same procedure was followed for UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, and IDF_2 samples of warp and weft readings were noted and average was calculated and recorded.

3.15.2 Fabric weight (GSM) (ASTM D 2646/D3776, IS0 3801)

Weight of fabric was expressed as weight of particular specimen in piece of gram/ square meter/ ounces / square yards. GSM cutter is used to cut the sample. The sample cutter cuts out quick and exactly circular sample of 100cm² which is exactly one hundredth of a square meter. Cut sample should be weighed and noted. The weight of the fabric is measured using electronic balance (plate10**)**. The value in grams are multiplied by 100, gives the GSM directly. For lock position: The blades are held inside the knobs. For unlock position: The blades are allowed for cutting and knobs are opened to cut the fabric.

The same procedure was followed for all the UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, and IDF_2 samples. All the samples were weighed five times and the mean value was calculated and recorded.

Grams per Square Meter = Specimen weight in grams * 100

3.15.3 Fabric thickness (ASTM D1777, ISO 1765)

Thickness of a fabric is defined as perpendicular distance through the fabric, which determines the dimension between the upper and lower side of the fabric. Three loads of different weights are given by the ASTM standards to use on fabric. It is given in kappa. In practice thickness measurement are rarely used as they are very sensitive to the pressure used in the measurement. Fabric thickness gauze is used to measure the thickness of the sample (textileall.blogspot.com, 2016). The specimen should be free from wrinkles, creases and folds and it is placed between anvil and pressure foot by uplifting pressure foot and leaving down 37 the pressure foot slowly on the specimen. The dial indicates the thickness of the sample and the readings were noted from dial gauze. (Plate-11)

Five readings were taken from different places of sample and the same procedure was followed for all the UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, and IDF_2 samples and the mean was calculated. It is expressed in mm.

3.15.4 Fabric stiffness (IS 6490:1971)

Stiffness is nothing but the ability to resist the bending of fabric and used to measure the resistance of bending property by external forces on fabric (textileadvisor.com 2020). The bending rigidity and fabric handling parameters are mostly judged by using stiffness test. Stiffness of a fabric is determined by using Shirley stiffness tester (Plate-12). The tester consists of flat surface platform supported with two side pieces engraved with index lines. The operator can view both index lines from convenient position with the help of mirror which is associated with instrument. With the help of scale, the bending length was measured in cm. The specimen was placed on the platform with template on it, so that the edges are coincide at zero on leading edge of specimen. The specimen was slowly pushed and the horizontal scale was moved slowly until the leading edged of specimen and template project beyond edge of platform. The sliding of specimen was stopped when it cuts both index lines. The five readings of warp and weft specimen were recorded.

The same procedure was followed for UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, and IDF_2 samples of warp and weft bending readings was noted. Five readings from weft and warp were taken and the average was calculated and recorded

3.15.5 Colour fastness

Colour fastness is nothing but the resistance in a material for changing its colour or transferring colour to adjacent materials or staining. Colour change can be seen in fading, colour changes or lightens and bleeding which means loss of colour or transferring colour to accompanied fibre material (Gersak et al, 2013). The colour fastness of dyed fabric was evaluated for washing, sunlight, wet and dry rubbing fastness according to standard test methods.

3.15.5.1 Fastness to Washing

Colour fastness to washing is defined as the degree of change in colour and staining after performing process. Following standards were used for washing fastness test (ISO 105 C06:2010, ISO 105 C08: 2010 and ISO 105 C09: 200). The specimen is stitched four sides of edges with reference fabrics. The specimen was washed using suiTable detergent solution at 40°C for 30 min. Then the specimen was rinsed and dried properly (Gersak et al, 2013). Colour change is assessed using grey scale.

The MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, and IDF_2 were tested for washing using five specimens five times. The level of colour change in dyed and mordant fabrics was compared and assed by using grey scale.

3.15.5.2 Fastness to Crocking

Fastness to crocking is nothing but the transfer of colour from dyed textile material to undyed textile material by means of rubbing. Crock meter equipment is used to test the fastness property of dyed fabrics (ISO 105X12:2001). The test is performed in two ways – using dry specimen and wet specimen against dyed fabric. A white fabric used to place on the top of dyed fabric and rubbed against on it in back and forth as straight line for about 10 cycles with downward movement with the help of handler. After testing the tested rubbing specimens were assessed using grey scale according to AATCC. Colour fastness to crocking is assessed in between 1- 5 level. The higher the number indicates the good colour fastness (Gersak et al, 2013., orinetbag.net 2016). (Plate-13)

The same procedure was performed for MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, AND IDF_2 specimen and the amount of colour transferred to the white fabric is assessed using AATCC grey scale

3.15.5.3 Fastness to Pressing

Fastness to pressing is defined as the level of change in colour and staining of colour on adjacent fabric after using pressing (ISO 105X11:1994). Pressing is done in two ways dry pressing and wet pressing. For dry pressing dry specimen is used and for wet pressing wet specimen and wet cotton adjacent fabric is used. All the samples are pressed using specified temperature and time with heating device. Colour change in specimen is assessed by using grey scale (in accordance with ISO 105-A02: 1993) (Gersak et al, 2013).

The MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, AND IDF_2 samples were tested and the amount of colour transferred to cotton cloth was assessed using grey scale.

3.15.6 Colour strength and Colour coordinates

The reflectance of dyed fabric appearance is determined via instrumentation procedure to find colour existence in fabrics. During dyeing procedure, it is hard to find the effect of colour. Colour Spectrophotometer Test (MS ISO 105- J03:2009) analysis was performed to specify the colour coordinates and colour difference presented in dye. Colour strength(K/S) is nothing but measuring the ability of a dye to impart colour to textile materials. Sample Colour strength was expressed in terms of the ratio of absorption (K) and scattering(S) coefficient (K/S) value using spectrophotometer. Commission Internationale de1; Eclairage (CIE) is indicates the difference between two colour (magnitude and direction) on the samples, it is expressed colour as three values L* for lightness from black to white, a* from green to red, and b* from blue to yellow. The fabric dyed without mordant and fabric dyed with mordant are compared and assessed using colour strength and colour coordinates values (Ayele et al., 2020).

3.16 Absorbency and wettability

3.16.1 Sinking

Sinking is nothing but something is falling down into liquid or on the surface of liquid.

Sinking test is done to find the wet ability of fabric. If samples took more than 1 min for sinking then those are marked as floated behaviour textiles (Saville, 1999). A 50 ml of distilled water is taken in transparent beaker. 25mmX25mm size samples were dropped on the surface of the water from certain standard height. Sinking time of sample is measured using stopwatch. Stopwatch was started when the fabric struck the surface of water and it was stopped when the fabric corners sank below the water surface.

The sample sink time was recorded. The UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, AND IDF_2 specimen in the size of 2.5cm*2.5cm were taken. The sample was dropped individually at certain height into 50ml of distilled water in glass beaker and the sinking time of sample was recorded using stopwatch. The same procedure was repeated for all the samples five times. The results were noted and mean value was calculated. (Plate 14)

3.16.2 Wicking

Wicking is spreading of liquid into fibre or fabric due to external forces or by capillary forces (Kissa,1996) or the spreading of liquid through the pores of free spaces of fabric is called capillary action or wicking (Morent et al., 2006). The sample was cut into the dimension of 2.5cmX15cm long. Required amount of distilled water is taken in transparent beaker then the samples were marked with ink discharged pen at 1cm from bottom edge of sample. The samples were hanged on a clamp and submerged in water. Stopwatch is used to record the readings. On the sample how far the water has moved upward with capillary action of fabric and crossed or reached marked line time was considered as wicking ability of fabric (Mahmud et al., 2016). The higher the wicking distance at the same interval the better the fabric in wicking AATCC197,2012 (Geyter et al., 2006).(Plate-15)

The UDF, MDF_1, ADF_1, IDF_1, SDF_2, ADF_2, AND IDF_2 are tested to wicking. The 2.5cm*10cm long specimen was taken and at the bottom of specimen 1cm height was marked and the sample was submerged in water. With the help of stopwatch readings were taken. The testing is done for five times and the mean value of all the samples were calculated and the results are noted

3.17. Nomenclature of the samples

The nomenclature of the samples are presented in Table II

TABLE-II

Nomenclature of samples

S.no	Description of sample	Nomenclature used
1	Undyed Fabric	UDF
2	Madder Dyed Fabric	DF_1
3	Alum dyed Fabric Madder	ADF_1
4	Iron water dyed Fabric	IDF_1
5	Sappan Dyed Fabric	SDF_2
6	Alum dyed Fabric Sappan wood	ADF_2
7	Iron water dyed Fabric Sappan wood	IDF_2

Plate – 8 FT-IR ANALYSIS

Plate – 9 UV-VIS ANALYSIS

Plate – 10 Fabric Weight

Plate – 11 Fabric Thickness

Plate – 12 Fabric Stiffness

Plate – 13 Crock Meter

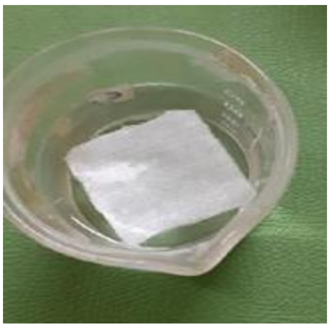


Plate – 14 Sinking

Plate – 15 Wicking

RESULTS AND DISCUSSION

IV.RESULTS AND DISCUSSION

The reults of the study **"Developing tints and shades in cotton fabric using *Rubia tinctorum* root and *Caesalpinia sappan* wood extract"** are discussed under the following headings.

