

**Eco-friendly Natural dye Extraction from Borassus flabellifer fruit for
colouration on cotton fabric**

By

Nivetha C

(21PTF011)

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 TEXTILES AND FASHION APPAREL**

May, 2023

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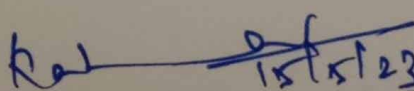
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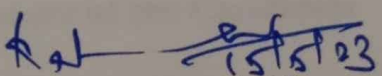
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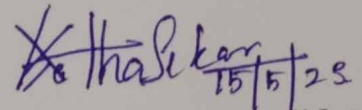
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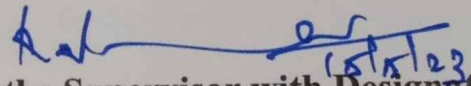
I declare that the dissertation entitled “Eco-friendly Natural dye Extraction from *Borassus flabellifer* fruit for colouration on cotton fabric” submitted for the degree of Master of Science (M.Sc.,) is the record of work carried out by me during the period from 2022 to 2023 under the guidance of Dr. K. KALAIARASI, M.Sc., Ph.D., M.Sc., M.Phil., Ph.D., Associate Professor and Head, Department of Textiles and Clothing, Avinashilingam Institute for Home Science Higher Education for Women, Coimbatore -642043 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.


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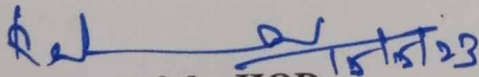
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I certify that dissertation entitled "**Eco-friendly Natural dye Extraction from Borassus flabellifer fruit for colouration on cotton fabric**" Submitted for the degree of Master of Science (M.Sc.,) Textiles and Fashion Apparel by **Nivetha C (21PTF011)** 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



Signature of the HOD

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1. INTRODUCTION

Natural dyes have been used since ancient times for colouring and printing. Since the middle of the 20th century, natural dye colours have been extracted from plant and animal sources. (Chatwal, 1985) The majority of natural dye pigments can be found in plant materials such as roots, bark, leaves, flowers, fruits, and seeds. Natural dyes are used to colour textiles, food, beverages, pharmaceuticals, cosmetics, printer ink, household products, and other items. (Gala et al., 2017)

Synthetic dyes made from petrochemical compounds were discovered due to the increasing demand for and prohibitive prices of natural dye extraction (Slama, 2021). Since the introduction of man-made synthetic dyes in the mid-nineteenth century, the dye market has been dominated by synthetic dyes due to a variety of competitive properties of synthetic dyes over natural dyes, such as lower cost, higher fastness, hue variety, ability to dye synthetic fibre, and availability on a large industrial scale (Kasiri et al., 2013). After the invention of Mauveine dye by William Perkin, the acceptance of synthetic dyes in the fields of food and textiles increased. (Teli et al.,2000;Bechtold et al, 2002)

However, the use of synthetic dyes creates an additional issue: environmental pollution. Synthetic dye waste that has not been properly processed will damage the environment and be hazardous to live organisms (Gala et al., 2017). A recent increase in interest in the development and application of dyes derived from natural sources has been recognised worldwide. This is due to these flaws as well as the growing awareness of environmentally friendly and healthy lifestyles. (Kasiri et al.,2013)

Natural dyes have a global demand of about 10,000 metric tonnes, which is equivalent to 1% of global synthetic dye consumption. This is expected to grow rapidly in the near future (Sivakumar, 2011). Natural dyes are non-toxic, health-curing, non-carcinogenic, renewable resources, and have ecologically beneficial properties that reduce pollution and cause no allergic reactions on the skin. (Ali et al.,2009;Leither et al.,2012;Sinha et al., 2012) No synthetic dye can compare to natural ones in terms of lustre, undertones of rich colour, fragrance, and that eye-pleasing combination of gentle light and shade. (Chatwal, 1985)

Besides this, the presence of a pigmental group comprising phenolic components confers antibacterial, antioxidant, and anticarcinogenic properties on plant-based natural dyestuffs.

(Selvan et al.,2015;Sathiyarayanan et al.,2010;Anwar et al.,2009) The market impact of environmentally friendly dyes is anticipated to grow in emerging nations like India over the next few years in both the industrial and small-scale segments. (S. Baliarsingh et al.,2012).

Natural dye has a low affinity for cotton, silk, and wool. Mordants are substances that create a bond between dye and the textile material, aiding in the improvement of fastness and dye uptake. Mordanting is the process of developing colour between natural dye and fabric. So here we use eco-friendly mordants like guava and pomegranate powder for the mordanting process. Pomegranate contains polyphenols (flavonoids and tannins) and guava (flavonoids and quercetin), which act as a colourant and mordant. (Adeel et al., 2009) Natural dyes can produce a variety of hues by mixing with other substances. The use of different mordants with the same dye may alter the wide range of hues, which is not possible with synthetic dyestuffs (Tasneem and Maria, 2016).

Palmyra (*Borassus flabellifer* L.) palm fruit has a bright yellow or orange mesocarp with a sweet, rich, juicy pulp and a pleasant smell. Carotenoids, a non-toxic natural colour with health-promoting properties, are abundant in ripe fruit. (Petchsomrit et al., 2021). The Aceraceae family, which includes plants from western Africa and Madagascar to eastern Indonesia and Papua New Guinea, consists of the Asian palmyra palm, also known by the scientific name *Borassus flabellifer* L. (Aman et al., 2018). The southern region of the Indian Union includes the states of Andhra Pradesh, West Bengal, Kerala, Tamil Nadu, Orissa, Gujarat, Bihar, Maharashtra, Madhya Pradesh, Karnataka, and Uttar Pradesh, which are likewise densely populated (Kumari et al., 2019). It serves as both the national tree of Cambodia and the official tree of Tamil Nadu. (Saidi et al., 2021)

In the present world, new pathogenic infections are developing with technological advancement. There have been reports of biological and curing actions in several portions of *Borassus flabellifer* Linn. Plants, including fruits' diuretic, antioxidant, anti-microbial, anti-tumour, anti-bacterial, wound-healing, and anti-malarial properties (Chitturi et al., 2018), Male inflorescence exhibits strong anti-inflammatory properties. (Paschapur MS et al., 2009). Due to its biological characteristics, *Borassus flabellifer* Linn. Could be used to create fabrics that are skin-friendly. The phytochemicals present in *Borassus flabellifer* Linn. fruit extract includes alkaloids, flavonoids, sterols, anthraquinones, proteins, carbohydrates, saponins, and cardiac glycosides.(Subramaniam et al.,2018)

Textile antioxidants can act as reservoirs, releasing antioxidants into the skin. Such textiles can scavenge free radicals produced by skin ageing and protect skin from oxidative stress and damage when in contact with the skin. Antibacterial and antioxidant fabrics can be used by sufferers of skin diseases, facial masks, anti-ageing products, and body care products.(Alonso et al., 2013; Mocanu et al., 2013) Bioactive molecules slowly release into the epidermal tissue when in direct contact with the skin. (Volpi et al., 2021)

Thus, this study aimed to examine the potential use of *Borassus flabellifer L.* extract for dyeing cotton fabric and optimise dye extraction and dyeing using the conventional method. The effectiveness of the antibacterial, antioxidant, and UV-Protecting properties is assessed.

Hence, the present study, "Eco-friendly natural dye from *Borassus flabellifer L.* fruit for coloration on cotton fabric," was designed with the following objectives:

- To extract dye from the *Borassus flabellifer L.* fruit
- To optimise dye extraction and dyeing conditions
- To evaluate the dyed fabric for physical and functional properties

2. REVIEW OF LITERATURE

2.1 Natural fibre- Cotton

2.1.1 History of cotton

2.1.2 Characteristic of cotton

2.1.3 Properties of cotton

2.1.4 Structure of cotton fibre

2.1.5 Advantage of cotton

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2.2.1 Introduction

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2.3.2 Alkali or acid extraction

2.3.3 Microwave and ultrasonic assisted extraction

2.3.4 Enzyme assisted extraction method

2.3.5 Solvent extraction

2.3.6 Super critical fluid extraction

2.4 *Borassus flabellifer* Linn

2.4.1 Introduction

2.4.2 Botanical description.

2.4.3 Properties and Medicinal uses

2.5 Mordant

2.5.1 Types of mordants

2.5.2 Application of mordants

2.5.3 Properties of mordant

2.1 Natural fibre – Cotton

Cotton is a soft, fluffy staple fibre that develops in a boll, or protective casing, around the seeds of cotton plants of the genus *Gossypium* in the mallow family *Malvaceae*. With a small amount of wax, fat, pectin, and water, cellulose makes up almost all of the fiber's composition. The cotton bolls will quicken the seeds' natural environment of dispersal.(Hu et al., 2019)

They are durable, dye-absorbing, abrasion- and temperature-resistant, as well as durable. Cotton fibres may be able to hold 24-27 times their own weight in water. Cotton creases, thus mixing polyester or applying a permanent finish gives clothing made of cotton the right qualities. Cotton fibres are often mixed with other fibres including nylon, linen, wool, and polyester to optimise each fiber's best properties.(Ravandi et al.,2011)

Cotton serves as the backbone of the world textile industry. It is also known as "White Gold" and "Fibre King." Because of its ability to spin, cotton, which is the most ancient textile fibre, is also the most important textile fibre. It is currently the most versatile and well-liked textile fibre. The most common textile material manufactured is cotton. It is now known that India was the first country to produce cotton. Natural cotton's ability to twist exceedingly tightly makes it possible to create strong fabrics. Cotton is known as a tropical fabric because it allows body heat to escape while also allowing the colder temperatures to infiltrate.(Priyanka et al., 2020)

Based on staple length and appearance, cotton fibres can be loosely divided into three big categories.

1. Long-staple cotton: This type of fibre is fine and lustrous, with a typical staple length of between 30 and 40mm, and it contains the highest-quality varieties, including Pima, Sea Island, and Egyptian cottons. Long-staple cottons are expensive and mostly used for fine textiles, yarns, and hosiery due to the difficulties of their cultivation and their small production.

2. Medium staple cotton: Cotton with medium staple length is widely available and uniform, such as American Upland, and has a staple length of approximately 25 to 33mm. 90% of the raw cotton fibre produced now in the world is made from medium-staple cotton. This fibre is commonly utilised in clothes, furnishings, and commercial goods.

3. Short-staple cotton: This coarse cotton, with fibres that range in length from 10 to 25 mm, is used only for very low-quality products since it is difficult to convert into textiles. (Yu, 2015).

Composition of cotton

The most widely used polymer in nature, cellulose, is found in its purest form in cotton fibres. The cellulose content of cotton fibres is close to 90%. To varying degrees, cellulose is present in all plants. About three-quarters of the cellulose found in plant stalk fibres like flax, jute, ramie, and kenaf is cellulose. Coniferous and deciduous wood both contain 40–50% cellulose, although other plant species or components do not. The cellulose found in cotton fibres is also the most crystalline, aligned, and fibrillar of all plant fibres, with the largest molecular weight. considering its abundance and structural order as the most abundant natural polymer, cotton is unsurprisingly regarded as a premium biomass and fibre.

Table I

Chemical composition of cotton fibre

Natural impurities

| Constituent | % |
|------------------|-----|
| Cellulose | 88 |
| Oils and Waxes | 0.5 |
| Pectins | 0.7 |
| Proteins | 1.1 |
| Colouring matter | 0.5 |
| Mineral Matter | 1.0 |
| Moisture | 8.0 |

2.1.1History of Cotton

In Mexico, remnants of cotton textile and cotton bolls dating back 7,000 years have been discovered. Three thousand years ago, cotton was being farmed, spun, and fashioned into textile in the Pakistani Indus River Valley. Natives of Egypt's Nile Valley began producing and

using cotton clothing around the same time. Around 800 A.D., Arab traders introduced cotton cloth to Europe. By the year 1500, cotton was well-known on a global scale. In 1730, cotton was mechanically spun for the first time in England. Cotton occupies a significant position in the world as a result of the industrial revolution in England and the development of the cotton gin in the United States (Gillow, 2004). In the 1690s, the East India Company brought cotton to Britain. The British Midlands became a particularly successful manufacturing region in the 1770s thanks to new inventions including the spinning mule, the water frame, and the spinning jenny as well as the industrial revolution (Prasad, 2018). Eli Whitney's development of the cotton gin in 1793 marks the beginning of the American cotton industry's expansion. The majority of the cotton grown worldwide by the early 1830s was produced in the United States (Philips and Lakwete, 2004). About 25 million tonnes or 110 million bales are currently estimated to be produced annually, which equates to 2.5% of the world's arable land. The largest producer of cotton in the world is India. For many years, the United States has been the world's top exporter (Deshmukh and Mohanty, 2016).

2.1.2 Characteristics of cotton

Softness -Cotton fabric tends to retain the soft feel of the cotton plant, which is a soft and fluffy plant.

Durability- Cotton fabrics are durable and wear-and-tear resistant attributed to the cotton plant's solid cellular structure.

Absorbency- Cotton fabric has a lot of space between its threads, which makes it incredibly absorbent.

Dye uptake- Cotton absorbs colour readily and can be dyed a wide range of hues due to its absorbent nature.

Breathability- Cotton is more breathable than synthetic fibres due to the structure of its fibres.

2.1.3 Properties of cotton

The features that easily differentiate cotton fibre from all other textile fibres depend on the molecular structure of the fibre and the conditions influencing fibre development.

Each significant fibre characteristic is categorised into the appropriate groupings (Steadman, 1997; Hunter, 1998).

- Properties according to Length
 1. Staple Length Values
 2. Span Length Values
 3. Uniformity
 4. Parameters for Length Distribution
 5. Short Fiber Content

- Transverse Dimensions of Cotton
 1. Micronaire
 2. Fineness
 3. Maturity

- Tensile Properties
 1. Strength
 2. Breaking Elongation.

- Non-Lint Content
 1. Average Trash
 2. Trash Particle
 3. Size Distribution
 4. Trash Type

- Dust
 1. Level and Size
 2. Seed Coat Fragments
 3. Foreign Matter and Contaminants

4. Neps

- Cotton Colour (Rathore, 2022).
- Miscellaneous properties of cotton
 1. Versatility
 2. Wrinkling
 3. Absorbency
 4. Sinkage

Physical properties of cotton

Tenacity-Long polymers, numerous, consistent hydrogen bond formations between adjacent polymers, and spiral filaments in the primary and secondary cell walls all contribute to cotton's enhanced durability. One of the few fibres that becomes stronger when wet, owing to better arrangement in the amorphous part of the fibre.

Elastic- Plastic nature- Because of the crystalline polymer structure that gives cotton fibre its inelastic nature, cotton fibres easily wrinkle and crease. The long lengths of the polymer molecules and the many hydrogen bonds that link them together prohibit the polymer molecules from sliding past each other without exerting an enormous amount of force. Since polymers are difficult to bend and crush due to their crystallinity, strong strain forces can cause them to fracture. Due to these polymer fractures, the polymer system develops weak points, which in turn causes weak spots in the fibre structure. Therefore, when force is applied, cotton fibres become creased and wrinkled.