4.1 Optimization of dye extraction conditions

4.1.1 Selection of solvent

4.1.2 Optimization of dye source concentration

4.1.3 Optimization of extraction time

4.1.4 Optimizzation of temperature

4.1.5 Optimized conditions for extraction of natural dye from ***Rubia tinctorum* root and *Caesalpinia sappan* wood**

4.2 Optimization of dyeing

4.2.1 Temperature

4.2.2 Time

4.2.3 Material liquor Ratio

4.2.4 Optimized conditions for dyeing cotton fabric with *Rubia* ***tinctorum* root and *Caesalpinia sappan* wood**

4.3 Mordanting techniques

4.3.1 Pre, Post and Simultaneous Mordanting

4.4 Dyeing under optimized conditions

4.5 Phytochemical Analysis

4.6 FT-IR Analysis

4.7 UV-VIS Analysis

4.8 Fabric Evaluation

4.8.1 Fabric Weight

4.8.2 Fabric Thickness

4.8.3 Fabric Stiffness Warp and Weft Direction

4.9 Colour Fastness to Washing, Crocking, Sunlight and Pressing

4.10 Absorbency and Wettability

4.10.1 Wicking

4.10.2 Sinking time

4.10.3 Spray test

4.1 Optimization of dye extraction conditions

4.1.1 Selection of Solvent

The impact of solvent on colour yield was analyzed. For dye extraction, different solvents such as water, alkali and ethanol were used independently. The extracted dye solution was used to dye the cotton fabric. The fabric colour coordinates and colour strength were investigated and the results are provided in the Table III and IV and Figure 4 and 5.

Table III

Selection of solvent

Rubia tinctorum root

Solvent	L*	a*	b*	K/S	Colour shade
Water	**65.406**	**1.488**	**23.506**	**18.897**	
Alkali (1% NaOH)	54.195	9.328	9.978	14.926	
Ethanol	61.111	-0.081	19.389	12.010	

From Table III, it is clear that the maximum colour strength (**18.897**) was found with water as a medium of extraction followed by alkali extraction. Hence, water was selected as medium of dye extraction from ***Rubia tinctorum*** root.

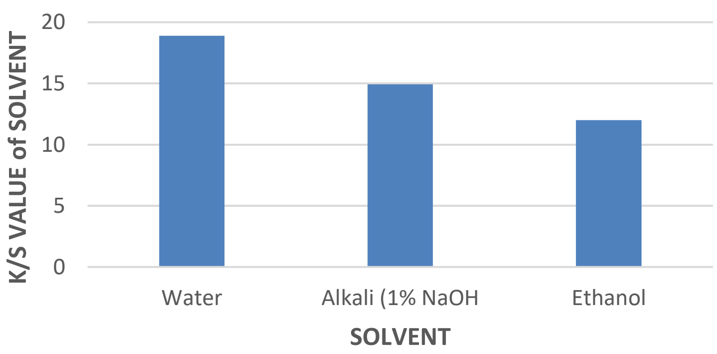


Figure 4- Effect of colour strength for various medium of extraction- *Rubia tinctorum* root

Table IV

Selection of solvent

***Caesalpinia sappan* wood**

Solvent	**L***	**a***	**b***	**K/S**	**Colour shade**
Water	**35.406**	**1.488**	**3.506**	**28.897**	
Alkali (1% NaOH)	34.195	1.328	2.978	24.926	
Ethanol	31.111	1.081	1.389	22.010	

From Table IV, it is clear that the maximum colour strength (**28.897**) was found with water as a medium of extraction followed by alkali extraction. Hence, water was selected as medium of dye extraction from ***Caesalpinia sappan*** *wood*.

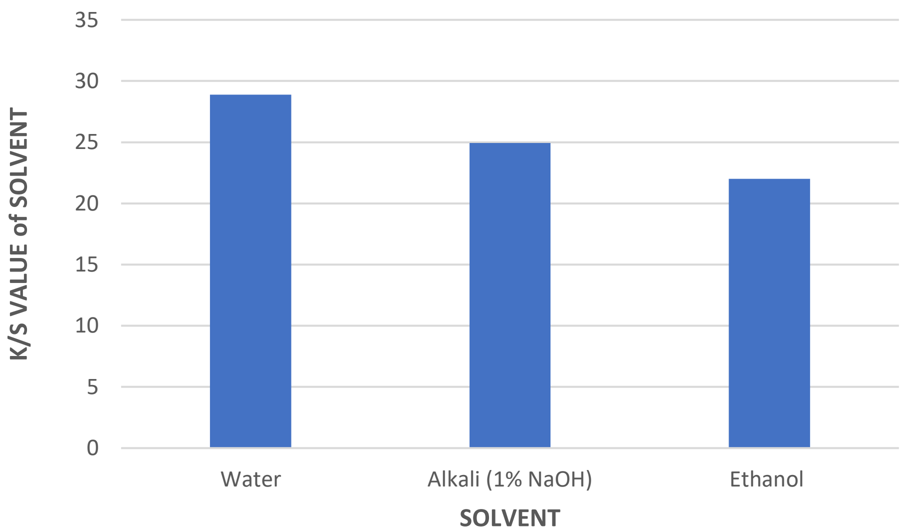


Figure 5– Effect of solvent on colour strength for various medium of extraction - *Caaesalpinia sappan* wood

4.1.2 Optimization of dye source concentration

The optimum dye source core was investigated and the results are presented in Table V and VI and Figure 6 and 7.

Table V

Optimization of Dye source concentration (Rubia tinctorum)

Source concentration (%)	**L***	**a***	**b***	**K/S**	**Colour shade**
3	94.753	2.191	1.222	23.304	
5	**94.566**	**1.813**	**2.093**	**31.134**	
7	95.194	2.314	1.091	30.976	

From Table V, it is clear that the maximum colour strength K/S (**31.134**) value was noticed at 5% dye source concentration. L* value was found to be lower in 6% dye source concentration which indicates that the colour yield is maximum at 5%. Hence, 5% dye source concentration was selected as optimum for the further experiments.

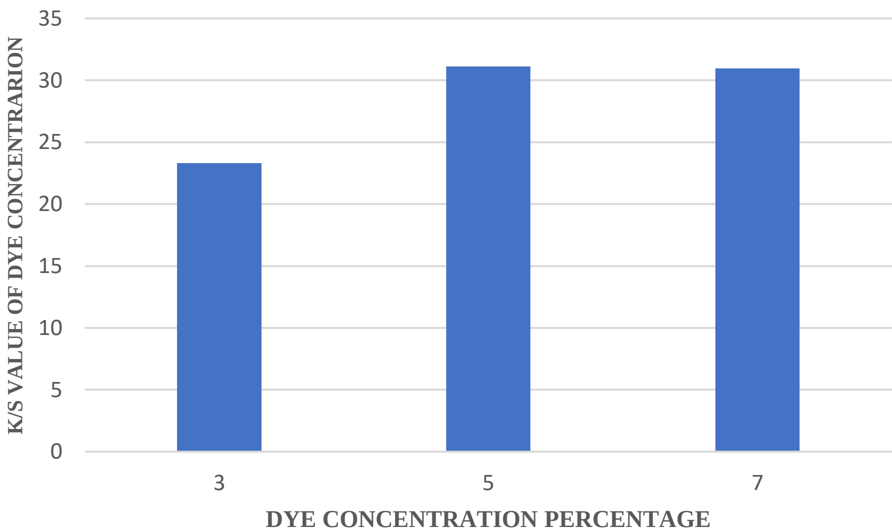


Figure 6- Effect of dye concentration on colour strength- *Rubia tinctorum root*

Table VI

Optimization of Dye source concentration (*Caesalpinia sappan*)

Source concentration (%)	L*	a*	b*	K/S	Colour shade
3	94.278	2.586	1.305	34.475	
5	**94.307**	**2.748**	**1.479**	**65.592**	
7	94.253	2.456	1.486	55.475	

From Table IV, it is clear that the maximum colour strength K/S(24.513) value was noticed at 5% dye source concentration. L* value was found to be lower in 7% dye source concentration which indicates that the colour yield is maximum at 5%. Hence, 5% dye source concentration was selected as optimum for the further study

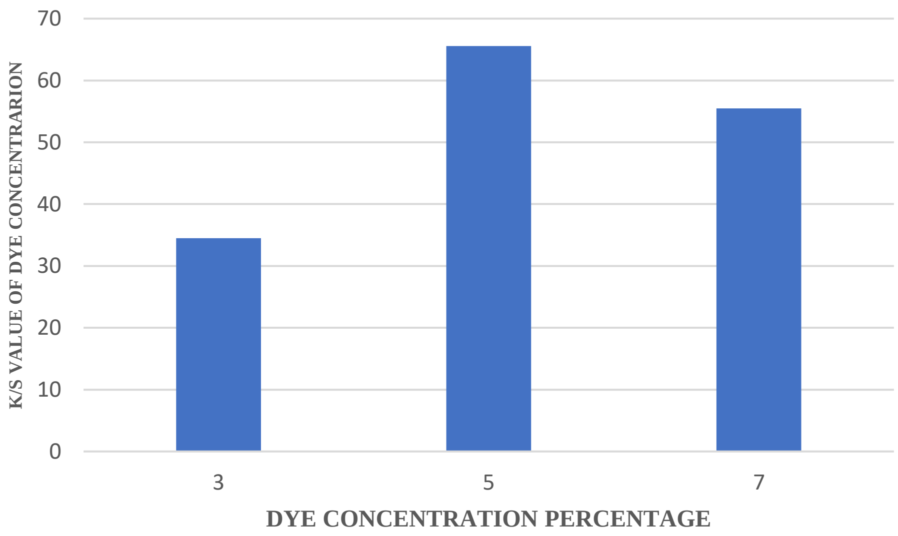


Figure 7 - Effect of dye source concentration on colour strength- *Caesalpinia sappan*

4.1.3 Optimization of extraction time

The dye was extracted at different time intervals (15, 30,45 min) and the extracted dye was used to dye the cotton fabric separately. The results of colour coordinates and colour strength of the dyed fabric are presented in Table VII and VIII and Figure 8 and 9

Table VII

Optimization of extraction Time

Rubia tinctorum

Time(min)	L*	a *	b*	K/S	Colour shade
15	94.245	2.001	1.369	19.501	
30	**94.354**	**2.071**	**1.514**	**20.997**	
45	94.445	2.020	1.769	19.901	

Table V, indicates that the colour strength K/S (20.997) value was found to be maximum in fabric dyed with dye extracted for 30 min. Further increase in extraction time does not found to increase the colour strength value of the fabric. Hence extraction time of 30min was fixed as optimum dye extraction time.

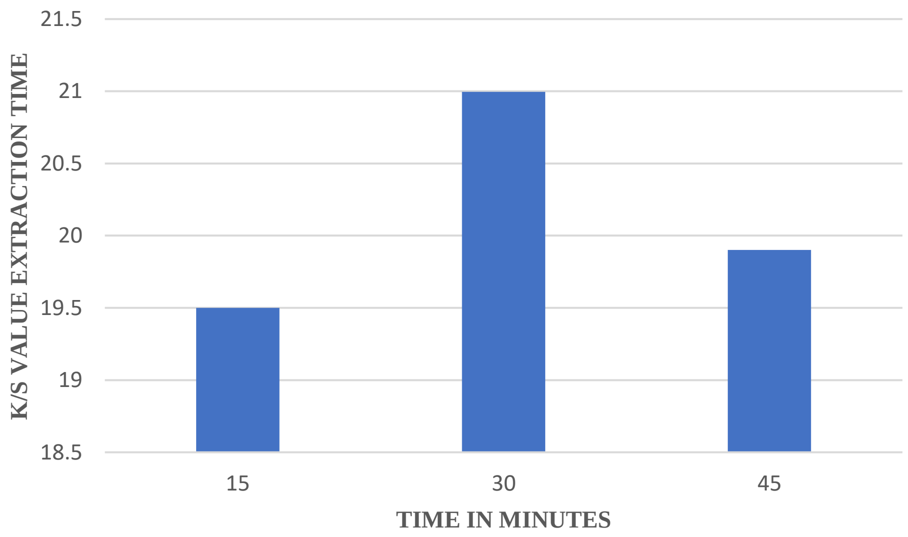


Figure 8 – Effect of extraction time on colour strength - *Rubia tinctorum*

Table VIII

Optimization of extraction Time

Caesalpinia sappan

Time(min)	L*	a *	b*	K/S	Colour shade
15	94.482	2.192	1.920	35.451	
30	**94.504**	**1.911**	**1.916**	**36.997**	
45	94.391	1.909	1.934	35.901	

Table VIII, indicates that the colour strength K/S (36.997) value was found to be maximum in fabric dyed with dye extracted for 30 min. Further increase in extraction time does not found to increase the colour strength value of the fabric. Hence extraction time of 30 min was fixed as optimum dye extraction time.

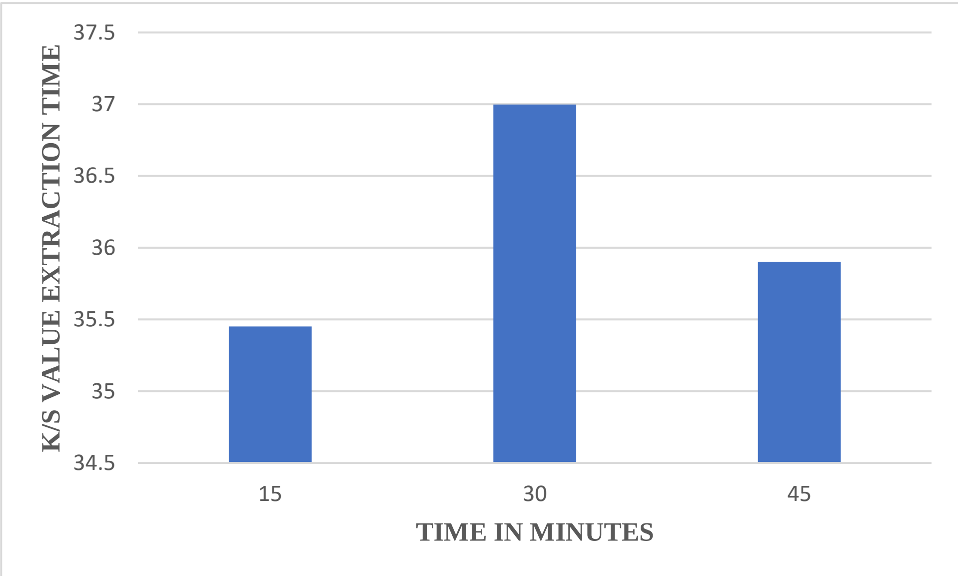


Figure 9 – Effect of extraction time on colour strength - *Caesalpinia sappan*

4.1.4 Optimization of extraction temperature

The extraction was carried out at various temperatures60º, 80º and 100º C to find the optimum temperature for effective dye extraction. The results of colour strength (K/S) and colour coordinates (L*, a*, b*) of the dyed fabric are presented in Table I X and X and Figure 10 and 11.

Table IX

Optimization of Extraction Temperature

Rubia tinctorum

Temperature (°C)	L*	a*	b*	K/S	Colour strength
60	92.180	6.515	4.202	29.046	
80	92.550	7.010	3.567	25.733	
100	**91.750**	**6.440**	**5.684**	**31.267**	

From Table VIII – it is evident that, the value of L*(31.267) was found minimum at 100°C. Also the colour strength of the dyed fabric K/S(15.827) was found to be maximum at 100°C. Colour strength values increased with rise in temperature from room condition to boiling condition (100°C). As the temperature increases it allows source to yield more dye into extract. The solubility of dye is increased at high temperatures and it gives more in filtrate and less in residue. As a result, the colour strength is increased with rise in temperature. Hence the extraction 100°C was selected as optimum temperature for the further experiments.

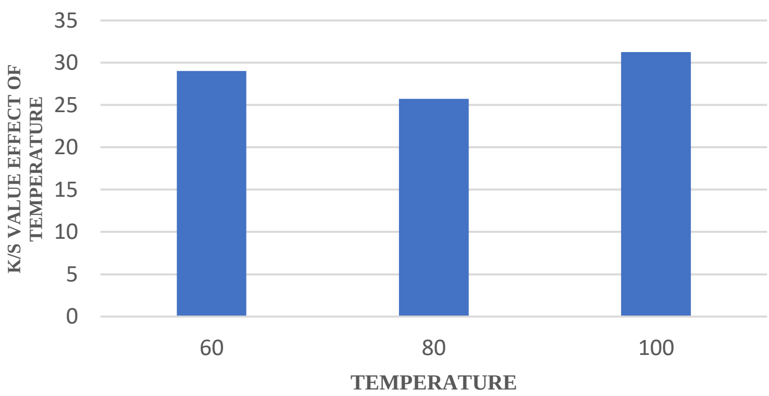


Figure 10- Effect of temperature on colour strength- *Rubia tinctorum*

Table X

Optimization of Extraction Temperature

Caesalpinia sappan

Temperature (°C)	L*	a*	b*	K/S	Colour strength
60	94.180	3.515	4.202	26.046	
80	94.550	3.010	3.567	29.733	
100	**94.450**	**3.340**	**4.684**	**33.267**	

From Table IX – it is evident that, the value of L*(33.267) was found minimum at 100°C. Also the colour strength of the dyed fabric K/S(15.827) was found to be maximum at 100°C. Colour strength values increased with rise in temperature from room condition to boiling condition (100°C). As the temperature increases it allows source to yield more dye into extract. The solubility of dye is increased at high temperatures and it gives more in filtrate and less in residue. As a result, the colour strength is increased with rise in temperature. Hence the extraction 100°C was selected as optimum temperature for the further experiments**.**

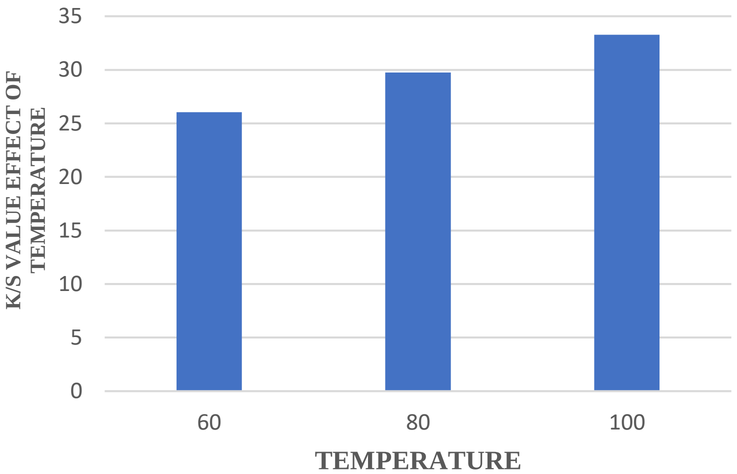


Figure 11- Effect of temperature on colour strength - *Caesalpinia sappan*

4.1.5 Optimized conditions for extraction of natural dye from *Rubia tinctorum* root and *Caesalpinia sappan wood*

Optimized conditions for extraction of natural dye from ***Rubia tinctorum root and Caesalpinia sappan wood*** are presented in Table IX

Table XI

Optimized conditions for dye extraction

S.No	Parameters	Optimized conditions *Rubia tinctorum*	Optimized conditions *Caesalpinia sappan*
1	Solvent	Water	Water
2	Dye source concentration	5%	5%
3	Time	30 min	30mins
4	Temperature	100 °C	100^0

4.2 Optimization of dyeing

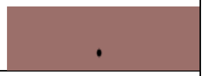
The extract obtained from the above method was filtered and used for the optimizationof dyeing at various effects of dyeing parameters such as temperature, dyeing time, liquor ratio and mordanting were noted for process optimization. Fabrics were dyed using extracted dye solution at 60° 80° and 100°C for temperature optimization and for optimized M:L ratio dyeing was done using 1:10, 1:20, 1:30, 1:40 and 1:50 material to liquid ratios. In order to observe the effect of dyeing time dyeing was carried out for 15, 30, and 45 minutes.

4.2.1 Temperature

Temperature plays an important role in dyeing because at high temperatures dyes mayundergo chemical degradation and at low temperatures, incomplete dyeing may occur (Adeel et al., 2009). The effect of temperature on dye uptake was studied at different temperatures (60°, 80° and 100°C) and the results of colour coordinates (L*, a*, b*) and colour strength(K/S*) values of dyed fabrics are presented in Table XII and Figure 12.

Table XII

Optimization of Dyeing Temperature

Temperature	L*	a*	b*	K/S	Colour shade
60°	61.218	7.293	20.059	19.046	
80°	**60.485**	**9.103**	**19.021**	**31.260**	
100°	61.787	7.228	20.698	35.733	

From Table XII, it is clear that the value of L*(60.485) was found to be minimum at 80°C.Also the colour strength value K/S (31.260) of dyed fabric found to be maximum at 80°C. Dyeing at 60°C resulted in poor exhaustion and dye uptake. When the temperature was raised, the kinetic energy of the dye molecules increased and it rushes into fix firmly in swelled fibre and disaggregation of dye at higher temperatures (Devi et el.,2020; Bhandari et al., 2021).