Hygroscopic nature-This huge polar -OH group on the cotton molecule, which draws water molecules to it, makes it an extremely effective moisture absorber. However, due to the extremely small intermolecular spaces in the crystalline region, the OH molecule can only enter the cotton molecule through the amorphous region. Cotton fibre swells when exposed to water in large part because water molecules in the amorphous area separate polymers. As it absorbs moisture from the finger's skin, cotton fibres typically feel crisp when touched. Cotton materials don't generate static electricity because of the fiber's hygroscopic nature. The

hydroxyl group in the polymer attracts the polarity of the water molecule, dissipating static charges as a result.

Thermal properties-Cotton fiber's capacity to conduct heat energy reduces any potentially harmful heat accumulation. Cotton fibres may therefore resist the high ironing temperatures. However, the fact that cotton fibre chars and burns when heated too much proves that it is not thermoplastic. This inhibits the fibre from shifting into the polymers' new position when heat is applied. On heating, the polymers start to vibrate and eventually fall apart.(Chatterjee, 2021).

Chemical Properties of Cotton Fibre

The following list summarises the primary chemical features of cotton fibre:

1. Action with alkali: Alkalis have no effect on cotton fibres because there is no attraction between the polymer and the alkalis. Mercerization without tensioning or idle mercerization results in swelling of cotton fibres, which is mainly caused by alkali and radicles entering the amorphous region of the fibre polymer system and forcing the polymers wide apart, which increases the viscosity of poorly oriented polymers.

2. Acid action: Intense acid damages fibres. The fibre is harmed by concentrated sulfuric and hydrochloric acids. However, the fibre is not harmed by weak acid.

3. Bleaching action: sodium hypochlorite and sodium perborate are commonly used for bleaching agent. When cotton is bleached with powerful oxidising agents, oxi-cellulose is generated.

4. Organic solvent action: Here, resistance is feasible so dry.

5. Sunlight prevention potential: Ultraviolet rays turn cotton into oxi-cellulose.

6. Mildew Prevention potential: There is a chance of being impacted.

2.1.4 Structure of cotton fibre

The multilayered structure of cotton fibres has been investigated for over a century. The cuticle, which is the outermost covering of the cotton fiber's primary cell wall, has a significant impact on the fiber's characteristics, processing, and application.⁸ A primary wall, secondary wall, and lumen make up the fibrillar structure of cotton, the characteristic elements of dry mature cotton

fibres are depicted. The outer layers of cotton fibre are where the majority of non-cellulosic elements can be found.

Each cotton fibre is a single, complete cell that forms in the cottonseed's surface layer. A cuticle, an outer wall, a secondary wall, and a central core or lumen make up cotton fibres. The cuticle is the cotton fiber's "extreme outer" or "skin." It is made out of a cotton wax-like layer. The nutrients for the plant's growth are provided by the lumen, a hollow canal that flows the length of the fibre. The lumen's size varies greatly depending on how mature the fibre appears.

Immature fibres have a very thin wall structure and a wide lumen, but mature fibres have a thick layer of cellulose in the secondary wall that results in a relatively narrow lumen. When seen longitudinally, cotton fibre has a ribbon-like appearance with twists spaced regularly along its length. The twists, which are also known as convolutions, provide cotton fibres an uneven surface that improves inter fibre friction and makes it possible to spin fine cotton yarns with sufficient strength.

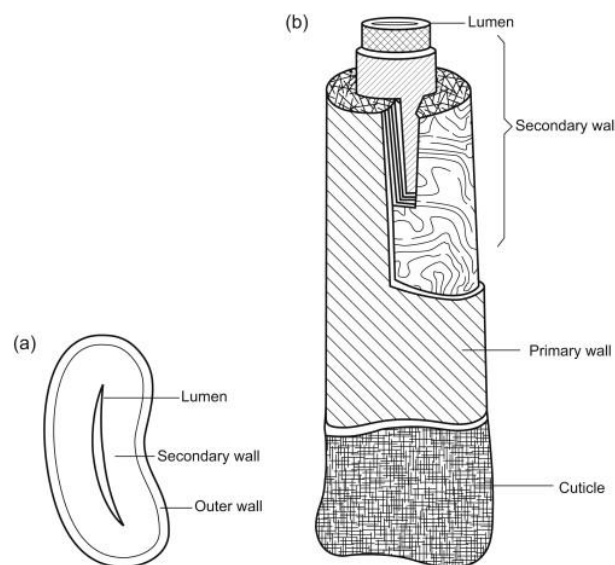


Figure 1 - Cross sectional view and vertical view of cotton fibre

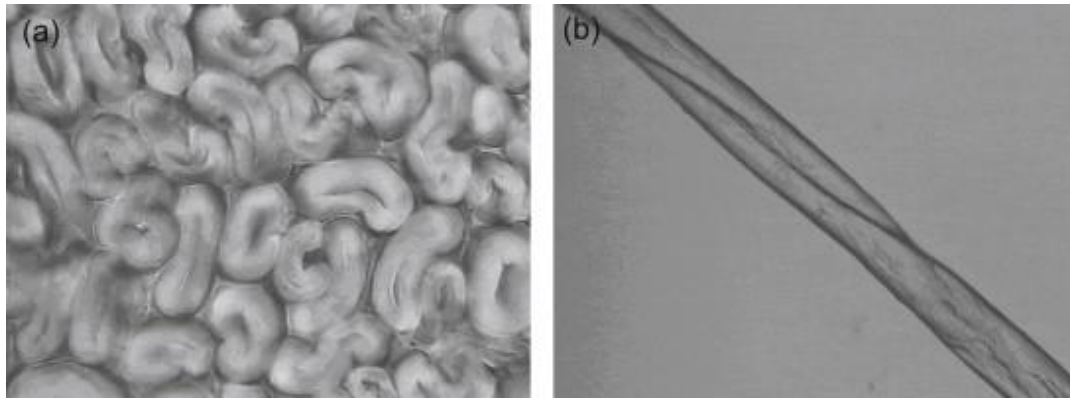


Figure 2 - Microscopic view of cotton fibre

2.1.5 Advantages of cotton

1. Cotton is inexpensive fabrics.
2. It has good absorbency
3. Cotton has high breathable property
4. High lustrous property
5. Comfortable to wear
6. Cotton fabric can withstand to wear and tear
7. Cotton fibre has greater resistance to alkali
8. It has good heat resistance.

2.2 Natural dye

2.2.1 Introduction

Since ancient times, natural dyes have been employed for a variety of tasks, but textile dyeing has been their primary application. Almost anything can be used to create natural colours, including minerals, plants, and even some insects. According to Shiva Kumar V., plants' roots, bark, leaves, flowers, skins, and shells contain the majority of natural dyeing pigments. Natural dyes are environmentally friendly, biodegradable, extracted under mild reaction conditions, and have no disposal issues because of this. They maintain ecological balance and don't cause any environmental issues during use or production.

Plant dye

The earliest dyes were of plant origin and were found by accidentally colouring clothing with fruit or plant liquids. Plants can produce vegetable dyes in a variety of ways, including through the production of leaves, flowers, fruits, pods, bark, etc. These vegetable dyes can be used either directly or in conjunction with other mordants.

Henna: The dried leaves of the Henna plant, *Lawsonia inermis*, are used to make the dye. It results in orange-yellow colour. It works well for dyeing silk and woollen fibres.

Indigo: Blue dye indigo is referred to as the "king of all-natural dyestuffs." It gives off a blue tint. It is derived from *Indigofera tinctoria*, a leguminous plant, and its leaves. It works well for dyeing wool and cotton.

Indian Madder: It causes various colours of red to appear on textiles. Fabrics made of cotton and wool can be dyed with it. It is extracted out of *Rubia tinctoria* roots.

Turmeric: It causes various yellow hues to appear on textiles. It works well for dyeing wool, silk, and cotton. The turmeric plant's ground root (rhizome) of the turmeric plant (*Curcuma longa*) is used to make the yellow colour.

Marigold: It is made from the flower of the *calendula officinalis* plant, which has yellow or orange petals. It works well for colouring wool and silk fibres.

Tea: Tea leaves (*Camellia sinensis*) or tea powder are employed to extract the dye. The tea leaves (*Camellia sinensis*) or tea powder are employed. It generates various brown hues.

Onion (*Allium cepa*): The onion's outermost skin or peel is used to extract the colour. The onion skins, if dried properly

Senegalia catechu: The resin is used to extract the dye. The plant material that comes from acacia trees. Brown hues are produced.

Old fustic, also known as yellowwood, is made from the heartwood of the dyer's mulberry, a sizable tropical American tree (*Chlorophora tinctoria*, or *Maclura tinctoria*), which belongs to the mulberry family, *Moraceae*. On wool that has been mordanted (fixed) with chromium salts, the dye causes yellowing.

Logwood: This term may also be used to describe plants in the willow family's genus *Xylosma*. The dye, which produces the colour black, is taken from the heart of the logwood tree.

The spice known as "saffron crocus" is made from the bloom of the *Crocus sativus* plant.

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2.2.2 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.

All natural textile fibres can be dyed using direct dyes for cotton, although acid dyes can often only be used on wool and silk. Tannic acid-treated cotton can also be dyed with basic dyes. On the other hand, mordant dyes can be used on both vegetable and animal fibres. The essential mordant dyes are cochineal, madder, and logwood. Vat dyes are a different category of dyes that are insoluble in water. These include Tyrian purple, woad, and indigo (Patel, 2011).

Vat dyes: Indigo is a water-insoluble dye that is first dissolved in water before use. With the use of sodium hydroxide and sodium hydrosulphite, natural indigo is solubilized. It is applied

to cellulosic fibre after being solubilized, and after dyeing, the colour is developed by oxidising with hydrogen peroxide. The indigoid class of vat dyes is represented by indigo dye.

Direct dyes: The direct dyeing process can be used with natural dyes that are water soluble, have a long and planar molecular structure, and contain conjugated (single and double bonds) linkages. The dye molecules may have sulphonic, amino, and hydroxyl groups. The direct dyeing process can be employed to use annatto, harda, pomegranate peel, and turmeric. It is possible to improve dye exhaustion by using table salt. At 100°C, the dyeing temperature is maintained.

Acid dyes: The structure of the dye molecules includes sulphonic or carboxylic groups, which provide an affinity for wool and silk fibre. The pH range for the dyeing is acidic (4.5–5.5). Tannic acid is used to increase fastness after dyeing. The acid dyeing procedure is used to colour wool and silk with saffron. The levelling effect of common salt in the dye bath is produced.

Basic dyes: When dissolved in water with an acidic pH, the dye molecules release coloured cations. The -NH₂ groups in the dye molecules react with the -COOH groups in wool and silk. Acetic acid is added to the dye bath to maintain a pH of 4-5. (Gupta, 2019)

2.2.3 Classification based on Colour

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

Yellow dyes

There are a total of 28 natural yellow dyes listed in the Colour Index; yellow is of particular significance in India, where it is still regarded as an auspicious colour with significant religious significance (Patel, 2011). Yellow is perhaps the most abundant hue in nature and represents growth and happiness.

Red dyes

There are 32 red natural dyes listed in the Colour Index. The majority of the red pigments are concealed in the bodies of the dull-gray insects or in the roots or barks of plants. Madder (Rubiactinctorum), manjistha (Rubiactordifolia), Brazil wood/sappanwood (Caesalpineasappan), Al or morinda (Morindacitrifolia), cochineal (Coccusacti), and lac dye

(Coccinellidae) are a few of the group's most well-known members (Yusaf et al., 2017; Vankar, 2000).

Blue dye

The Colour Index lists only four blue natural dyes, viz: natural indigo, sulphonated natural indigo, Kumbh (Manipur), and the blossoms of the Japanese 'Tsuykusa' plant, which are mostly used to make awobana paper (Sinha et al., 2012). Indigo gives all fibres the blue colours that are the brightest and change the fastest. Indigo (*Indigofera tinctoria*) and woad (*Isatis tinctoria*), which are the primary sources of indigotin, are the main sources of colour (Patel, 2011). These colours have great washability on cotton. Using functional UV absorbers, gardenia blue's photo-stabilization may be induced (Oda, 2012; Oda et al., 2013).

Black dye

Because it was found by the Spaniards on the Bay of Campeche in Mexico, Logwood (*Haematoxylum campechianum*), sometimes known as Cam peachy wood, is a significant source of natural black dye. Even now, it is still employed to dye silk in rich hues using an iron tannate mordant. On the majority of natural and synthetic fibres, it also provides outstanding depth and fastness. Additional significant sources of black dyes include tannins. The hydrolyzable tannic flavogallol found in pomegranate rind reacts with iron salts to produce deep blacks.

Brown dyes

Naphthoquinones and anthraquinones, which are quinone-based pigments, are the main sources of natural brown colours. Commonly used as mordants, copper and iron salts have a tendency to transform colours into deep, drab colours, especially browns. One such natural dye, called catch, uses ions of copper and chromium to create deep brown hues. This colour is a flavonoid with a tannin basis that comes from wild *Acacia arabica* and *Acacia catechu*.

Green dye

Green dye-producing plants are quite rare. Since ancient times, woad (*Isatis tinctoria*) and indigo have both been combined with yellow dyes to create green hues. Woollen fabric that had been dyed yellow with dyer's green weed and mordanted with alum was then overdyed with woad and later with indigo to create the formerly well-known Kendal green. When iron

mordant is applied to fabrics that have been coloured yellow, soft olive greens are also produced (Mansour, 2018).

Orange dyes

Orange dyes can be produced by the same dyes that produce reds and yellows. Barberry, annatto, sweet pepper blood roots, and other natural sources are used to make orange dye (Tayade et al., 2013; Yusaf et al., 2017).

2.2.4 Classification based on Chemical constitution

The key phytochemical components, such as flavonoids, anthocyanins, tannins, carbohydrates, alkaloids, cardiac glycosides, steroids, phenols, anthroquinone, leucoanthocyanin, diterpenes and saponins, were present in plant extract.

2.2.5 Advantages of natural dye

The use of natural dyes has become more popular in recent years. The main reason for this is that natural colourants outperform synthetic dyes in a number of ways.

1. Natural dyes usually provide hues that are relaxing, glossy, and pleasant to the eye.
2. Natural dyes are frequently renewable since they are agro-renewable, biodegradable, and skin-friendly. They may also have health benefits for the wearer compared to fundamental raw materials for synthetic colours, which are non-renewable.
3. In other circumstances, the waste produced during the extraction and dyeing process of natural plants like harda and indigo is ideal fertiliser for use in agricultural areas. This natural waste can therefore not be disposed of.
- 4- It is safe for skin contact and typically not harmful to human health because some of its constituents are anti-allergens.
- 5- Very few or no chemical interactions take place when dye is being prepared.
- 6- Subsequent research demonstrates that they might also be used to colour the majority of synthetic textiles.