As a result, the dye uptake and colour strength values was found to be maximum. Increase in dyeing temperature increases the colour value of the dyed fabric. Hence, dyeing temperature of 80°C was fixed as optimum dyeing temperature.

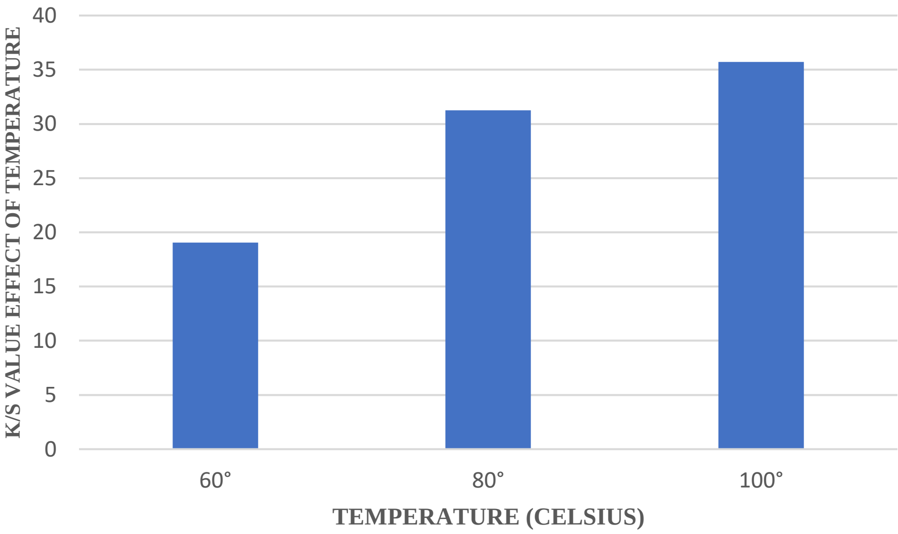


Figure 12- Effect of dyeing temperature on colour strength- *Rubia tinctorum*

4.2.2 Time

The impact of time on dye uptake has been investigated, and the findings are presentedin Table XIII and Figures 13.

TABLE XIII

Optimization of Dyeing Time

Time(MINS)	L*	a*	b*	K/S	Colour shade
15	94.554	2.095	1.414	19.807	
30	**94.354**	**2.071**	**1.514**	**20.997**	
45	94.445	2.001	1.769	19.901	

From Table XIII, it is clear that the colour strength K/S (20.997) value of dyed fabric was foundto be maximum at 30 min. Increase in dyeing time does not found to increase in colour strength value above 30 min. Long- and short-time dyeing gives the same effect as the variation of temperature. During constant heat for a long-time, decomposition of the dye material might occur while short-time causes incomplete dyeing (Adeel et al., 2009). The decline in the dye ability after 30min may be due to the desorption of the dye molecules as a consequence of long dyeing time (Prabu et al., 2002). And the positive values of a* and b* indicates that the fabric colour lies in redder and yellower region. Hence, the dyeing time of 30 min was fixed as optimum dyeing time.

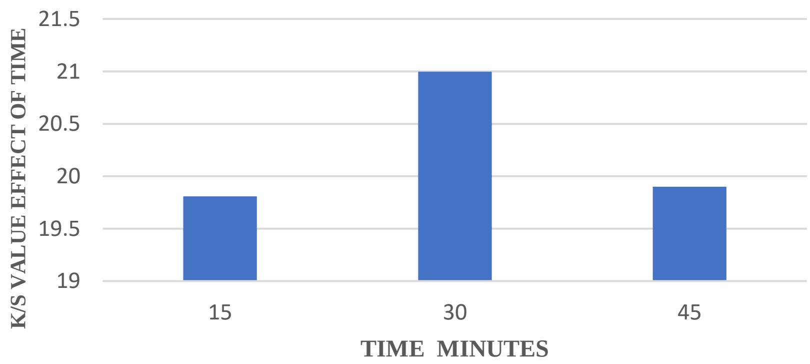


Figure 13- Effect of dyeing time on colour strength

4.2.3 Material Liquor Ratio

Material liquor ratio indicates the amount of liquor to be taken for dyeing a given weight of the material expressed in terms of M: L ratio. A ratio of 1:30 indicates that the weight of liquor should be 30 times the weight of material to be dyed. The relationship between the exhaustion, affinity and liquor ratio is important to maximize the exhaustion ofdye. The impact of material liquor ratio on dye uptake was studied, and the findings are presented in Table XIV and XIV and Figures 14.

Table XIV

Optimization of M.L.Ratio

M.L.Ratio	L*	a*	b*	K/S	Colour shade
1:10	94.566	1.813	2.093	21.301	
1:20	94.354	2.071	1.514	19.901	
1:30	**94.748**	**1.807**	**2.574**	**27.301**	
1:40	94.794	1.798	2.690	23.829	
1:50	94.445	2.001	1.769	20.997	

From Table XIV, it is clear that the colour strength K/S value (27.301) increased with increase in material liquor ratio and reached maximum at 1:30. Increased in material liquor ratio above 1:30 does not increase the colour strength K/S value. Increasing the volume of extract to dye the same weight of fabric which means more dye is available. Due to this more dye molecules approached fabrics, resulting an increase in colour strength value (Farooq et al., 2013). However, a further increase in M:L ratio above 1:30 results in dull shades due to the dilution of the molecules (Khan et al., 2014). Hence, material liquor ratio of 1:30 was fixed as optimum for further studies.

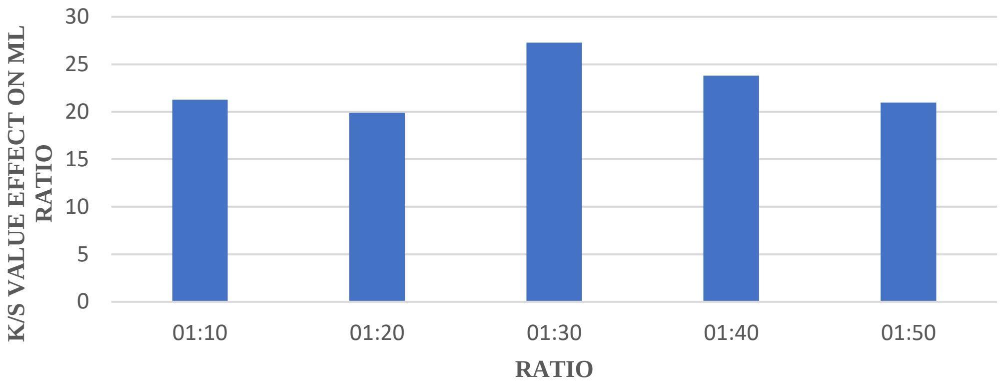


Figure 14- Effect of ML RATIO on colour strength

4.2.4 Optimized conditions for dyeing of cotton fabric with *Rubia tinctorum* root and *Caesalpinia sappan* wood extracts.

The optimized dyeing conditions for dyeing cotton fabric using ***Rubia tinctorum root*** *and* ***Caesalpinia sappan wood*** extract are presented in Table XV

Table XV

Optimized dyeing conditions

S.no	**Parameters**	**Optimized conditions *Rubia tinctorum***	**Optimized conditions *Caesalpinia sappan***
1.	Temperature	80°C	80°C
2.	Time	30 min	30 min
3.	M.L.Ratio	1:30	1:30

The dye uptake was maximum at the temperature of 80°C with material Liquor Ratio of 1:30at dyeing time of 30 min for both ***Rubia tinctorum*** **and *Caesalpinia sappan.***

4.3 MORDANTING

4.3.1 Pre ,Post and Simultaneous mordanting

The material was treated with alum and iron water before and after dyeing. Also dye and mordants were added simultaneously in separate baths. The colour strength(K/S) and colour coordinate (L*, a*, b*) values of the dyed cotton fabric are presented in Table XVI

Table XVI

Effect of Pre, Post And Simultaneous mordanting

SOURCE 1- MADDER

MORDANT 1- ALUM

MORDANTING TECNIQUES	L*	a*	b*	K/S	Shade Produced
PRE MORDANTING	94.681	1.953	2.459	35.645	
SIMULTANEOUS MORDANTING	94.758	1.623	2.619	26.876	
POST MORDANTING	94.623	1.728	2.229	17.338	

From Table XVI, it is evident that the colour strength K/S(35.645) value of pre-mordanting fabric was found to be maximum at 80°C. Lower temperature will cause fabric polymers shrink and no space for penetration of dye molecules into fabric. whereas higher temperature make fabric polymers free and provide space for penetration of dye molecules (Farooq et al., 2013).As a result the colour strength K/S was found to be higher at 80°C in pre-mordanting fabric.

Table XVII- Effect of Pre, Post And Simultaneous mordanting

SOURCE : MADDER

MORDANT 2: IRON WATER

MORDANTING TECNIQUES	L*	a*	b*	K/S	Shade Produced
PRE MORDANTING	**94.243**	**1.443**	**1.065**	**32.359**	
SIMULTANEOUS MORDANTING	94.378	1.105	1.407	26.471	
POST MORDANTING	93.378	1.005	1.307	23.571	

From Table XVII, it is evident that the colour strength K/S(32.359) value of pre-mordanting fabric was found to be maximum at 80°C. Lower temperature will cause fabric polymers shrink and no space for penetration of dye molecules into fabric. whereas higher temperature make fabric polymers free and provide space for penetration of dye molecules (Farooq et al., 2013). As a result the colour strength K/S was found to be higher at 80°C in pre-mordanting fabric

Table XVIII- Effect of Pre, Post And Simultaneous mordanting

SOURCE : SAPPON WOOD

MORDANT 1: ALUM

MORDANTING TECNIQUES	L*	a*	b*	K/S	Shade Produced
PRE MORDANTING	**94.113**	**3.010**	**0.949**	**31.637**	
SIMULTANEOUS MORDANTING	94.395	1.633	1.581	24.743	
POST MORDANTING	95.395	1.033	1.881	21.743	

From Table XVIII, it is evident that the colour strength K/S(31.637) value of pre-mordanting fabric was found to be maximum at 80°C. Lower temperature will cause fabric polymers shrink and no space for penetration of dye molecules into fabric. whereas higher temperature make fabric polymers free and provide space for penetration of dye molecules (Farooq et al., 2013). As a result the colour strength K/S was found to be higher at 80°C in pre-mordanting fabric

Table XIX- Effect of Pre, Post And Simultaneous mordanting

SOURCE: SAPPON WOOD

MORDANT 2: IRON WATER

MORDANTING TECNIQUES	L*	a*	b*	K/S	Shade Produced
PRE MORDANTING	**94.026**	**0.898**	**0.136**	**49.631**	
SIMULTANEOUS MORDANTING	94132	0.485	0.374	43.099	
POST MORDANTING	94.032	0.385	0.274	43.001	

From Table XIX, it is evident that the colour strength K/S(49.631) value of pre-mordanting fabric was found to be maximum at 80°C. Lower temperature will cause fabric polymers shrink and no space for penetration of dye molecules into fabric. whereas higher temperature make fabric polymers free and provide space for penetration of dye molecules (Farooq et al., 2013). As a result the colour strength K/S was found to be higher at 80°C in pre-mordanting fabric

DYEING UNDER OPTIMIZED CONDITIONS

The material was dyed with optimized conditions of parameters- solvent, dye concentration, Time, temperature, pH and M.L.Ratio. The colour strength (K/S) and colour coordinates (L*, a*, b*) of cotton fabric under optimized conditions are presented in Table XX

Table –XX
Colour strength and colour coordinates of dyed fabric

SEQUENCE OF DYEING	COLOUR COORDINATES			COLOUR STRENGTH K/S	SHADE PRODUCED
	L*	a*	b*		
PRE-MORDANT – ALUM DYE- MADDER	94.681	1.953	2.459	35.645	
PRE-MORDANT -IRON WATER DYE- MADDER	94.243	1.443	1.065	32.359	
PRE-MORDANT – ALUM DYE- SAPPAN WOOD	94.113	3.010	0.949	31.637	
PRE-MORDANT – IRON WATER DYE- SAPPAN WOOD	94.026	0.898	0.136	49.631	

4.4 Phytochemical analysis

Phytochemical analysis of natural dye extracted form *Rubia tinctorum* root and *Caespalinia sappan* wood are showed in Table XXI and XXII

TABLE XXI – Phytochemical Analysis – MADDER

S.No.	Metabolite	Test performed	Observation	Results
1.	Alkaloids	+Mayer's reagent	Absence of Cream coloured precipitate	-
		+ Dragendorff's reagent	Absence of reddish brown precipitate	-
2.	Flavonoids	Alkaline test	No colour change	-
		$+H_2SO_4$	Presence of reddish Orange colour	+
		+lead acetate	Absence of white Precipitate	-
		Shinoda test	Absence of crimson Pink colour	-
3.	Sterols (Libermann test)	$+CHCl_3$+ Acetic anhydride +Conc.H_2SO_4	Presence of reddish brown ring	+
4.	Terpenoids (Libermann test)	+ $CHCl_3$+ Acetic anhydride + Conc. H_2SO_4	Absence of green colour	-
5.	Anthraquinone (Borntrager's test)	+ $FeCl_3$ + Conc.HCl+diethyl ether +Ammonia	Presence of reddish orange colour	+
6.	Anthocyanin	HCl Test	No Colour change	-
7.	Proteins	+2%Ninhydrin reagent	Absence of Purple colour	-
		+2%$CuSO_4$+ 95%ethanol+KOHpellet	Absence of blue colour	-
		+conc. HNO_3	Presence of Yellow Colouration	+
8.	Phenolic compounds	+5% neutral $FeCl_3$	Absence of bluish green coloured solution	-

		Gelatin test	Absence of white precipitate	-
		Ellagic acid test	Absence of nigger brown precipitate	-
9.	Quinones	Conc.HCl	Absence of yellow precipitate	-
		Alcoholic KOH	Absence of reddish solution	-
10.	Carbohydrates	Molisch's test	Presence of Violet ring	+
		Fehling's test	Presence of Red precipitate	+
11.	Tannin	Braymer's test	Absence of bluish green colour	-
		+Gelatin test	Absence of white precipitate	-
		10% NaOH test	Absence of emulsion	-
12.	Saponins	Shaken with water	Presence of foam	+
13.	Cardiac glycosides	+Baljet reagent	Absence of yellow orange colour	-
		Bromine water test	Absence of yellow precipitate	-
		Keller-killani test	Presence of brown ring	+
14.	Glycoside's test	Borntrager's test	Absence of pink coloured solution	-
		Aq.NaOH test	Absence of yellow coloured solution	-
15.	Lignin	+Gallic acid	Absence of olive-green colour	-
16.	Coumarins	Fluorescence test	No yellow fluorescence	-
		+10%NaOH + $CHCl_3$	Absence of yellow colour	-
17.	Volatile oils	Fluorescence test	No pinkish fluorescence	-

TABLE XXII -Phytochemical Analysis -SAPPAN WOOD

S.No.	Metabolite	Test performed	Observation	Results
1.	Alkaloids	+Mayer's reagent	Absence of Cream coloured precipitate	-
		+ Dragendorff's reagent	Absence of reddish brown precipitate	-
2.	Flavonoids	Alkaline test	No colour change	-
		+H_2SO_4	Presence of reddish Orange colour	+
		+lead acetate	Absence of white Precipitate	-
		Shinoda test	Absence of crimson Pink colour	-
3.	Sterols (Libermann test)	+$CHCl_3$+ Acetic anhydride +Conc.H_2SO_4	Presence of reddish brown ring	+
4.	Terpenoids (Libermann test)	+ $CHCl_3$+ Acetic anhydride + Conc. H_2SO_4	Absence of green colour	-
5.	Anthraquinone (Borntrager's test)	+ $FeCl_3$ + Conc.HCl+diethyl ether +Ammonia	Absence of reddish orange colour	-
6.	Anthocyanin	HCl Test	No Colour change	-
7.	Proteins	+2%Ninhydrin reagent	Absence of Purple colour	-
		+2%$CuSO_4$+ 95%ethanol+KOHpellet	Absence of blue colour	-
		+conc. HNO_3	Presence of Yellow Colouration	+
8.	Phenolic compounds	+5% neutral $FeCl_3$	Absence of bluish green coloured solution	-

		Gelatin test	Absence of white precipitate	-
		Ellagic acid test	Absence of nigger brown precipitate	-
9.	Quinones	Conc.HCl	Absence of yellow precipitate	-
		Alcoholic KOH	Absence of reddish solution	-
10.	Carbohydrates	Molisch's test	Presence of Violet ring	+
		Fehling's test	Presence of Red precipitate	+
11.	Tannin	Braymer's test	Absence of bluish green colour	-
		+Gelatin test	Absence of white precipitate	-
		10% NaOH test	Absence of emulsion	-
12.	Saponins	Shaken with water	Presence of foam	+
13.	Cardiac glycosides	+Baljet reagent	Absence of yellow orange colour	-
		Bromine water test	Absence of yellow precipitate	-
		Keller-killani test	Absence of brown ring	-
14.	Glycoside's test	Borntrager's test	Absence of pink coloured solution	-
		Aq.NaOH test	Absence of yellow coloured solution	-
15.	Lignin	+Gallic acid	Absence of olive-green colour	-
16.	Coumarins	Fluorescence test	No yellow fluorescence	-
		+10%NaOH + $CHCl_3$	Absence of yellow colour	-
17.	Volatile oils	Fluorescence test	No pinkish fluorescence	-