2.2.6 Disadvantage of Natural dye

Despite these advantages, there are also significant drawbacks to natural colours. The entire use of natural dyes in the textile industry is just about 1% due to the technical and environmental difficulties involved in their production and use. These constraints and difficulties are as follows:-

- 1 Using natural dyes can be challenging since they differ from location to place, species to species, phase of maturity to species, and crop season to crop season.
2. It is challenging to create a recipe for utilising natural dyes since the materials, as well as the colour component, as well as the colour component, affects the natural dyeing process and colour development.
3. The precise technical information of extraction and colouring techniques is not readily available.
- 4- Only a small number of coloured fabrics have good light and washing fastness, and many coloured textiles can change colour when exposed to sunshine, perspiration, and air.
- 5 Almost all natural dyes, with a few exceptions, require the use of mordants to bind to the textile substrate. While dyeing, a sizable amount of mordant remains unused in the leftover dye bath, creating a significant barrier for the removal of effluent.
6. Natural dyes may need to be used in greater quantities than synthetic dyes in order to colour a certain amount of fabric. For instance, five grammes of synthetic dye can colour one gramme of cotton, whereas 230 grammes of natural dye are needed to colour the same amount of fabric. Natural hues are therefore more expensive than manufactured dyes.

2.3 Extraction of natural dyes

Eco-friendly and biodegradable materials have drawn a lot of interest in recent years all around the world. For instance, natural dyes and colours are now widely accepted as a viable alternative to synthetic dyes in the production of textiles as well as in the colouring of foods, cosmetics, and pharmaceutical products.

Natural items only occasionally include very small levels of natural colour. The use of diverse plant extracts to lengthen the freshness of various fruits and vegetables has excited the curiosity of researchers. The first step in the process of natural dyeing is the extraction of the colourant.

Extraction is the process of separating the desired colour component from the plant into a solvent medium by physically or chemically rupturing the plant's cell wall.

One of the most important steps in the treatment of textiles to get the desired dyeing qualities may be the extraction of dyes from natural sources. For a certain natural source, standardising the extraction process and maximising the extraction variables is also economically relevant and affects the cost of the finished products. Aqueous methanol, ethanol, or water are used to extract many bioactive substances.

Due to the fact that natural colouring agents do not exist in a single chemical form and that the plant matrix also contains a variety of non-dye plant substances, the extraction of natural dyes is also a challenging process.

The composition and solubility characteristics of the colouring components must be well established before performing an extraction process. They need to employ a unique procedure to extract the dye from its source.

The methods listed below can be used to extract natural colours from their source materials. For example, enzymatic, supercritical fluid, aqueous, solvent, and ultrasonic extraction methods are currently the subject of extensive research due to their greater efficacy to more traditional methods.

2.3.1 Aqueous extraction method

Traditional techniques for extracting plants and other materials include aqueous extraction. employs water for extraction, either with or without salt, acid, alkali, or alcohol added to the extraction bath. Colours were typically extracted from plants and other materials via aqueous extraction.

The dye-containing substance is first broken down into little bits or powdered, then filtered to improve extraction efficiency. After being submerged in water in metal containers for a long time—typically overnight to dissolve the cell structure, it is then heated to extract the dye solution, which is subsequently filtered to eliminate any remaining non-dye plant material. The process of boiling and filtration is repeated to get rid of as much colour as possible. Filtering the extraction fluid using a standard filtration technique, followed by solvent evaporation,

washing, and lastly drying to obtain the completely natural colour, can be used to calculate the colour yield.

The drawback of this extraction method is that some of the dye breaks down during the boiling phase. Only colours that do not degrade at boiling temperatures are therefore suitable for this process. The molecules ought to be water soluble. Since only the colour components that are soluble in water are extracted—and many dyes have low water solubility—there is a lengthy extraction period, a significant volume of water must be used, high temperatures are used, and the dye yield is limited.

2.3.2 Acid or Alkali extraction

Alkaline or acidic media are used for dye extraction. For this, it is also possible to employ diluted acidic or alkaline solutions, which aid in the hydrolysis of glycosides and improve extraction. Tesu natural dye is extracted from tesu flowers using an acidic process. For colour extraction involving phenolic groups, an alkaline media is required. Colour extraction from lac insect, annatto seeds, safflower petals are extracted by this method (Chungkarg et al., 2021)

2.3.3 Microwave and ultrasonic assisted extraction

Actually, microwave- and ultrasound-assisted extraction methods are ones in which the use of ultrasound or microwaves improves extraction efficiency, reducing the amount of solvent used, extraction time, and extraction temperature. Very small bubbles or cavitations emerge in the liquid when the natural dye made from plant components is handled with water or any other solvent while ultrasound is present. These enlarge, but once a certain size is reached, they lose their ability to maintain their shape. High temperature and pressure are produced as a result of the cavity collapsing or the bubbles bursting. These bubbles form in their millions and burst every second. The extraction efficiency is quickly increased when extremely high temperatures and pressures are created. Additionally, because the procedure may be carried out at lower temperatures, it is better to extract heat-sensitive dye molecules. Many researchers have lately reported using this extraction technique as research into novel dye sources and efforts to optimise the dye extraction process continues.

In microwave extraction, the natural sources are processed in the presence of microwave energy sources with the least amount of solvent possible. The processes are accelerated by the microwave, allowing for faster and more effective extraction. According to Sinha et al., microwave energy was used to remove the annatto colourant. Their team had previously

described the butterfly pea's blue pigment being extracted with the help of a microwave. Due to the lower energy consumption from reduced extraction temperatures, solvent usage, and duration, microwave and ultrasound extractions can be regarded as green procedures (Mansour, 2018).

Ultrasound-assisted extraction (UAE)

Ultrasound-assisted extraction (UAE) is a method that has the potential to be helpful because it does not require the use of expensive or difficult instruments. For colourant extraction, mordanting, and coloration treatments, ultrasonic energy offered a quick and effective solution. In contrast to conventional methods. These extraction techniques use microwaves or ultrasound to increase extraction efficiency. This reduces the amount of solvent needed, the extraction time, and the extraction temperature, and has been shown to be environmentally benign.

2.3.4 Enzyme assisted extraction method

Enzymes have garnered a lot of attention recently for their potential to be used to extract beneficial natural compounds from plants.

The production and colouring of cotton, wool, and other material filaments with natural dyes utilising various types of enzymes has been approached in a variety of ways. They determined that the extraction of natural colourants was a result of enzymatic treatment. There is an increase in colourant absorption in every circumstance.

By dissolving the plant's cell walls and membrane and improving the extraction of active chemicals, the enzymatic extraction approach can be used to increase extraction efficiency under mild conditions.

This technique may be useful for removing colourants from hard plant components like bark, roots, and so forth. It is a practical strategy for separating dyes from the studied material because it has a smaller impact on the environment and significantly reduces extraction time as compared to the conventional approach. Additionally, this method can reduce the amount of energy used as well as the amount of solvent used. Enzymatic-assisted extraction has been suggested as an alternative method of extracting the natural product as a consequence.

2.3.5 Solvent extraction

Depending on their nature, natural colouring elements can also be extracted using organic solvents like acetone, petroleum ether, chloroform, ethanol, methanol, or mixtures of solvents like ethanol and methanol, water and alcohol, etc. The water/alcohol extraction method can be used to extract both water-soluble and water-insoluble chemicals from plant resources. Solvent extraction calls for the use of solvents like alcohol, hexane, or benzene as well as the right extraction tools, like the Soxhlet extractor. The collection of glycosides and colour bodies is improved by the use of an alcohol solvent, acid, or alkali. It is easier to purify extracted pigments, remove solvents using distillation, and recycle. In general, ethanol is used because it produces good-quality dyes, requires little water, and allows for lower-temperature extraction.

As a result, because more chemicals and colouring components may be extracted than with the aqueous method, the extraction yield is higher. Toxic residues and the impact of greenhouse gases are the method's drawbacks. When waxy chemicals and chlorophyll are removed simultaneously, issues arise.

2.3.6 Supercritical fluid extraction (SFE)

Specific compounds from plants can be extracted via supercritical fluid extraction (SFE) at temperatures close to ambient, avoiding thermal denaturation of the material. SFE technology is well known for being an effective analytical strategy comparable to conventional chemical analysis techniques. The qualitative and quantitative study of natural product constituents is also adequately catered for by its design.

The procedure has the benefit of producing an extract free of heavy metals, residual solvent traces, and other contaminants. It also produces an extract with a light colour because polar polymerizing agents are not used in the process. Despite the fact that SFE is an old method of solvent extraction, its commercial use takes a very long time because of the complex and expensive high-pressure machinery and technologies needed, as well as the inefficiency of polar compound extraction.

2.4 *Borassus flabellifer* Linn.

2.4.1 Introduction

Palmyra (*Borassus flabellifer* L.) palm fruit has a bright yellow or orange mesocarp with a sweet, rich, juicy pulp and a pleasant smell. Carotenoids, a non-toxic natural colour with health-promoting properties, are abundant in ripe fruit. (Petchsomrit et al., 2021). The Aceraceae family, which includes plants from western Africa and Madagascar to eastern Indonesia and Papua New Guinea, consists of the Asian palmyra palm, also known by the scientific name *Borassus flabellifer* L. (Aman. et al., 2018). The southern region of the Indian Union includes the states of Andhra Pradesh, West Bengal, Kerala, Tamil Nadu, Orissa, Gujarat, Bihar, Maharashtra, Madhya Pradesh, Karnataka, and Uttar Pradesh, which are likewise densely populated (Kumari et al., 1987). It serves as both the national tree of Cambodia and the official tree of Tamil Nadu. (Saidi et al., 2021)

There have been reports of biological and curing actions in several portions of *Borassus flabellifer* Linn. Plants, including fruits' diuretic, antioxidant, anti-microbial, anti-tumour, anti-bacterial, wound-healing, and anti-malarial properties (Chitturi et al., 2018), Male inflorescence exhibits strong anti-inflammatory properties. (Paschapur et al., 2009). Due to its biological characteristics, *Borassus flabellifer* Linn. Could be used to create fabrics that are skin-friendly.



Figure -3 *Borassus flabellifer* Linn fruit

2.4.2 Botanical description

Kingdom: Plantae

Order: [Arecales](#)

Family: [Arecaceae](#)

Genus: *Borassus*

Species: *B.flabellifer*

Common names: **doub palm, palmyra palm, panai maram, toddy palm, wine palm, ice apple**

2.4.3 Uses of *Borassus flabellifer* Linn

➤ green synthesis of copper and silver nanomaterials using palmyra fruit pulp, and it was evaluated for anti-tumor, antimicrobial, and antioxidant activity .

➤ This, on the other hand, produced excellent results against prostate cancer cell lines and bacteria such as pseudomonas, Escherichia coli, Staphylococcus aureus, and Klebsiella pneumoniae. Furthermore, copper-cobalt bimetallic nanoparticles were synthesized using palmyra tree fruit extract.

➤ This nanomaterial was extremely effective as a mosquito repellent against the Culex mosquito. This pesticide is safer, more common, and less hazardous to the environment.

➤ In addition it is used for different ailments, for instance; its bark is used to make mouthwashes and dentifrice, palm sugar helps to reduce the inflammation and it reduces the obesity and diabetics, spadix of the palmyra reduces the heart burn and toddy is used to reduce the ulcers.

➤ However, because of its distinct physical, chemical, and biological properties, it is used as an anti-microbial in food technology, cosmetics , biotechnology, textiles, and as biosensors and nutrient carriers.

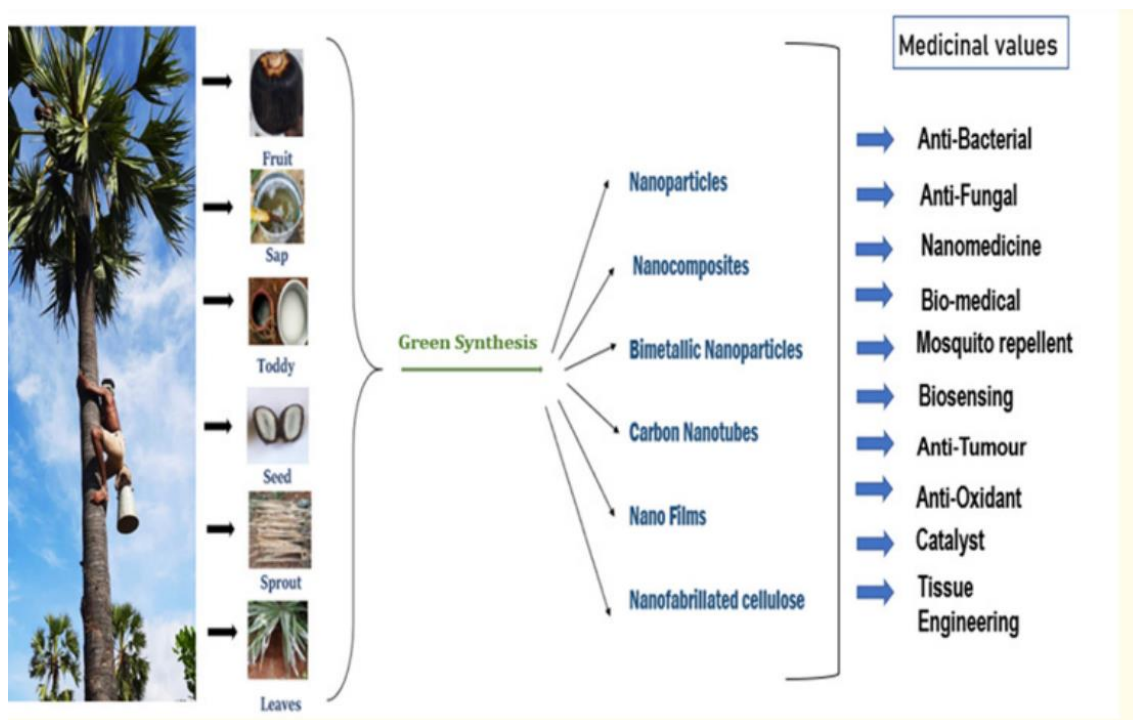


Figure- 4 Medical properties of Palmyra palm

2.5 Mordants

The Latin word "mordere" (which means "to bite") is where the word "mordant" originates. The mordant which acts as a bridging group between the natural dye and the fabric. Mordants have an impact to enhance physical qualities including colourfastness, dye brightness, and rubbing fastness. In the absence of a mordant, colour fastness is poor and the shade range is restricted. The use of mordant in dyeing not only increases dye take-up and colourfastness, but the use of various mordants on a natural dye can result in diverse colours and hues. The mordant which acts as a bridging group between the natural dye and the fabric.

2.5.1 Types of mordanting

Mordants are basically three types -metallic mordants such as (i) metal salts of aluminum, copper, tin 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; Kareem and Omar, 2012). Generally, on the basis of origin mordants are two types- Synthetic and Natural mordants. 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, lemon peel, pomegranate rind used as natural mordants.