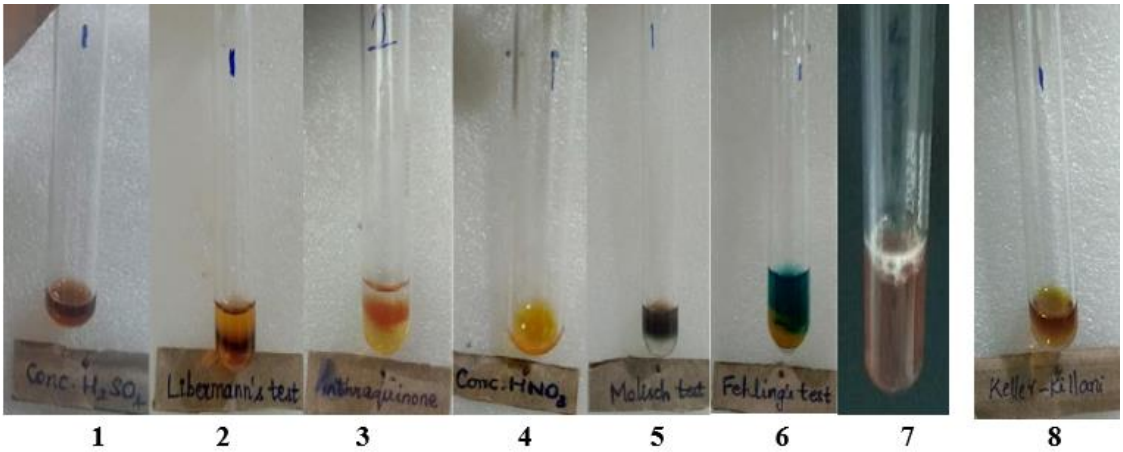


Figure 15- Phytochemical screening Tests MADDER

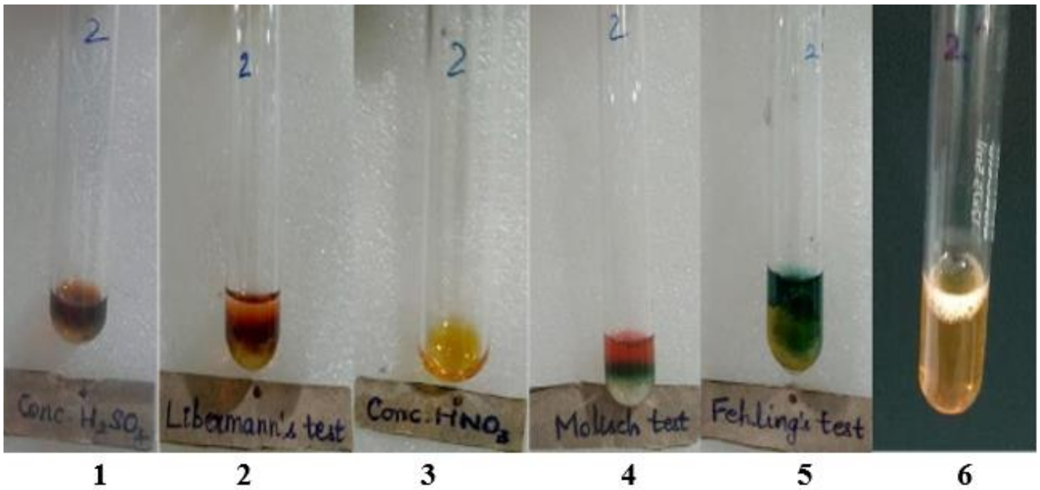


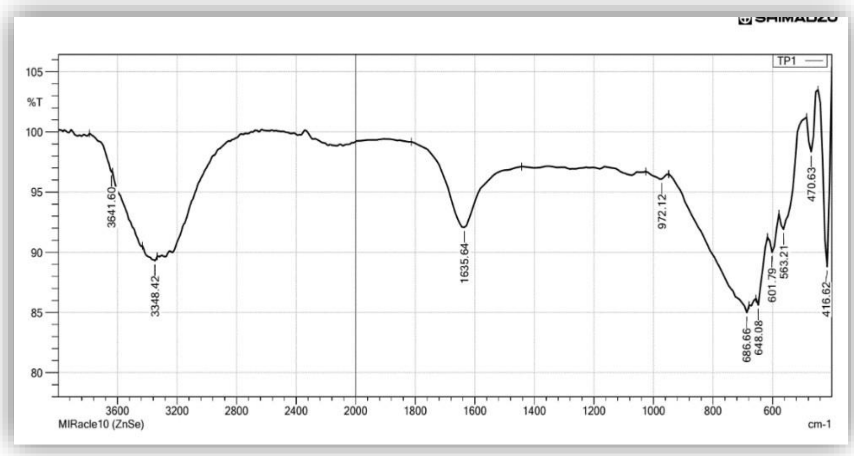
Figure 16- Phytochemical screening Tests SAPPAN WOOD

Note : '+' Positive, '_' Negative

Table XV, reveals the composition of phytochemicals presented in extracted dye. Thespesia populnea flower dye has rich in sources of alkaloids, flavonoids, sterols, anthraquinone, anthocyanin, proteins, phenolic compounds, carbohydrates, tannins, saponins, cardiac glycosides, coumarins and volatile oils.

4.5 FT-IR

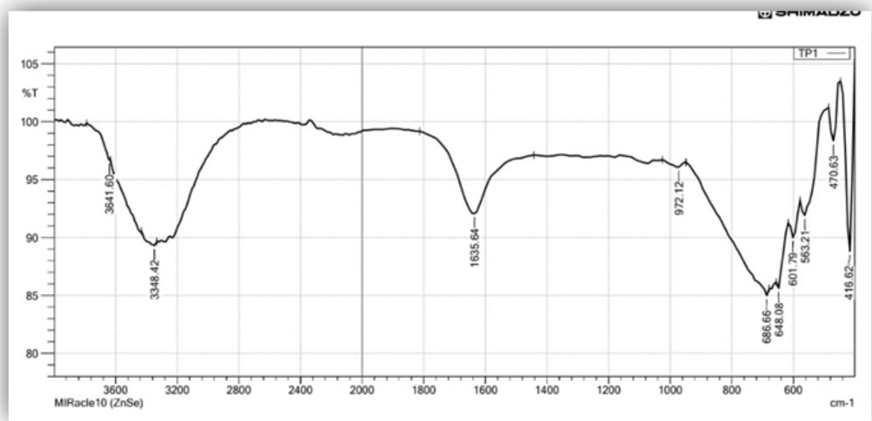
The FT-IR spectroscopy of the natural dye extracted from the flower of ***Rubia tinctorum root*** and ***Caesalpinia sappan wood*** are showed in Figure 15 and 16



Wavenumber (cm^{-1})

Figure 17 – FT-IR spectra of *Rubia tinctorum* root extract

The FTIR spectral analysis of the extracted dye show distinct peaks at 3348.42, 1635.64, 686.66 and 648.08 cm^{-1} respectively (Figure 17). The broad and strong band at 3348.42 cm^{-1} can be attributed to bonded -OH stretching groups. The peak at 1635.64 cm^{-1} indicative of C=C stretching vibration. The strong peak at 686.66 cm^{-1} represents C-Br stretching halo compoundgroup. Finally, the peak at 648.08 cm^{-1} represents strong C-Br stretching group. Hence, FTIR spectral analysis show the presence of different chemical functional groups like -OH, C=C, C-Br and C-Br in the extracted dye.



Wavenumber (cm^{-1})

Figure 18 – FT-IR spectra of *Caesalpinia sappan* wood extract

The FTIR spectral analysis of the extracted dye show distinct peaks at 3348.42, 1635.64, 686.66 and

648.08 cm-1 respectively (Figure 18). The broad and strong band at 3348.42 cm-1 can be attributed to bonded -OH stretching groups. The peak at 1635.64 cm-1 indicative of C=C stretching vibration. The strong peak at 686.66 cm-1 represents C-Br stretching halo compound group. Finally, the peak at 648.08 cm-1 represents strong C-Br stretching group. Hence, FTIR spectral analysis show the presence of different chemical functional groups like -OH, C=C, C- Br and C-Br in the extracted dye.

4.6 UV-VIS analysis

UV-VIS analysis of ***Rubia tintorum*** **root** extract results are presented in Table XXIII

Table XXIII- UV-VIS analysis MADDER ROOT

Wavelength (nm)	Absorbance
220	8.418
280	5.151
281	5.088
282	5.028
283	4.975
400	1.428
600	0.411
800	0.148

From Table XXIII , it reveals that the wavelength of ***Rubia tintorum*** **root** extract was peak at 220nm and the absorbance level of dye extract absorbed at 0.148 wavelength of 800

Table XXIV- UV-VIS analysis SAPPAN WOOD

Wavelength (nm)	Absorbance
220	9.418
280	7.151
281	7.088
282	7.028
283	6.975
400	2.428
600	0.411
800	0.148

From Table XXIV , it reveals that the wavelength of ***Rubia tintorum*** **root** extract was peak at 220nm and the absorbance level of dye extract absorbed at 0.148 wavelength of 800 (Fig 15)

4.7 FABRIC EVALUATION

4.7.1 Fabric weight

GSM of undyed fabric, dyed fabric and mordant dyed fabric samples were analyzed and the values are presented in Table-XXV.

TableXXV

Weight of the fabric

S.no	Sample	Mean fabric Weight (GSM)	Gain or loss over original	% gain or loss over original
1	UDF	1.705	-	-
2	MDF_1	1.830	1.25	7.3
3	ADF_1	1.877	1.72	10.8
4	IDF_1	1.887	1.82	10.7
5	SDF_2	1.810	1.50	6.2
6	ADF_2	1.868	1.63	9.6
7	IDF_2	1.873	1.68	9.9

From Table XVII, it is observed that the weight of all dyed and mordant dyed fabric is increased when compared to undyed fabrics. Among the dyed and mordant dyed samples ADF_1 and IDF_2 showed 10 and 9 percent imcrease in weight respectively. (Figure 19).

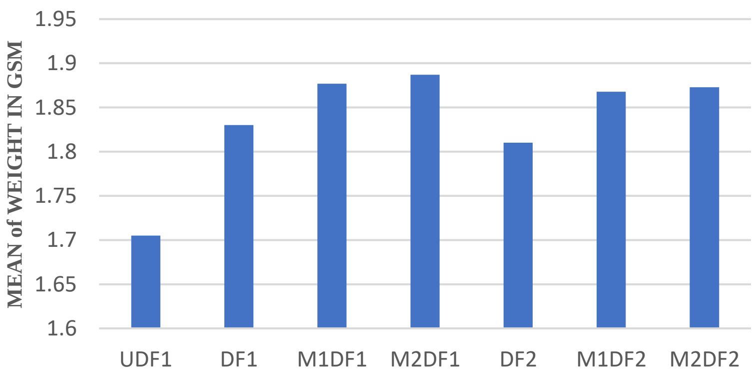


Figure19

Fabric Weight

4.7.2 Fabric Thickness

The thickness of undyed, dyed and mordant dyed samples are analyzed and presented in Table XXVI.

Table XXVI

Fabric Thickness

S.no	Sample	Mean fabric thickness(mm)	Gain or loss Over original	%gain or loss over original
1	UDF	0.35	-	-
2	MDF_1	0.43	0.08	22.5
3	ADF_1	0.48	0.13	37.14
4	IDF_1	0.50	0.15	42.8
5	SDF_2	0.44	0.09	25.7
6	ADF_2	0.56	0.19	54.3
7	IDF_2	0.51	0.16	45.7

From Table XXVI, it is observed that the thickness of all dyed and mordant dyed fabric is increased when compared to undyed fabrics. Among the dyed and mordant samples IDF_1 AND ADF_2 showed 42 and 54 percent increase in thickness respectively. (Figure 20).

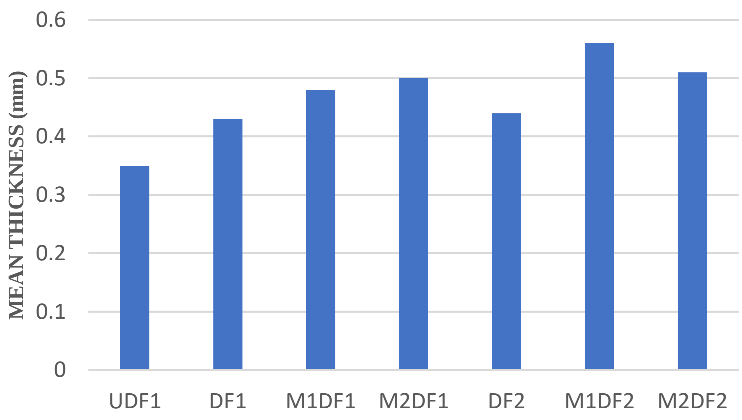


Figure –20

Fabric Thickness

4.7.3 Fabric Stiffness

The stiffness of samples in warp and direction are analysed and values are presented in Table-XXVII

Table XXVII

Fabric stiffness

S.no	Sample	Mean fabric stiffness(cm)		Gain or loss Over original		%gain or loss over original	
		Warp	Weft	Warp	Weft	Warp	Weft
1	UDF	3	3.8	-	-	-	-
2	MDF1	2.9	3.6	0.1	0.2	3.3	5.3
3	ADF1	2.8	3.3	0.2	0.5	6.7	13.2
4	IDF1	2.5	3.3	0.5	1.5	16.7	39.5
5	SDF2	2.9	3.7	0.6	0.4	20	10.5
6	ADF2	2.4	3.4	0.1	0.1	3.3	2.6
7	IDF2	2.8	3.3	0.2	0.5	6.7	13.2

From Table XIX, it is observed that the stiffness of undyed fabric is increased when compared to dyed and mordant dyed fabrics. Among the dyed and mordant samples MDF_1 and ADF_2 showed 3 and 3 percent stiffness loss in warp direction and 5 and 2 percent stiffness in weft direction respectively. (Figure 21).

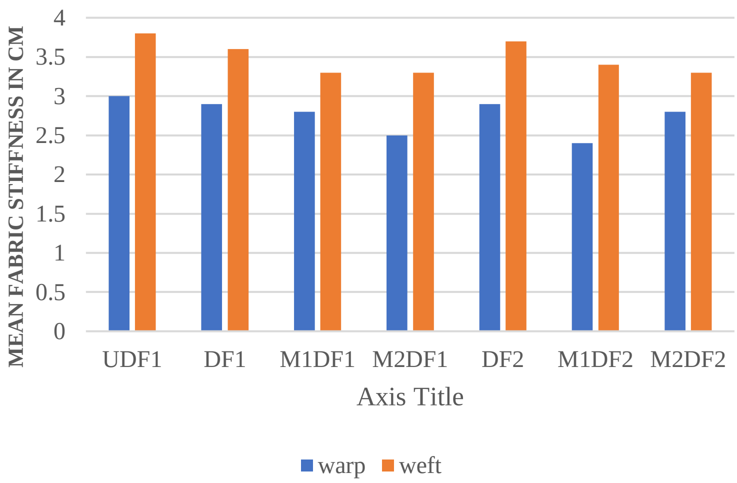


Figure-21
Fabric stiffness

4.7.4 Fabric strength

The fabric strength of undyed, dyed and mordant dyed fabrics in both warp and weft are represented in XXVIII

Table - XXVIII
Tensile strength

S.no	Sample	Mean fabric Tensile strength(kg)		Gain or loss Over original		%gain or loss Over original	
		Warp	Weft	Warp	Weft	Warp	Weft
1	UDF	46	56	-	-	-	-
2	MDF1	49	59	2	3	4.5	5.3
3	ADF1	53	60	7	4	5.2	6.2
4	IDF1	48	61	2	5	4.3	8.9
5	SDF2	51	59	5	3	10.2	5.4
6	ADF2	48	62	2	6	4.3	10.7
7	IDF2	50	61	4	5	8.7	8.9

From Table XXVIII, it is observed that dyed and mordant dyed fabrics has increased tensile strength in both warp and weft direction compared to undyed fabric. Among the dyed and mordant dyed samples, MDF_1 and SDF_2 showed 4.5 and 10.2 percent in warp direction and SDF_2 and ADF_2 showed 5.4 and 10.7 percent in weft direction respectively.

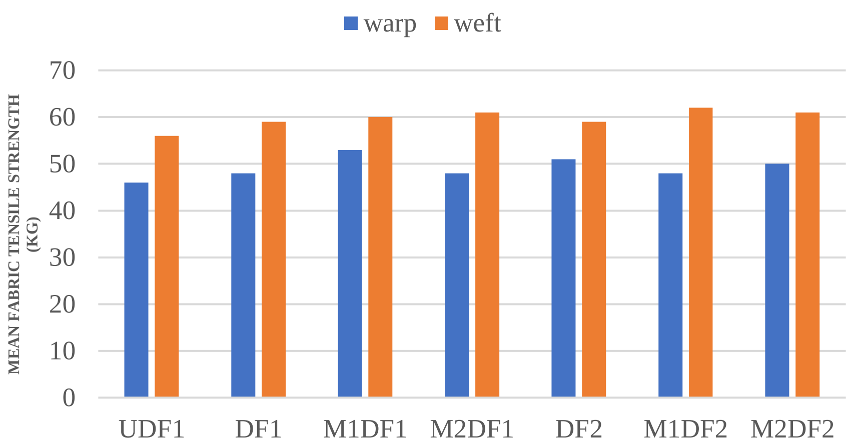


Figure 22

Fabric Tensile Strength

4.8 Color fastness to washing, crocking, sunlight and pressing

Natural dyed cotton fabric color fastness properties are assessed to washing, crocking and sunlight and results are presented in Table -XXIX

Table- XXIX

Colour fastness to washing, crocking, sunlight and pressing

S.no	Sample	Washing	Sunlight	Crocking		Pressing	
				Dry	Wet	Dry	Wet
1	DF	2	2	4/5	3/5	3/5	3/5
2	ADF1	4	3	4/5	3/5	4/5	3/5
3	IDF1	4	3	4	3	4	3
4	ADF2	4	3	4	3	4	3
5	IDF2	4	3	4	3	4	3

From Table XXIX, it is evident that the colour fastness of dyed and mordant dyed fabrics shows good fastness properties. However, un mordant natural dyed samples showed poor to fair fastness properties. Mordant treated fabric shows good fastness properties for washing and rubbing than dyed fabric. Dyed and mordant dyed fabric shows fair to good fastness properties in rubbing and pressing (Figure 23).

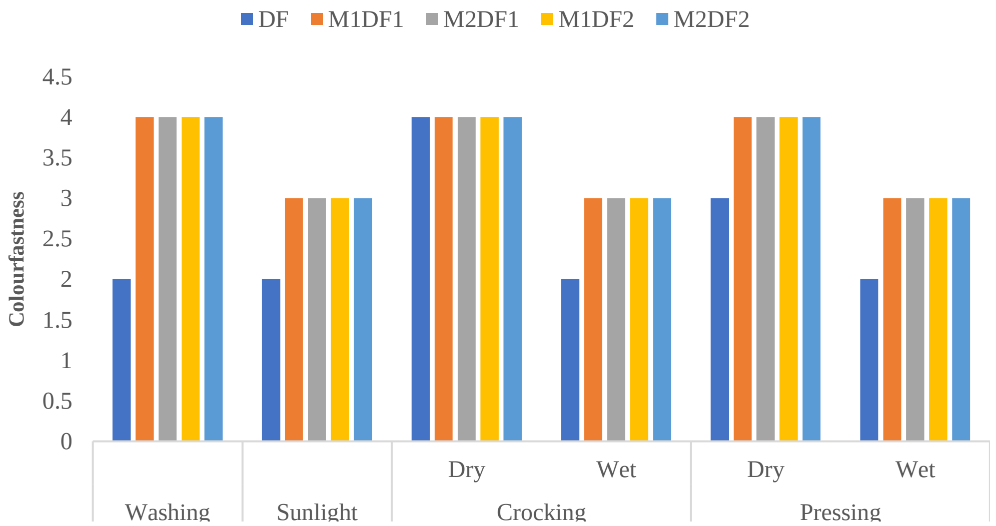


Figure-23 Colourfastness

4.9 ABSORBENCY

4.9.1 Wicking

The results of wicking ability of undyed, dyed and mordant fabric are presented in the Table – XXII and Figure 24**.**

TableXXX

Wickability of fabric

S.no	Sample	Mean absorbency in 1 minutes(cm)			
		Time constant	Extended absorbability (5cm)	Gain or loss over original	%gain or loss over original
1	UDF	1min	0.26	-	-
2	MDF1	1min	0.43	0.65	65
3	ADF1	1min	1.68	0.96	96
4	IDF1	1min	1.65	0.95	95
5	SDF2	1min	0.43	0.65	65
6	ADF2	1min	1.68	0.96	96
7	IDF2	1min	1.65	0.95	95

From Table XXX, it is evident that the mordant dyed samples increased when compared to dyed fabric. Mordant dyed fabrics ADF1, IDF1, ADF2 and IDF2 exhibits high absorbency when compared to dyed and undyed fabric (figure 24)

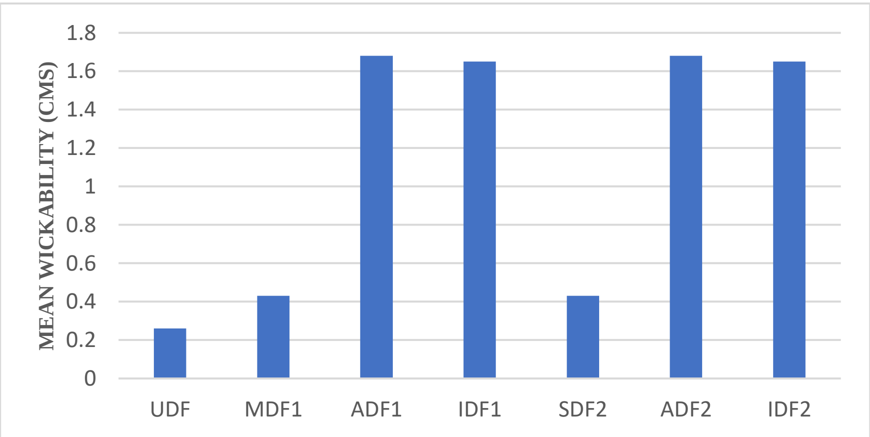


Figure24–Wickability of fabric

4.9.2 Sinking time

The results of fabric sinking time of undyed, dyed and mordant dyed fabrics are represented in XXXI and figure 22.