2.5.2 Application of mordants

The three methods used for mordanting are:

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

Pomegranate rind powder

Pomegranate peels are the leathery skins and seeds of pomegranate fruit and although they are not normally eaten, are high in tannin and super useful as a natural dye. Pomegranate (*Punica granatum*), is also known as Anaar in India and Granada in Spain. It grows wild in India, Italy, North Africa, and China and is used in Ayurvedic remedies. Pomegranate is an aromatic dye that yields a green-yellow colour and shifts to olive and dark Gray with iron. Pomegranate is used as both a tannin-rich mordant in India, and also as a dye. Dried peels are crushed and powder form is used to extract the dye solution (<https://botanicalcolors.com>).

Guava leaf powder

The guava (*Psidium guajava*) tree, a member of the Myrtaceae family, is a particularly special and traditional plant that is farmed for its numerous medicinal and nutritional benefits. *Psidium Guajava* is a naturally occurring plant source creating brilliant brown colours. It is a non-toxic, non-allergenic, biodegradable, cost-effective, and widely available substance. Guava has been farmed and used as an essential fruit in tropical places such as India, Indonesia, and Malaysia. Guava leaves are dark green, circular, and oval, with an oblique tip. the guava leaf with a dorsal view on the left and a ventral view on the right.

2.5.3 Properties of mordants

- Mordant dyes have no affinity for textile fibres.
- They are attached to the fibres with the help of mordants.
- Mordant has 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 is soluble in cold water.
- Wide range of hues can be produced from mordant dyes
(<http://textileengg.blogspot.com/2015>)

3. METHODOLOGY

Experimental process for the study on “Eco-friendly natural dye from *Borassus flabellifer* L. fruit for coloration on cotton fabric,” is discussed the following headings:

3.1 Flowchart of the study

3.2 Selection of fabric

3.3 Pre- treatment of fabric

3.3.1 Desizing

3.4 Selection of dye source

3.5 Extraction of dye source

3.6 Optimisation of dye extraction conditions

3.6.1 Optimisation of Solvent

3.6.2 Optimisation of Dye source concentration

3.6.3 Optimisation of extraction Time

3.6.4 Optimisation of extraction Temperature

3.7 Characterisation of the dye extract

3.7.1 UV- Vis Analysis

3.7.2 Phytochemical Analysis

3.7.3 % Yield of natural colourant

3.7.4 FT-IR

3.7.5 Antioxidant analysis

3.8 Optimisation of dyeing conditions

3.8.1 Optimisation of Dyeing Time

3.8.2 Optimisation of dyeing Temperature

3.8.3 Optimisation of dyeing Material Liquor ratio

3.9 Selection of mordant

3.9.1 Extraction of mordanting

3.9.2 Mordanting Method

3.10 Dyeing under optimized conditions

3.11 Fabric evaluation

3.11.1 Fabric weight

3.11.2 Fabric thickness

3.11.3 Fabric stiffness

3.11.4 Absorbance

3.11.4.1 Sinking

3.11.4.2 Wicking

3.11.5 Colour Fastness

3.11.5.1 Fastness to washing

3.11.5.2 Fastness to crocking

3.12 Assessment of functional properties

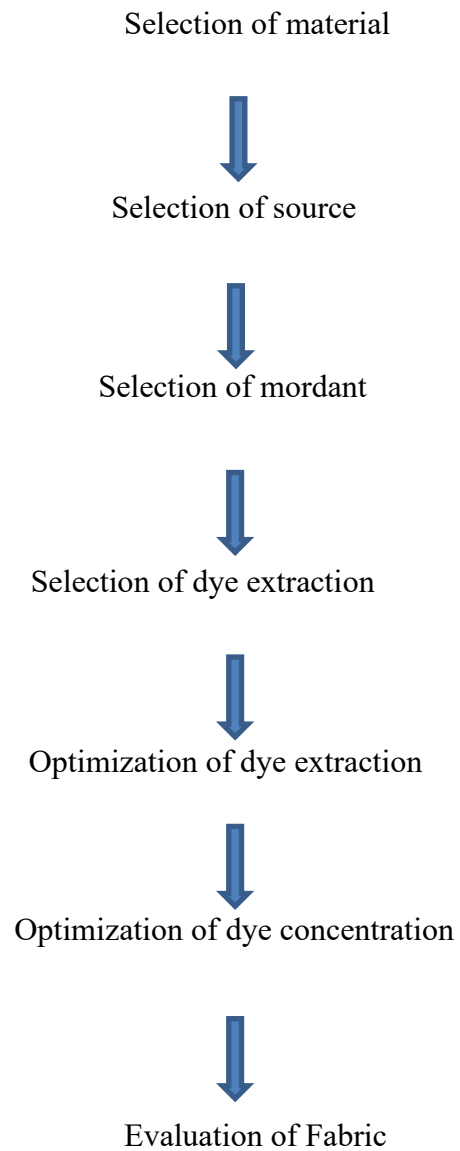
3.12.1 UPF analysis

3.12.2 Antibacterial analysis

3. METHODOLOGY

3.1 Flowchart of the study

The flowchart explains the methodology followed in this study under following heads



3.2 Selection of fabric

A plain weave of desized, scoured, and bleached cotton fabric 138 g/m²· 58 ends per inch and 43 picks per inch samples were purchased from Murugan Mills, National Textile Corporation (NTC), Coimbatore, India. Before use, the cotton fabric was treated with a solution containing 3 g/L detergent at 100⁰C for 1 hour. Finally, the fabric was rinsed with water and dried.

3.3 Pretreatment of fabric

Cotton fabrics contain some inherent impurities such as oil, wax, and pectin within their structure. It helps to improve the aesthetic appearance and processability of fibre to fabric.

3.3.1 Desizing

To prevent breakages during the weaving process, sizing chemicals (such as starch, polyvinyl alcohol, etc.) are usually applied to the warp strands. The woven fabric also takes up certain contaminations from machine oil and grease during the fabric formation process, and as a result, all of these contaminants provide the fabric its hydrophobic properties. In order to prepare the textile substrate for further colouring or finishing steps, pretreatment procedures for cotton fabrics are essential. During the pretreatment, the fabric is cleaned, made absorbent, and substantially whitened through the de-sizing process (Shore, 1998; Ibrahim et al., 2004)

RECIPE:

ML Ratio: 1:30

Detergent – 3%

Soap oil – 2%

Temperature – 100° C

Time – 1hr

3.4 Selection of dye source

The dye source selected for this study was *Borassus flabellifer L.*, which is commonly known as Palmyra palm fruit and has a bright yellow colour. *Borassus flabellifer Linn* Fruits were collected from Karur, Tamil Nadu. There have been reports of biological and curing actions in several portions of *Borassus flabellifer Linn*. Plants, including fruits' diuretic, antioxidant, anti-microbial, anti-tumour, anti-bacterial, wound-healing, and anti-malarial properties (Chitturi et al., 2018), for this present study, the fruits were processed by removing the seeds and black husk. The pulp is gathered and air-dried under the sun for 2-3 days. Then the dried fruit is blended in a blender.

3.5 Extraction of dye source

The extraction process was carried out using aqueous method. Natural dye was extracted by boiling 5 g of powdered *Borassus flabellifer Linn*. Fruits in 100 ml of water for 1 hour at 100°C. The dye extract was filtered and stored in refrigerator.



Plate 1- Desizing of fabric



Plate 2 – *Borassus flabellifer* L. Fruit



Plate 3- *Borassus flabellifer* L. Fruit extract



Plate 4- Pomegranate rind Powder



Plate 5- Guava Leaf powder

3.6 Optimization of dye extraction conditions

Natural dye extraction from *Borassus flabellifer* Linn Fruit powder was optimised for various parameters.

3.6.1 Selection of Solvent

To determine a suitable solvent for dye extraction, dye was extracted using water, sodium hydroxide (1%), and ethanol (80:20). Dye was extracted using 6% of the dye source, and the extracted dye was used to dye fabric individually. Dyed fabric was examined for colour strength.

3.6.2 Optimization of Dye source concentration

To determine the optimum dye source concentration, dye was extracted using different dye source concentrations, such as 2 g, 4g, 6g, 8 g, and 10 g. The concentration that shows the maximum yield is selected as the optimum condition for the extraction of the dye source.

3.6.3 Optimization of extraction Time

To find the optimum time for dye extraction, dye was extracted at various time intervals, such as 15, 30, 45, 60, and 75 min. The impact of extraction time is assessed. The extraction time that shows maximum colour yield was chosen as the optimum time for the extraction of dye.

3.6.4 Optimisation of extraction Temperature

To find the suitable temperature for the extraction of dye, the temperature was altered (30°C, 50°C, 70°C, 90°C, and 100°C) at a fixed time and dye source concentration. The colour yield was analysed for extraction, which shows the maximum yield, and was selected as the optimum temperature for dye extraction.

3.7 Characterisation of the dye extract

3.7.1 UV-Visible analysis

UV-VIS spectrometry is an instrumental technique for the determination of micro and semi-micro quantities of analyses in a sample. It is about measuring the effects of electromagnetic radiation's interactions with absorbing species like atoms, molecules, or ions in the UV range.

(Hussain, 2019). The *Borassus flabellifer Linn* dye extract is analysed for the UV-visible spectrophotometer.

3.7.2 Phytochemical analysis

The phytochemicals give a plant its colour, flavour, and fragrance, as well as immunity against disease and predators. The phytochemicals can improve the immune system, reduce the growth of cancer cells, and protect DNA from damage that can cause cancer. (Kumar, 2019). Some of the bioactive phytochemicals present in the fruit include phenolic acids, flavonoids, tannins, carotenoids, sterols, and lignans. A phytochemical analysis is used to evaluate the plant compounds present in plants. The bioactive components present in *Borassus flabellifer Linn* fruit extract have been identified.

3.7.3 % Yield of natural colorant

The % yield of natural colourant is examined using gravimetric analysis. 3 g of dry powder was mixed with 50 ml of water, and dye was extracted. Using a hot air oven, the water from the extract is evaporated. The dry powder weight is measured. The following formula is used to get the percent of the total yield (Zulrushdi et al.,2016)

$$\text{yeild of natural colourant}\% = \frac{\text{natural dye obtained}}{\text{weight of the sample}} * 100$$

3.7.4 FT-IR Analysis

To understand chemical and surface chemistry, Fourier transform infrared (FTIR) spectroscopy has been considered one of the most effective techniques (M.A. Mohamed et al., 2017). A dye source extract is used to analyses the FTIR to find the functional group present in the solution.

3.7.5 Antioxidant Activity

The scavenging activity of *Borassus flabellifer Linn.* fruit extract was estimated using 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical assay. (Yu et al.,2008) In a test tube, 1,000 ml of DPPH solution and 800 ml of Tris-HCl buffer were added. then combined in 200 ml of the testing extract solution. For 30 minutes, the solution was left at room temperature. Finally, the solution's absorbance at 517 nm was measured. (Lim, 2012)

$$DPPH(\%) = \left[\frac{(\text{Absorbance of control} - \text{Absorbance of sample})}{\text{Absorbance of control}} \right] * 100$$

The inhibition percentage was calculated using this formula.

3.8 Optimisation of Dyeing condition

3.8.1 Optimisation of dyeing Temperature

The natural dye extracted under optimised extraction conditions was used for dyeing cotton fabric at various temperatures (30°C, 50°C, 70°C, 90°C, and 100°C) with a material liquor ratio (M: L:R) of 1:20 for 60 minutes in order to establish the ideal dyeing temperature. The fabric that shows the maximum colour strength value is selected as the optimum temperature for dyeing cotton fabric.

3.8.2 Optimisation of dyeing Time

To determine the optimum time for dyeing, fabric samples were dyed at various time intervals (15, 30, 45, 60, and 75 min) at the optimal dyeing temperature in order to find the ideal dyeing time. The dyed samples were evaluated by a colour lab spectrophotometer, and the temperature that showed maximum colour strength was selected as the optimum time for dyeing.

3.8.3 Optimisation of dyeing Material liquor ratio (MLR)

Fabric samples were dyed at MLRs of 1:10, 1:20, 1:30, 1:40, and 1:50 independently at the optimised dyeing temperature and time in order to evaluate the effect of MLR on dyeing. Colour strength and colour coordinates were evaluated for all dyed fabric samples. The optimum fabric dyeing conditions have been determined to be those that exhibit the greatest colour strength.

3.8.4 Optimisation of dyeing Dyeing

Dyeing was carried out using dye extracted from *Borassus flabellifer* L. fruit at optimized conditions such as solvent, dye source concentration, time, temperature. The optimized extract was assessed for UV-Visible spectrophotometer.

3.9 Selection of mordants

Pomegranate contains polyphenols (flavonoids and tannins) and guava (flavonoids and quercetin), which act as a colourant and mordant. (Adeel et al., 2009) Tannin is present in the skin of pomegranate rind and it work well with cotton and other plant fibre. It yields ochre yellow dye (Kumar et al.,2021). Hence the mordant selected for the study was pomegranate rind powder and guava leaf powder.

3.9.1 Extraction of mordant

The extraction process was carried out using aqueous method. Pomegranate rind and guava leaf was dried and made into fine powder. The powder was taken as 6% Of concentration per 100ml of water. The solution was heated for 45min at 100°C then the solution was filtered and used as mordant.

3.9.2 Mordanting techniques

Mordants are substances that create a bond between dye and the textile material, aiding in the improvement of fastness and dye uptake. Mordanting is the process of developing colour between natural dye and fabric. Guava and pomegranate rind were chosen as mordants due to their eco-friendliness. The selected cotton fabric was modranted with pomegranate rind and guava leaf powder (6%) using a 1:20 MLR for 60 min at 100°C.

The fabric was mordanted with selected mordants using pre- mordanting, simultaneous mordanting, and post mordant techniques. The mordant fabric was analysed for colour strength.

3.9.2.1 Pre-mordanting

Pre-mordanting was done before dyeing the fabric with the dye source. Cotton fabric was first treated with mordant solution (pomegranate rind and guava leaf powder) individually with a M.L. ratio of 1:20 for 60 min at 100 °C. After pre-mordanting, the sample was dyed with *Borassus flabellifer* dye extract.

3.9.2.2 Simultaneous mordanting

The cotton fabric was dyed simultaneously by both *Borassus flabellifer* dye extract and mordants in the same dye bath at MLR 1:20 for 60 min at 100°C. The dyed fabric was then dried.

3.9.2.3 Post- Mordanting

First, the cotton fabric should be dyed using fruit extract, and later, the fabric should be added to the mordanting solution (pomegranate rind and guava leaf powder).

3.10 Dyeing under optimised conditions

The cotton is dyed using *Borassus flabellifer L.* fruit under optimised dye extraction and dyeing conditions such as dye source concentration, temperature, time, and MLR. And the fabric was dyed with appropriate mordanting techniques and dried at room temperature, and the dyed fabric was assessed for colour strength and colour coordination.

3.11 Evaluation of dyed fabric

3.11.1 Fabric weight (GSM) (ASTM D 2646/D3776, ISO 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. The value in grams is 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, DF, PMF and GMF samples. Three samples were weighed five times and the mean value was calculated and recorded (plate 6)

Grams per Square Meter = Specimen weight in grams * 100

3.11.2. 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 gauge 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 the pressure foot slowly on the specimen. The dial indicates the thickness of the sample and the readings were noted from dial gauge. Five readings were taken from different places of sample and the same procedure was followed for all the UDF, DF, PMF and GMF samples. Five readings were taken from different places of the fabric sample and the mean was calculated. It is expressed in mm (plate 7)

3.11.3 Fabric stiffness (IS 6490:1971)

Stiffness is 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 is mostly judge by using stiffness test. Stiffness of a fabric is determined by using Shirley stiffness tester. 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 readings of warp and weft specimen were recorded. The same procedure was followed for UDF, DF, PMF and GMF samples of warp and weft bending readings was noted. Two reading from weft and warp were taken and the average was calculated and recorded (plate 8)

3.11.4 Absorbency

3.11.4.1 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 behavior 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 UFD, DF, PMF and GMF specimen were taken as 2.5cm*2.5cm. The sample were dropped at certain height into 50ml of distilled water in glass beaker and the sinking time of sample were recorded using stopwatch. The same procedure was repeated for all the samples five times. The results were noted and mean value was calculated.

3.11.4.2 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 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).

The UDF, DF, PMF and GMF 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 same procedure is followed for DF, PMF and GMF samples. The testing is done for five times and the mean value of all the samples were calculated and the results are noted.



Plate 6- Fabric weight

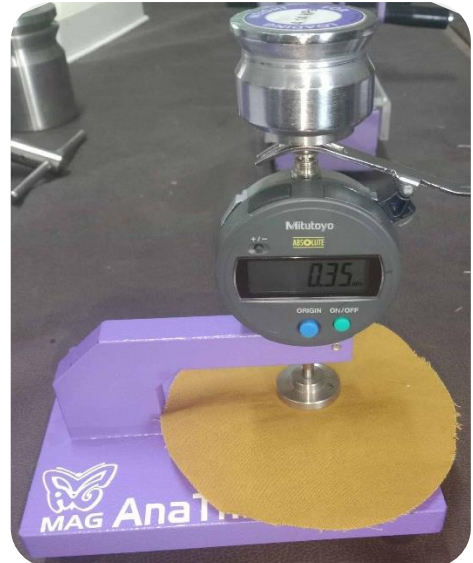


Plate 7- Fabric thickness



Plate 8- Fabric stiffness



Plate 9- Crock meter

3.11.5 Colour fastness

Colour fastness is 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.

3.11.5.1 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 DF, PMF and GMF were tested for washing using five specimens five times. The level of colour change in dyed and mordant fabrics was compared and assessed by using grey scale.

3.11.5.2 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). The same procedure was performed for DF and MDF specimen and the amount of colour transferred to the white fabric is assessed using AATCC grey scale (plate 9)

3.12 Assessment of functional properties

3.12.1 UPF Analysis

The UV absorption and UV protection properties of the control fabric, and the final dyed fabric with *were* evaluated according to AATCC test method 183-2010.

3.12.3 Antibacterial assay

The control fabric, mordanted fabric, and dyed fabric are individually tested for antibacterial activity against *Staphylococcus aureus* and *E. coli* strains. The samples were placed in culture medium and compared with standard antibiotics. The plate was aerobically incubated at 37°C for 24 h. The diameters of the inhibition zones against the tested bacteria were measured. (Alamelumangai et al., 2014 and Li et al., 2019)

3.14 NOMENCLATURE OF THE SAMPLES

The nomenclature of the samples is presented in Table II

Table II

Nomenclature of the samples

| S.no | Description of sample | Nomenclature used |
|------|---------------------------------|-------------------|
| 1 | Undyed fabric | UDF |
| 2 | Dyed fabric | DF |
| 3 | Mordant fabric Pomegranate | MFP |
| 4 | Mordant fabric Guava | MFG |
| 5 | Pomegranate mordant dyed fabric | PMF |
| 6 | Guava mordant dyed fabric | GMF |

4. RESULTS & DISCUSSION

The result of the study “Eco-friendly natural dye from *Borassus flabellifer* L. fruit for Coloration on Cotton Fabric,” is discussed under the following headings:

4.1 Optimisation of dye extraction conditions

4.1.1 Optimisation of Solvent

4.1.2 Optimisation of Dye source concentration

4.1.3 Optimisation of extraction Time

4.1.4 Optimisation of extraction Temperature

4.1.5 Optimised conditions for extraction of natural dye using *Borassus flabellifer* L. fruit extract

4.2 Characterisation of the dye extract

4.2.1 UV- Vis Analysis

4.2.2 Phytochemical Analysis

4.2.3 % Yield of natural colourant

4.2.4 FT-IR

4.2.5 Antioxidant analysis

4.3 Optimisation of dyeing conditions

4.3.1 Optimisation of Dye concentration

4.3.2 Optimisation of Dyeing Temperature

4.3.3 Optimisation of dyeing Time

4.3.4 Optimisation of dyeing Material Liquor ratio

4.3.5 Optimised condition for dyeing cotton using *Borassus flabellifer L.* fruit extract

4.4 Mordanting technique

4.4.1 Effect of mordanting techniques

4.5 Dyeing under optimized conditions

4.6 Fabric evaluation

4.6.1 Fabric weight

4.6.2 Fabric thickness

4.6.3 Fabric stiffness

4.6.4 Absorbancy

4.6.4.1 Sinking

4.6.4.2 Wicking

4.6.5 Colour Fastness

4.6.5.1 Fastness to washing

4.6.5.2 Fastness to crocking

4.7 Assessment of functional properties

4.7.1 UPF analysis

4.7.2 Antibacterial analysis

4. RESULTS AND DISCUSSION

4.1 Optimization of dye extraction conditions

4.1.1 Selection of solvent for dye extraction

To select the suitable solvent, the dye was extracted with various solvents, namely water, sodium hydroxide, and ethanol, and the results are presented in Table III.

TABLE III

Selection of solvent

| Solvent | Optical density at 350nm |
|-----------------|--------------------------|
| Water | 0.477 |
| NaOH (1%) | 5.029 |
| Ethanol (80:20) | 1.028 |

From the table, it is clear that the dye yield is maximum with NaOH, with absorbance of 5.029. Hence, alkaline medium was selected as a suitable solvent for dye extraction.

4.1.2 Optimization of dye source concentration

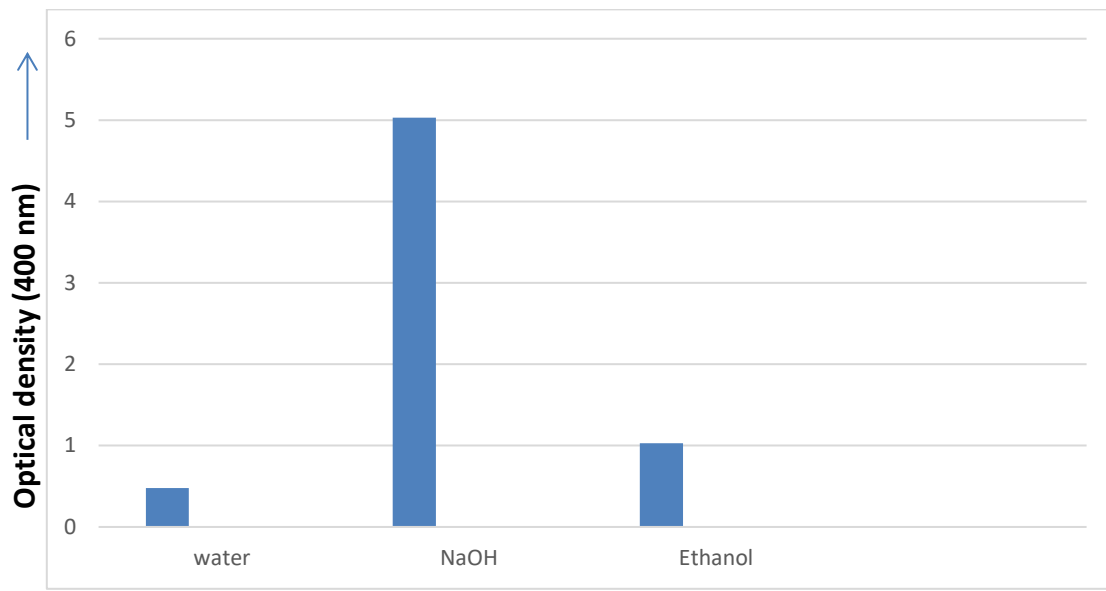
The influence of dye source concentration on colour yield is shown in Table IV.

Table IV

Optimization of dye source concentration

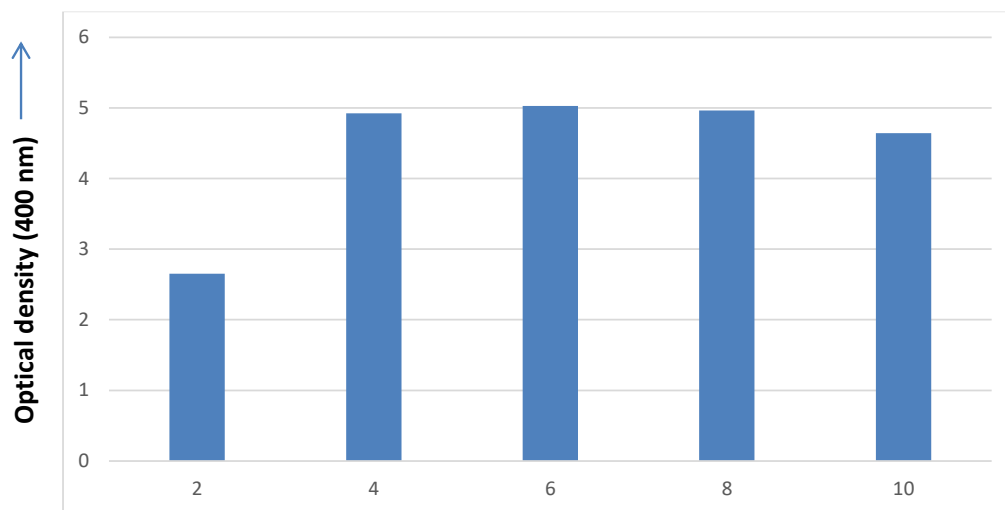
| Dye source Concentration (%) | Optical density at 400 nm |
|------------------------------|---------------------------|
| 2 | 2.653 |
| 4 | 4.925 |
| 6 | 5.029 |
| 8 | 4.963 |
| 10 | 4.643 |

It is observed from the results that increase in dye source concentration increase dye yield and reaches maximum at 6% (5.029). Whereas increase in concentration above 6% decrease the dye yield. Hence, dye source concentration of 6 % was found as optimum.



Solvent →

Figure 5- Selection of solvent



Dye source concentration (%) →

Figure-6 Dye source concentration

4.1.3 Optimization of extraction time

Extraction time is an important parameter that influences dye yield. To determine the optimum extraction time, the dye was extracted at varied time intervals, and the results are presented in Table V.

Table V

Optimization of extraction time

| Time (min) | Optical density at(400 nm) |
|-------------------|-----------------------------------|
| 15 | 2.624 |
| 30 | 4.521 |
| 45 | 5.134 |
| 60 | 5.004 |
| 75 | 4.971 |

It is observed that an increase in time increases dye yield up to 45 min (5.134). A further increase in extraction time doesn't show a significant increase in colour yield. This is due to the decomposition of colouring compounds when the extraction of dye is prolonged for a longer time. Hence, an extraction time of 45 minutes was selected as the optimum time.

4.1.4 Optimization of extraction temperature

The effect of extraction temperature on colour yield is shown in Table VI.

Table VI

Optimization of extraction temperature

| Temperature (°C) | Optical density at (400 nm) |
|-------------------------|------------------------------------|
| 30 | 0.497 |
| 50 | 0.984 |
| 75 | 1.807 |
| 100 | 2.624 |

The results indicate that an increase in temperature increases colour yield and reaches its maximum at 100 °C. Hence, 100 °C was selected as the optimum temperature for dye extraction. This is due to the better solubility of dye molecules at higher temperatures.(Ali, 2008)