Table XXXI

Sinking time

S.no	Sample	Mean sinking time (60 seconds)
1	UDF	45
2	MDF1	27
3	ADF1	22
4	IDF1	23
5	SDF2	27
6	ADF2	23
7	IDF2	21

From Table XXXI, it is clear that the time taken for sinking of undyed fabric is increased when compared to their dyed and mordant dyed fabric. The sinking time of dyed and mordant dyed fabric was found to be decreased than undyed fabric. From the above absorbency test, it can be concluded that absorbency was found to be increased in mordant dyed fabric. Among the dyed and mordant dyed samples ADF_1 and IDF_2 showed high absorbency at 22 and 21 seconds respectively (Figure22)

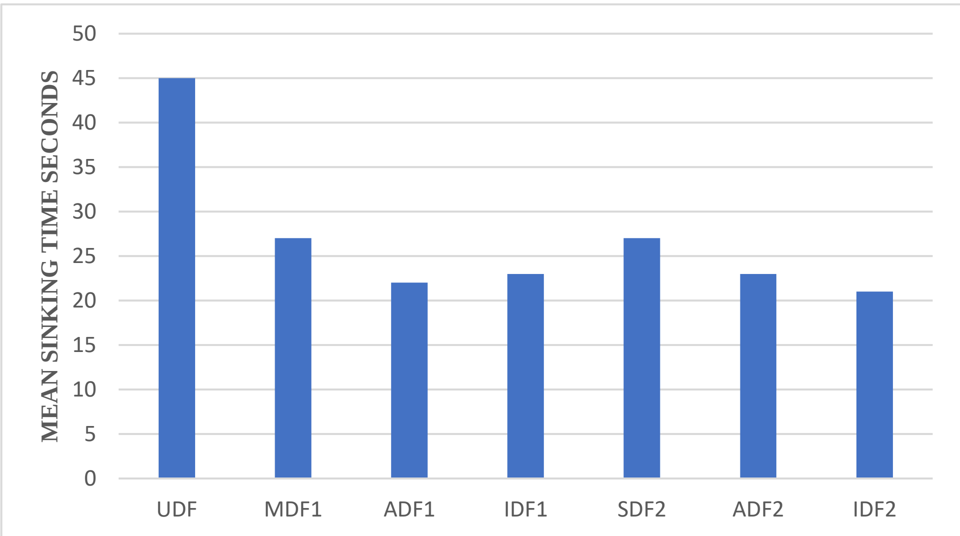


Figure25
Fabric sinking Time

SUMMARY AND CONCLUSION

V. SUMMARY AND CONCLUSION

In human life colours has played a dominant role since time immemorial. It adds beauty to the world. Colouring to textiles is one of the fascinating art. Dyes are available in two forms either natural or synthetic and both these dyes are used for dyeing fibres, yarns and fabrics. Nowadays mostly colouring textiles with natural dyes has become popular because of disadvantages associated with synthetic dyes. The use of synthetic dyes has cut down significantly due to toxic effluent resulting from the dyeing process of these dyes. As people become more aware of environmental concerns and sustainability make natural dyes back to moment. Natural dyes were obtained from animal, vegeTable and mostly various parts of plants such as leaves, fruits, roots, barks and flowers. Natural dyes can be classified based on their chemical structure, origin, method of application and colour. The twomain advantages of natural dyes compared to synthetic dyes are: synthesis processes within natural materials performed by nature without environmental pollution and biodegradability ofnatural materials, thus not affecting hazardous effluent upon degradation in the environment.

These dyes produce very uncommon, soothing and soft shades. These dyes are bio degradable, eco-friendly, renewable and less allergic. It makes everyone to practice natural sources as colouring of textiles. These products are comforTable for the wearer as it has soothing colour and non-allergic properties. The use of natural dyes will also generate more employment for rural people. Naturally dyed products represent the good opportunity for value added manufacturing as the process is eco-friendly. Using plant dyes at full potential for dyeing of textiles, it needs a suiTable and scientific technique for extraction of dyes and their application on textile materials without compromising the quality of fabric and these dyes poses medicinal properties. Application of natural dyes on textiles felt a needto reinvestigate and rebuilding of traditional process of natural dyeing to control each treatment and pre-dyeing process and producing uncommon shades with balanced fastness properties and eco-performance on textiles (Alemayehu, 2014, Samanta and Agarwal, 2009).

Mordant is known as the element which help in binding of dyes to fabric by forming chemical bridge from dye to fibre thus improving the staining ability of a dyewith increasing its fastness properties. Mostly it applies on fabric in three separate ways- pre-mordanting, meta mordanting and post mordanting. Usage of different mordants with same dye can shift the colours of a wide range or create totally new colours. Natural dyes produce high valued textiles by artisan/craftsman, small scale/ cottage level dyers and printers as well as small scale exporters worldwide. Mostly commercial dyers looking for the possibilities of using natural dyes for regular basis. Therefore, new pigment crops and modern dyeing techniques are being investigated to meet the growing demand for natural colourants.

Common drawbacks of natural dyes are their non-reproducible and non-uniform shades, poor to moderate colour fastness due to lack of scientific information on the chemistry of extraction and dyeing. Extraction of colour component from natural sources is an importantstep for dyeing any textile substrate. In order to evaluate their dyeing characteristics and maximize the colour yield on textile fabrics standardized dyeing method yields good results. The techniques of standardization such as parameters of pH, time, temperature and dye concentration for dye extraction and dyeing are optimized conditions in standardized dyeing method. Standardization of extraction process and optimizing the extraction variables both, have technical and commercial importance on colour yield and cost of extraction process as well as dyeing cost. Standardised recipe and dyeing technique help to maintain natural dyes as reproducible dyes, creating newer shades, improving fatness process and affinity of dyes onall natural fibres.

Hence, the present study entitled "**Developing tints and shades in cotton fabric using *Rubia tinctorum* root and *Caesalpinia sappan* wood extracts"** was carried out with the following objectives

- To select natural dye source for mordanting and dyeing
- To extract and optimize dye from the selected plant sources.
- To select and prepare the cotton fabric for dyeing with natural surfactants
- To optimize the dyeing conditions to develop the tints and shades of the selected plants sources
- To access the colour strength and evaluate the dyed fabric for performance properties.

Experimental procedure

The methodology pertaining to the study is presented in following steps:

- The substrate selected for the study was 100 percent natural fibre -cotton fabric.
- The preparatory process for all textiles is necessary to remove all the impurities uniformly before dyeing prior to finishing processes. Desizing process is carried out as a pre -treatment step to remove starch from cotton fabric.
- The dye source selected for the study was *Rubia tinctorum root and Caesalpinia sappan heartwood*. The dye was extracted from *Rubia tinctorum root and Caesalpinia sappan heartwood* by aqueous extraction.
- The mordant selected for the study was alum and iron water.
- Various parameters such as solvent (water), dye source concentration (3%,5%&7%), time (15, 3 0 & 4 5 min) and temperature (60°C , 8 0 & 100°C) and pH were optimized for dye extraction.
- Optimization of dyeing conditions was carried out at various parameters such as time (15min – 45min)., temperature (60°C- 100°C), material liquor ratio (1:10 – 1:50).
- Fabric was treated with selected mordants with various mordanting techniques. Dyeing was done at optimized conditions.
- Colour strength of the optimized dyed fabric was measured using premier colour scam spectrophotometer.
- Physical properties of fabric such as fabric thickness, weight, stiffness and absorbency through wicking and sinking was assessed for all the dyed fabrics.
- Colour fastness properties of fabric to washing, crocking and light were analyzed.

Findings of the study:

- Water was found to be suiTable solvent for dye extraction.
- Concentration of 5% (*Rubia tinctorum root and Caesalpinia sappan heartwood*) was found to be optimum concentration for dye extraction.
- Optimum dye extraction time was found to be 30 min.
- Dye extraction was found to be maximum at 1 0 0°C
- The optimum material liquor ratio selected for the dyeing cotton fabric was 1:30

- ❖ Optimum dyeing time and temperature was found to be 30 min & 80 °C respectively.
- ❖ The dye uptake was found to be increased after using mordant (alum and iron water).
- ❖ The maximum uptake of dye was found in pre-mordanting technique.
- ❖ In pre-mordanting technique, the dye uptake was found to be good at 80°C
- ❖ Weight of the fabric was found to be increased in mordant dyed fabrics ADF_1 and IDF_2
- ❖ The thickness of the mordant dyed fabrics IDF_1 AND ADF_2 were found to be increased when compared to dyed fabric.
- ❖ The stiffness of dyed fabrics was found to be lower when compared to undyed fabric in both warp and weft direction.
- ❖ With regards to colour fastness tests to washing, sunlight and crocking, the mordant dyed fabrics showed excellent fastness properties whereas the fabric dyed without mordant showed good fastness properties.
- ❖ In absorbency test, the wicking ability of mordant dyed fabrics ADF1, IDF1, ADF2 and IDF2 were found to be increased. The sinking time was found to be increased in mordant dyed fabrics ADF1 and IDF2

Conclusion

The results of the present study revealed that *Rubia tinctorum root and Caesalpinia sappan heartwood* extract was found to be potential natural dye source for textile dyeing. The process of extraction of dyeing is environmentally friendly and causes minimum environment pollution. The dye exhibited goodfastness properties. The study reveals that the extraction and dyeing parameters have significantinfluence on colour characteristics and quality of fabric. The optimized conditions were; solvent as water, dye source concentration-5%, extraction time 30min, temperature 100°C and pH 6, while the most suiTable dyeing parameters were; M.L.ratio 1:30, dyeing time 30mins and temperature 80°C. Excellent fastness properties were achieved with the use of pre-mordant technique. This work clearly indicates that the extract from *Rubia tinctorum root and Caesalpinia sappan heartwood* could be used as source for natural dyes and for dyeing of cotton fabric. The method of standardized dyeing for natural dyes produces uncommon shades with good fastness properties. This will at the same time has potential to reduce the usage of synthetic dyes.

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APPENDICE

VII. APPENDICE

Pre- mordanted with iron water and dyed with madder	
Pre- mordanted with alum and dyed with madder	
Pre- mordanted with iron water and dyed with sappan wood	
Pre- mordanted with alum and dyed with sappan wood