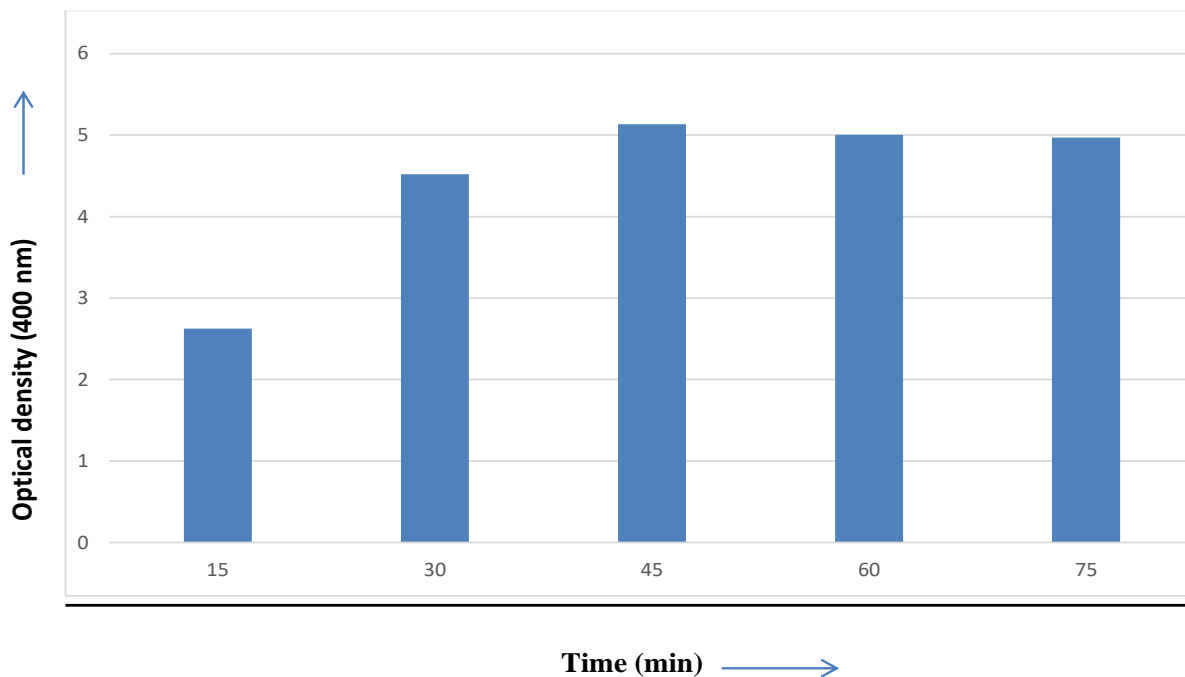


Figure-7 Optimization of extraction time

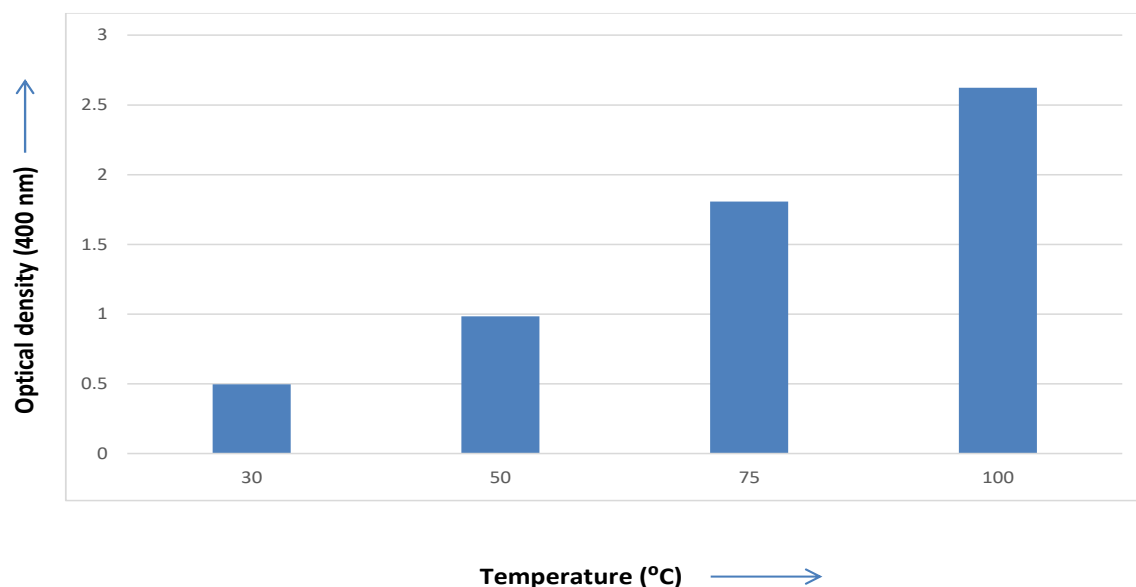


Figure -8 Optimization of extraction temperature

4.1.5 Optimized conditions for extraction of natural dye from *Borassus flabellifer L.* fruit

Optimized conditions for extraction of natural dye from *Borassus flabellifer L.* fruit in Table VII

Table VII

Optimized conditions for dye extraction

| S.No | Parameters | Optimum conditions |
|------|--------------------------|--------------------|
| 1 | Solvent | NaOH (1%) |
| 2 | Dye source concentration | 6% |
| 3 | Time | 45 min |
| 4 | Temperature | 100°C |

The optimized extract condition shows that, the colour yield was maximum with water as solvent, 6% dye source concentration and extraction time of 45 min at 100 °C.

4.2 Characterisation of dye extract

4.2.1 UV-VIS analysis

UV-VIS analysis of *Borassus flabellifer L.* fruit extract results are presented in Table VIII. Table VIII, reveals that *Borassus flabellifer L.* fruit extract showed maximum absorbance peak at 250nm in UV region and at 400 nm in visible region

Table VIII

UV-Visible analysis

| Wavelength (nm) | Absorbance |
|----------------------------|-------------------|
| 200 | 7.905 |
| 250 | 5.680 |
| 300 | 4.663 |
| 400 | 0.946 |
| 500 | 0.403 |
| 600 | 0.246 |
| 700 | 0.177 |
| 800 | 0.125 |

Table XIV, reveals that *Borassus flabellifer L.* fruit extract showed maximum absorbance peak at 250nm in UV region and at 400 nm in visible region

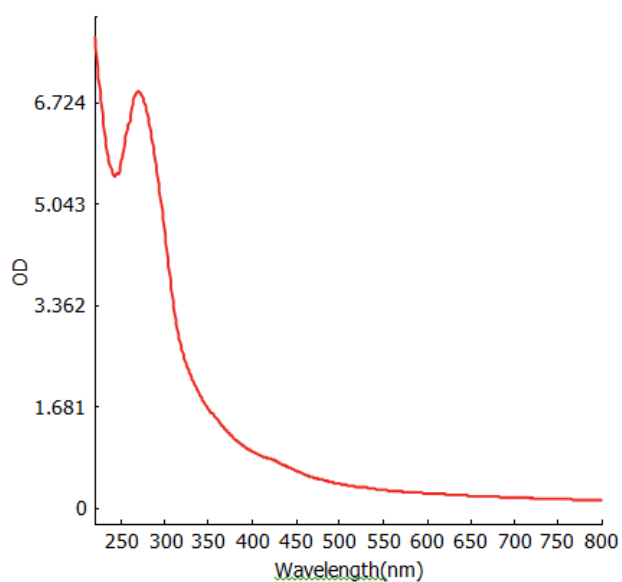


Figure 9- UV-VIS spectrum of *Borassus flabellifer L.* fruit extract

4.2.2 Phytochemical Analysis

The alkaline extract of *Borassus flabellifer* L. was analysed for the presence of chemical compounds using qualitative phytochemical test and the results are presented in Table IX

Table IX

Results of Phytochemical Screening of Dye extract

| S.N o. | Metabolite | Test performed | Observation | Result | Figure No. |
|-----------|--------------------------------------|---|---|--------|---------------|
| 1 | Alkaloids | + Mayer's reagent | Presence of Cream coloured precipitate | + | 1 |
| | | + Dragendorff's reagent | Presence of reddish- brown precipitate | + | 2 |
| 2 | Flavonoids | Alkaline test | Intense yellow with NaOH and becomes colourless with dil. HCl | + | 3 |
| | | + H ₂ SO ₄ | Presence of reddishorange colour | + | 4 |
| 3 | Sterols (Liebermann test) | + CHCl ₃ + Acetic anhydride +Conc.H ₂ SO ₄ | Presence of reddish-brown ring | + | 5 |
| 4 | Anthraquinone (Borntrager's test) | + FeCl ₃ + Conc. HCl diethyl ether+ Ammonia | Presence of reddishorange colour | + | 6 |
| 5 | Proteins | + conc. HNO ₃ | Presence of Yellow colouration | + | 7 |
| 6 | Phenolic compounds | + 5% neutral FeCl ₃ | Presence of bluishgreen coloured solution | + | 8 |
| | | + Ellagic acid test | Presence of niggerbrown precipitate | + | 9 |

| | | | | | |
|---|---------------|-----------------------|-----------------------------|---|-----------|
| 7 | Carbohydrates | Molisch's test | Presence of Violet ring | + | 10 |
| | | Fehling's test | Presence of Red precipitate | + | 11 |

For the Table IX, it is observed the presence of alkaloids, flavonoids, sterols, anthraquinones, proteins, carbohydrates, saponins, and cardiac glycosides in dye extract.

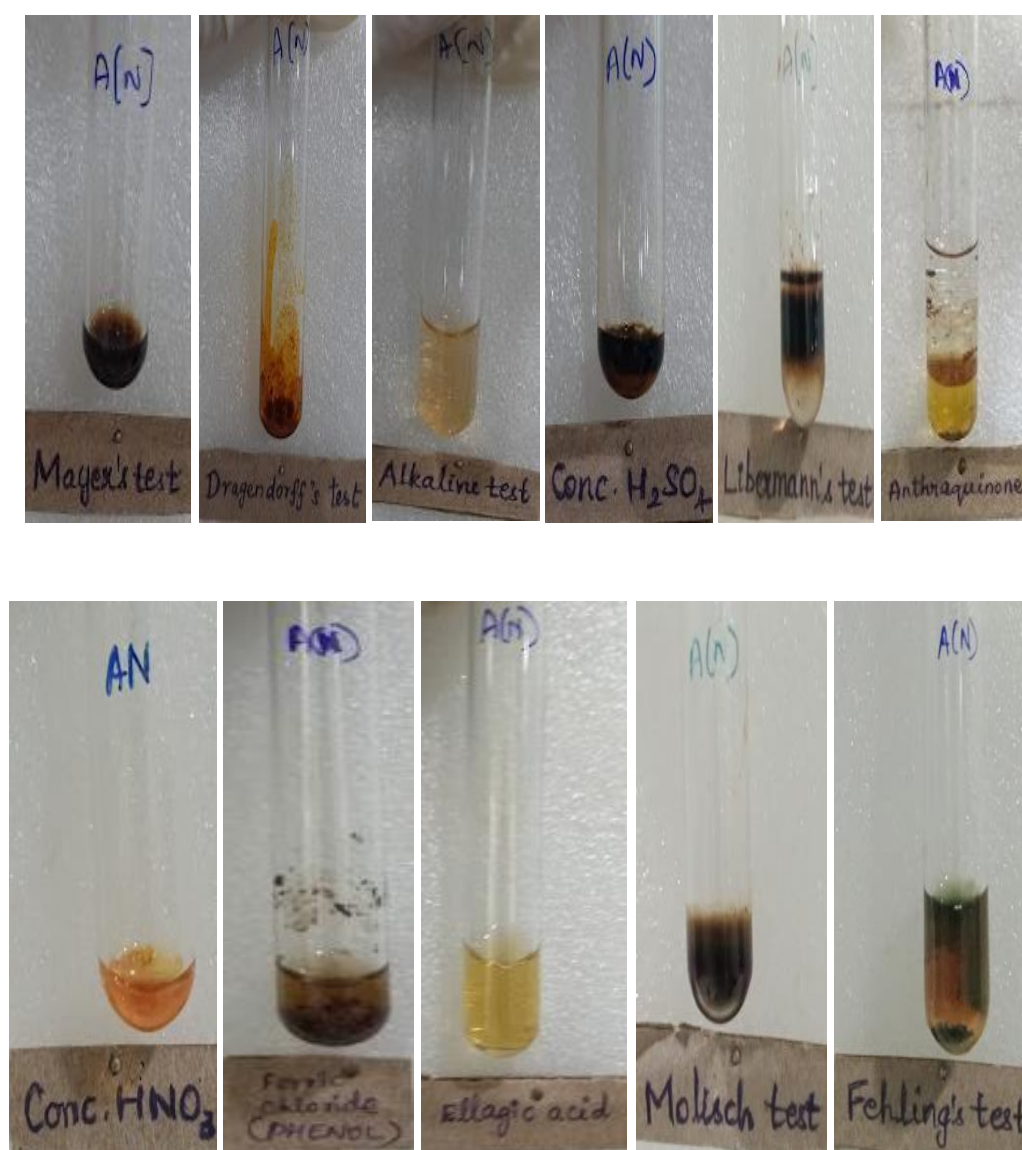


Figure- 10 Phytochemical Screening of Dye extract

4.2.3 % Yield of natural colourants

Dye yield is obtained by evaporating the water to procure the quantity crude dye from the alkaline extract. Dye yield is the quantity of crude dye powder obtained after evaporating the water from the alkaline extract. After this process, crude powder is weighted without purification. The dye yield for *Borassus flabellifer L.* extract was found to be 55.03 %

4.2.4 FT-IR

The FTIR spectroscopy of the natural dye extract from *Borassus flabellifer L.* fruit extract is shown in Figure 11

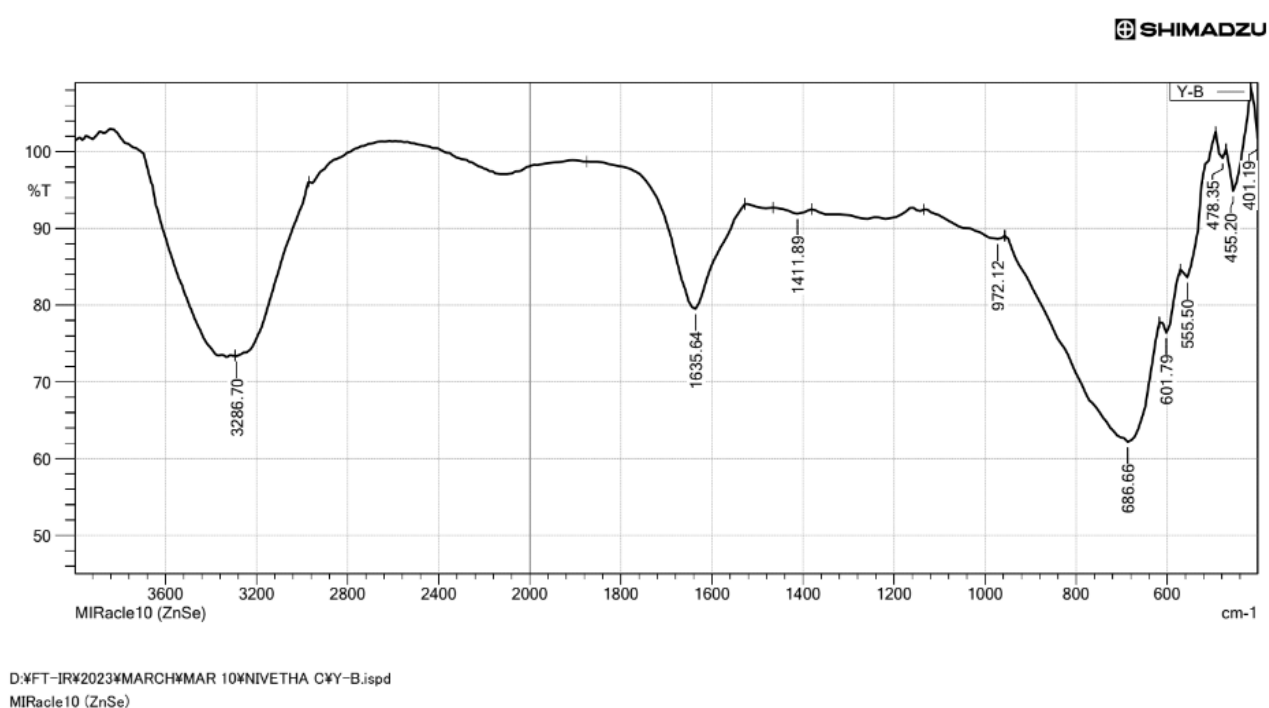


Figure – 11 FTIR spectra of *Borassus flabellifer L.* fruit extract

Functional groups on dye extract are analyzed by analyzing the infrared spectrum's absorption peaks. The FTIR spectra of the extract are shown in Figure 11, the results of these spectra are utilized to observe changes in the structure or functional group of the substance. The band 3286 cm⁻¹ corresponding to the OH- stretching vibration of alcohol was detected. The 1634 cm⁻¹, 972 cm⁻¹, and 686 cm⁻¹ peaks indicate the existence of C=C stretching. S=O stretching vibration

is indicated by the strong band at 1411 cm^{-1} . C- Br stretching formation is identified by the peak at 601 cm^{-1} . The presence of a strong band of C=Cl stretching at 555 cm^{-1} is also detected. (Gala, 2018; Singh, 2014)

4.2.5 Antioxidant activity

The antioxidative activity of alkaline extract was measured by the ability to scavenge DPPH free radicals and compared with the standard solution, ascorbic acid. The absorbance of the extract is measured at 517nm. The scavenging activity of the alkaline extract and ascorbic acid on DPPH radical were expressed as half maximal inhibition concentration (IC₅₀) values. (Prasad G, 2015) The antioxidant level of *Borassus flabellifer* L. extract is represented in Table X

Table X

Antioxidant activity

| Concentration ($\mu\text{g/ml}$) | % Inhibition |
|--|---------------------|
| 3 | 62.30 |
| 6 | 70.49 |
| 9 | 83.61 |
| 12 | 90.16 |
| 15 | 95.90 |

From the Table X, it is observed that the % inhibition is maximum at the concentration of 15%.

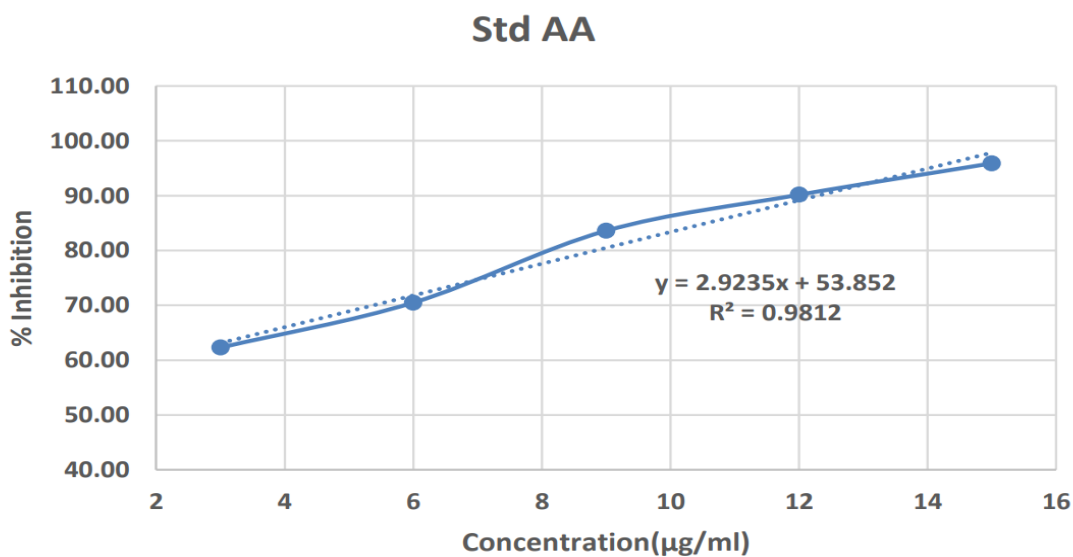


Figure-12 Antioxidant activity for *Borassus flabellifer L.* fruit extract

Figure 12 shows the comparison of standard ascorbic acid and *Borassus flabellifer L.* fruit extract. The plant extract reaches its maximum at 9% concentration by 83.61

4.3 Optimization of dyeing conditions

Borassus flabellifer L. dye extract was used to dye cotton fabric. For the dyed fabric, a strength value is observed for varied parameters. The higher the colour coefficient, the more colour strength. The colour strength and colour coefficient are evaluated using a premium colorsan spectrophotometer by light reflection technique. It is examined by varying the dye concentration, MLR, time, and temperature for optimisation of dyeing by utilising one variable at a time.


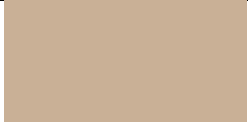


4.3.1 Optimization of dye concentration

The influence of dye concentration on colour strength k/s value is shown in Table XI.

It is observed that an increase in dye concentration increases the colour strength value and reaches its maximum at 100% dye extract. This is because when the concentration of extract is higher, the availability of dye molecules will be greater to dye the fabric. Hence, 100% dye extract is selected as the optimum concentration to dye the fabric.

Table XI

Optimization of dye concentration

| Dye Concentration | L* | a * | b * | K/S | Shade Produced |
|--------------------------|-----------|------------|------------|------------|---|
| 25:75 | 95.055 | 0.538 | 3.017 | 5.956 |  |
| 50:50 | 94.987 | 0.613 | 2.884 | 8.019 |  |
| 75:25 | 95.063 | 0.606 | 3.077 | 14.866 |  |
| 100 | 95.218 | 0.591 | 3.455 | 22.950 |  |

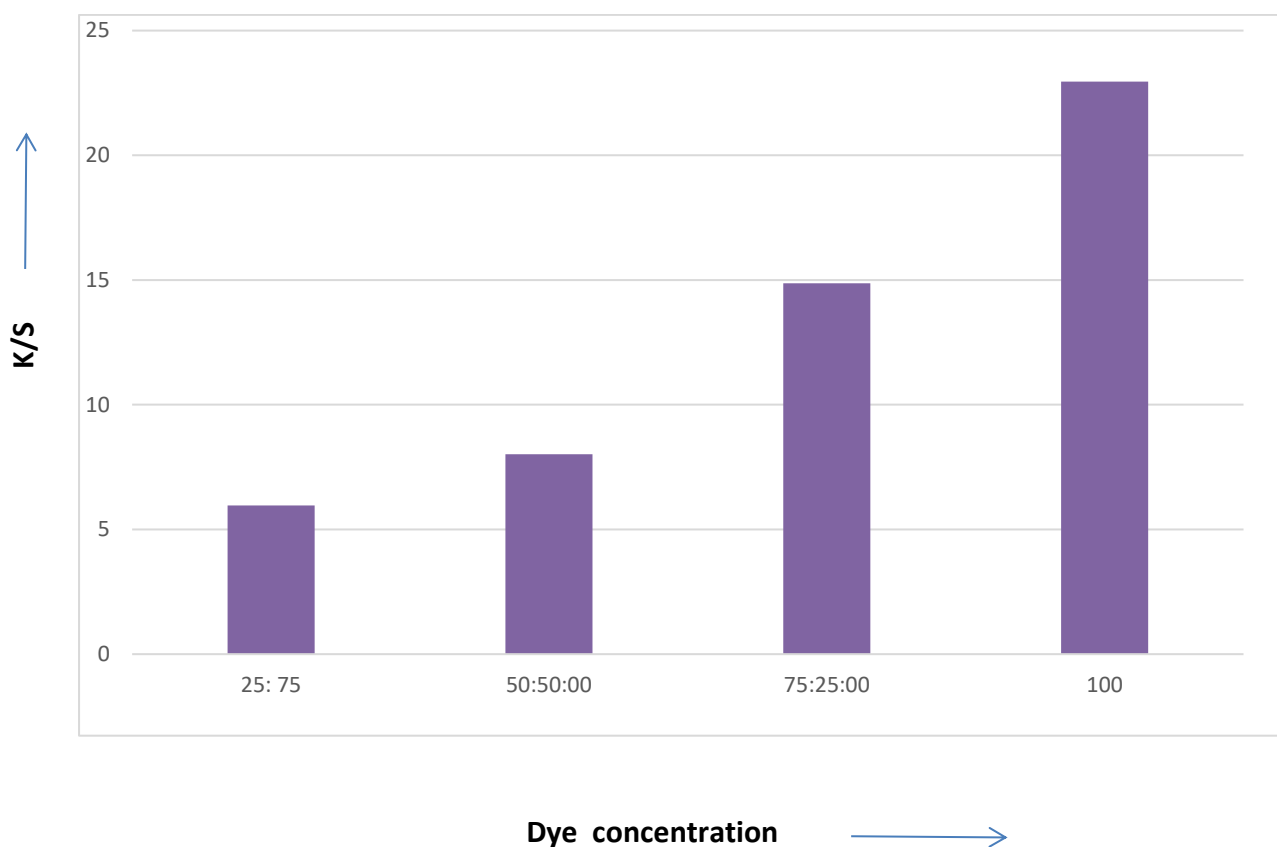
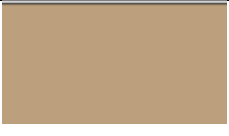





Figure-13 Optimization of dye concentration

4.3.2 Optimization of dyeing Temperature

The effect of dyeing temperature is demonstrated in Table XII. It is clear that the colour strength increases with increasing dyeing temperature and reaches a maximum of 100°C (34.216); this is due to the increase in kinetic energy of dye molecules and swelling of fibre and disaggregation of dye at high temperatures. Hence, 100°C was selected as the optimum temperature for dyeing.

Table XII
Optimization of dyeing Temperature

| Temperature (°C) | L* | a * | b * | K/S | Shade produced |
|-------------------------|-----------|------------|------------|------------|---|
| 30 | 95.166 | 0.405 | 3.363 | 14.634 |  |
| 50 | 95.216 | 0.388 | 3.498 | 24.843 |  |
| 75 | 95.042 | 0.638v | 3.113 | 29.457 |  |
| 100 | 95.098 | 0.523 | 3.238 | 34.216 |  |

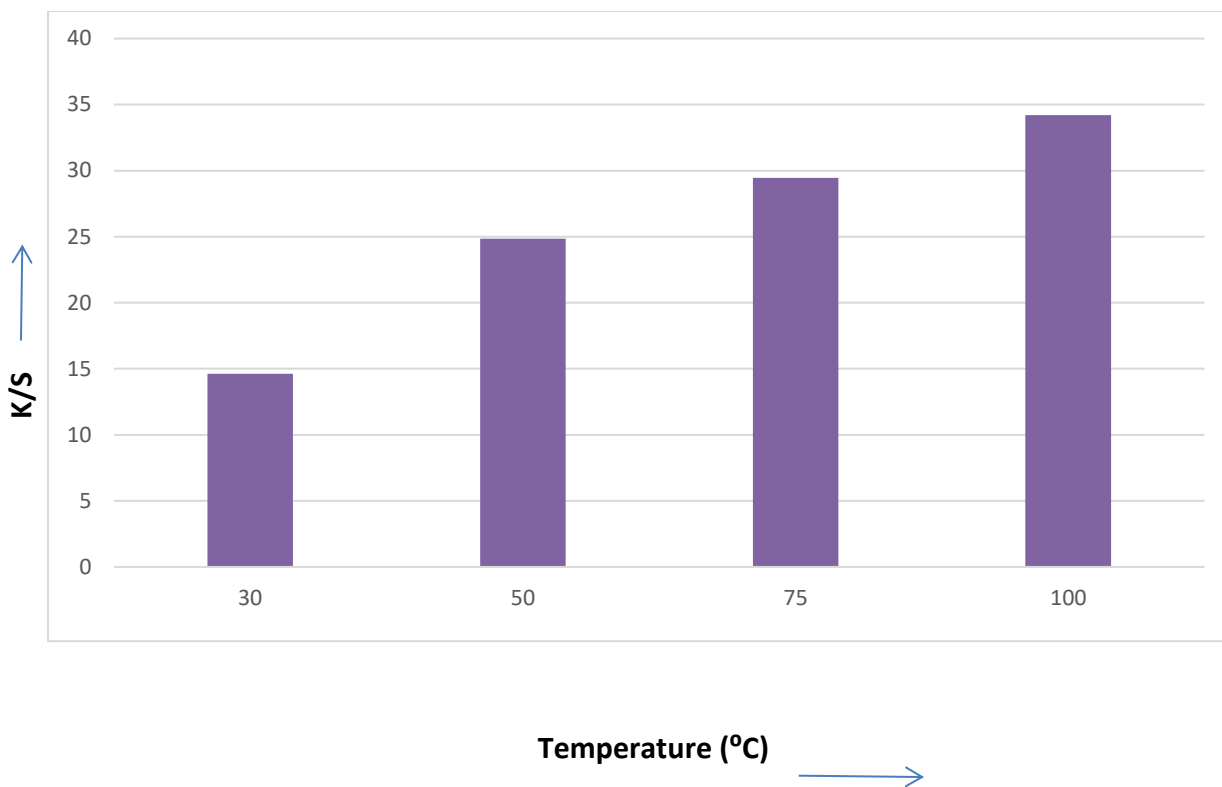
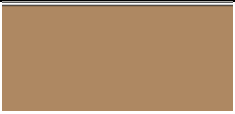

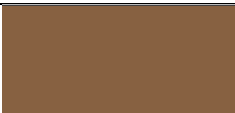




Figure-14 Optimization of dyeing Temperature

4.3.3 Optimisation of Dyeing time

The effect of dyeing time on the colour strength value of *Borassus flabellifer* L. extract is shown in Table XIII.

Table XIII**Optimisation of Dyeing time**

| Time (min) | L* | a* | b* | K/S | Shade Produced |
|-------------------|-----------|-----------|-----------|------------|---|
| 15 | 95.288 | 0.588 | 3.612 | 28.359 |  |
| 30 | 95.185 | 0.569 | 3.379 | 36.335 |  |
| 45 | 95.230 | 0.619 | 3.492 | 58.750 |  |
| 60 | 95.154 | 0.773 | 3.358 | 103.836 |  |
| 75 | 95.170 | 0.671 | 3.380 | 76.410 |  |

It is observed that the colour strength increases with an increase in dyeing time and reaches its maximum at 60 minutes (103.836). Further increase in dyeing time cause the dye molecules that have already been absorbed onto the fabric surface to desorb, reducing the dye's colour strength values and uniformity. (Kamel,2007)

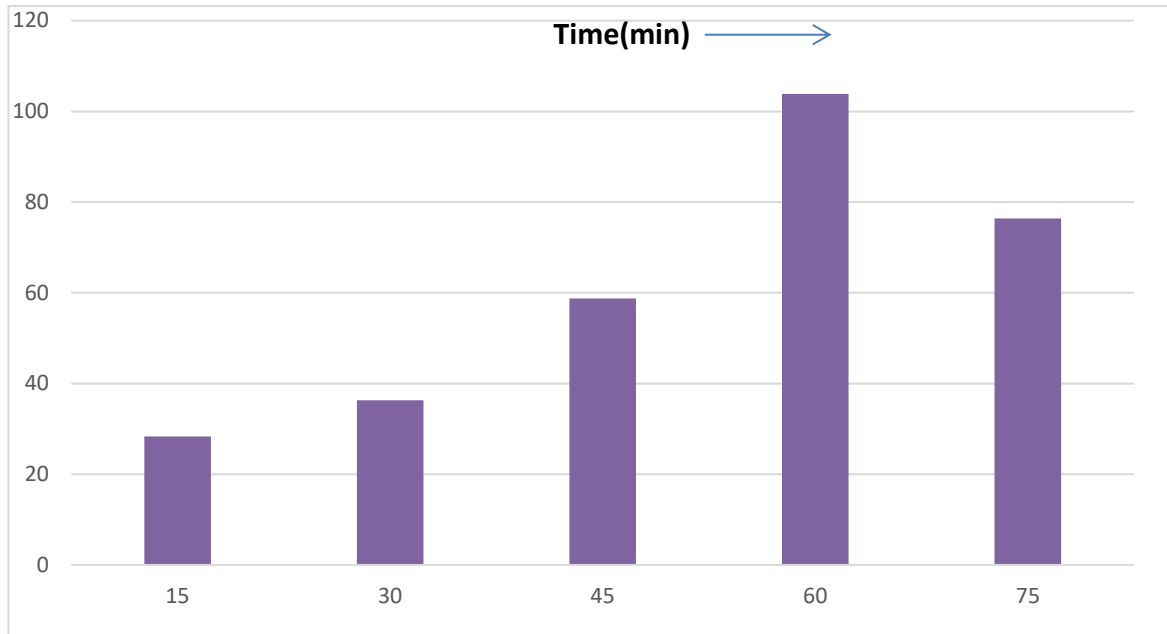


Figure-15 Optimisation of Dyeing time






4.3.4 Optimization of Material Liquor Ratio

The amount of liquor to be used in the dye bath determined by the weight of the fabric is known as the material liquor ratio. The influence of the liquid ratio on the colour strength of dyed cotton is shown in Table XIV.

An increase in MLR from 1:10 to 1:20 increases the colour strength value, and a further increase in MLR results in decreased colour strength. This is due to the overcrowding of dye molecules, which collide with each other and decrease the attraction for fabric. (FAROOQ,2012)

Table XIV

Optimization of Material Liquor Ratio

| MLR | L* | a * | b * | K/S | Shade produced |
|------------|-----------|------------|------------|------------|---|
| 1:10 | 95.191 | 0.582 | 3.395 | 39.352 |  |
| 1:20 | 95.221 | 0.591 | 3.467 | 50.005 |  |
| 1:30 | 95.218 | 0.591 | 3.455 | 22.950 |  |
| 1:40 | 95.119 | 0.569 | 3.220 | 21.246 |  |
| 1:50 | 95.285 | 0.633 | 3.613 | 18.148 |  |

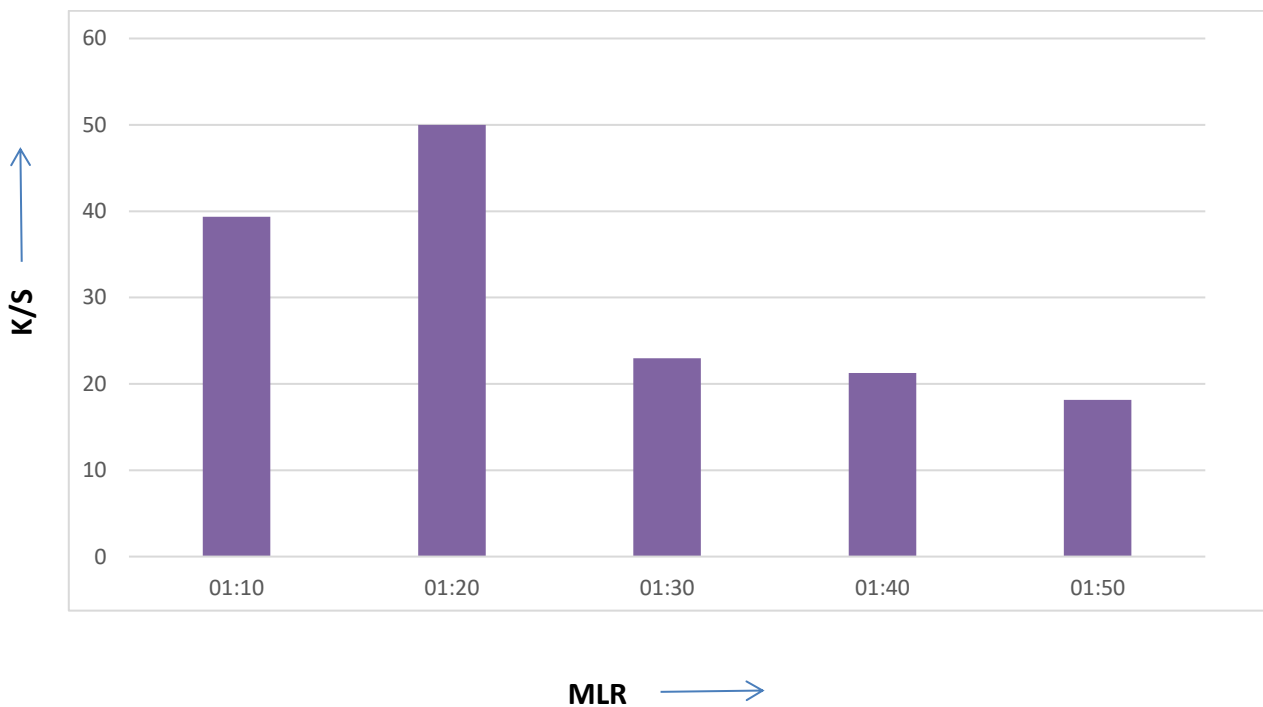


Figure- 16 Optimisation of MLR

4.3.5 Optimized conditions for dyeing of cotton fabric with *Borassus flabellifer L.* fruit

Optimized conditions for dyeing of cotton fabric with *Borassus flabellifer L.* fruit extract is presented in Table XV

Table XV

Optimized Dyeing condition

| S.No | Parameters | Optimum conditions |
|------|-------------------|--------------------|
| 1 | Dye concentration | 100% |
| 2 | Time | 60 min |
| 3 | Temperature | 100°C |
| 4 | MLR | 1:20 |

The dyeing colour strength was maximum at a temperature of 100°C with a material liquor ratio of 1:20 at a dyeing time of 60 min. Hence, the fabric is dyed under optimised conditions.

4.4 Mordanting technique




Mordant helps to bind natural colourants to the fabric. Mordants help to increase the fastness of light, washing, and rubbing. Mordants help create a variety of hues when dyed with a dye source. For this process, we use pomegranate rind and guava leaf as bio mordants. Pomegranate contains polyphenols (flavonoids and tannins) and guava (flavonoids and quercetin), which act as mordants.

4.4.1 Effect of mordanting techniques

Cotton fabrics were mordanted using different mordanting techniques, such as pre-mordanting, simultaneous mordanting, and post-mordanting, and dyed with *Borassus flabellifer L.* fruit extract. The colour strength value of dyed fabric is shown in Table XVI and XVII. Pre-mordanting technique was found to be effective for mordanting cotton fabric with pomegranate rind and guava leaf at 6% concentration. In the pre-mordanting technique, the k/s value of the pomegranate rind was 129.652, and for the guava mordant, it was found to be 105.846. Due to the interaction between the mordant and fibre, there were differences in colour strength values with different mordanting techniques.

Table XVI

Effect of mordanting technique on cotton- Pomegranate rind

| | L* | a* | b* | K/S | Shade Produced |
|--------------------------------|-----------|-----------|-----------|------------|---|
| PRE MORDANTING | 95.670 | 0.106 | 4.457 | 105.846 |  |
| SIMULTANEOUS MORDANTING | 95.315 | 0.370 | 3.704 | 40.785 |  |
| POST MORDANTING | 95.834 | 0.360 | 4.496 | 61.976 |  |

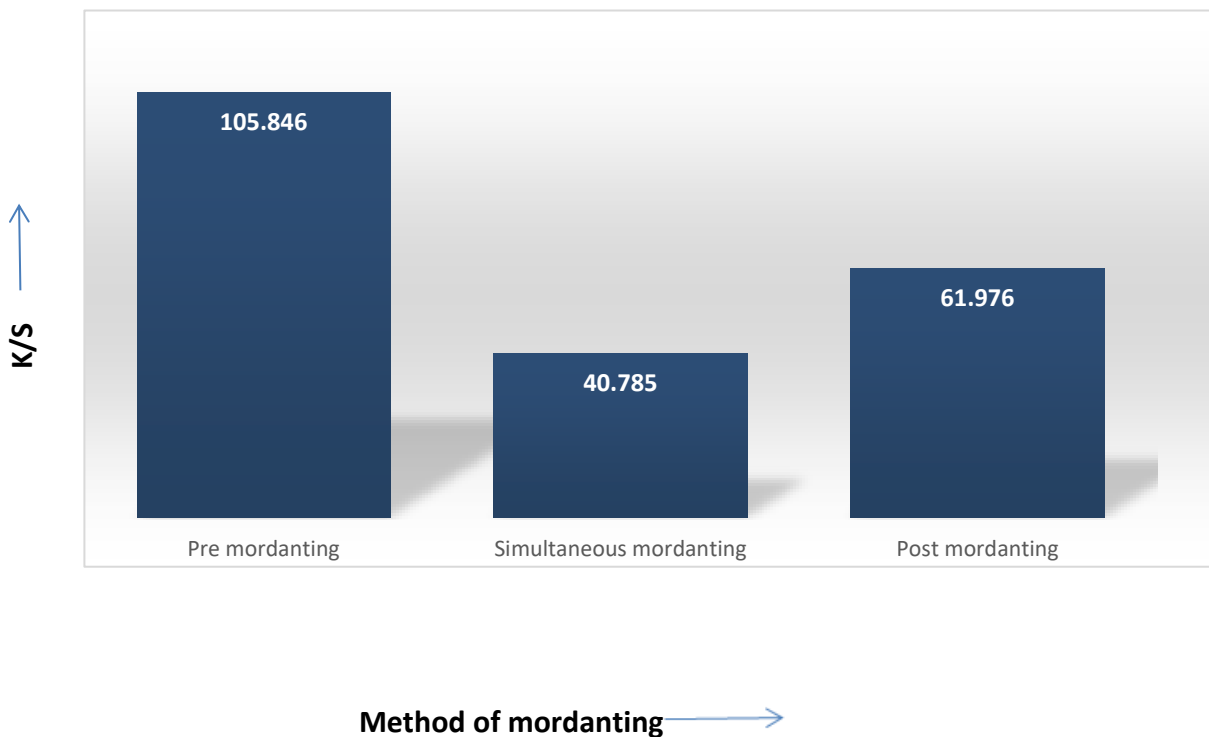





Figure 17- Effect of mordanting on cotton fabric- Pomegranate rind

Table XVII

Effect of mordanting technique on cotton- Guava leaf

| | L* | a* | b* | K/S | Shade Produced |
|--------------------------------|-----------|-----------|-----------|------------|---|
| PRE MORDANTING | 95.243 | 0.803 | 3.660 | 129.652 |  |
| SIMULTANEOUS MORDANTING | 95.235 | 0.643 | 3.569 | 74.402 |  |
| POST MORDANTING | 95.157 | 0.736 | 3.351 | 61.638 |  |

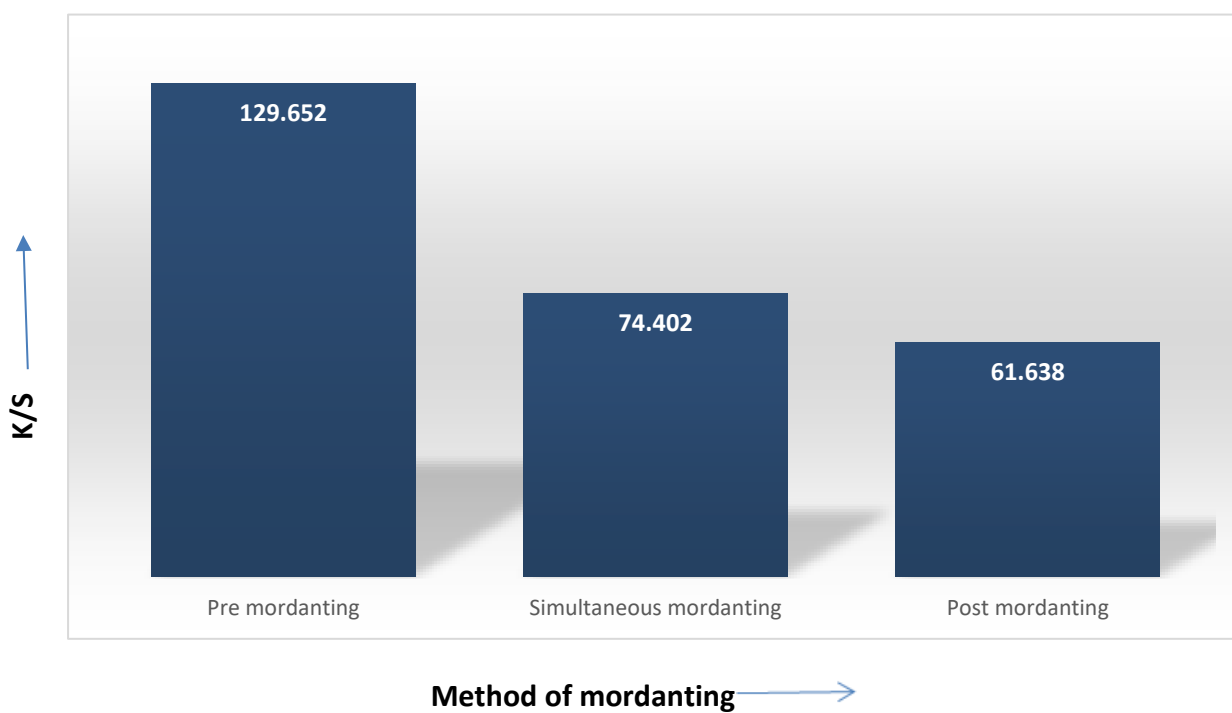



Figure 18 - Effect on Mordanting technique on Cotton- Guava leaf

4.5 Dyeing under optimised conditions

The cotton fabric was dyed under optimised condition parameter such as dye concentration, time, and temperature and MLR. The colour strength (K/S) and colour coordinates (L*, a*, b*) of cotton fabric under optimized conditions are presented in Table XVIII

Table XVIII

Colour strength and colour coordination of dyed fabric

| Colour coordination | | | Colour strength | Colour shade |
|---------------------|-------|-------|-----------------|---|
| L* | a * | b * | K/S |  |
| 95.221 | 0.591 | 3.467 | 50.005 | |

4.6 Fabric evaluation

4.6.1 Fabric weight

the weight of fabric (GSM) of undyed fabric, dyed fabric and mordant dyed fabric samples were analysed and the value are presented in Table XIX

Table XIX

Fabric weight

| S.no | Sample | Mean fabric weight (GSM) | Gain or loss over original | % gain or loss over original | T- test |
|------|--------|--------------------------|----------------------------|------------------------------|------------|
| 1. | UDF | 1.39 | - | | 2.70875435 |
| 2. | DF | 1.52 | 0.13 | 8.55 | |
| 3. | GMF | 1.60 | 0.21 | 13.12 | |
| 4. | PMF | 1.65 | 0.26 | 15.75 | |

From Table XIX, it is observed that the weight of undyed fabric is lower at 1.39 GSM when compared to dyed and mordant dyed fabric. There is an increase in weight of dyed and mordant dyed fabric of 8% and 13%,15% respectively. But mordantly dyed fabric shows the highest increase in weight compared to undyed and dyed fabric. The "t" test result proves that the increasing in fabric weight is found to be significant.

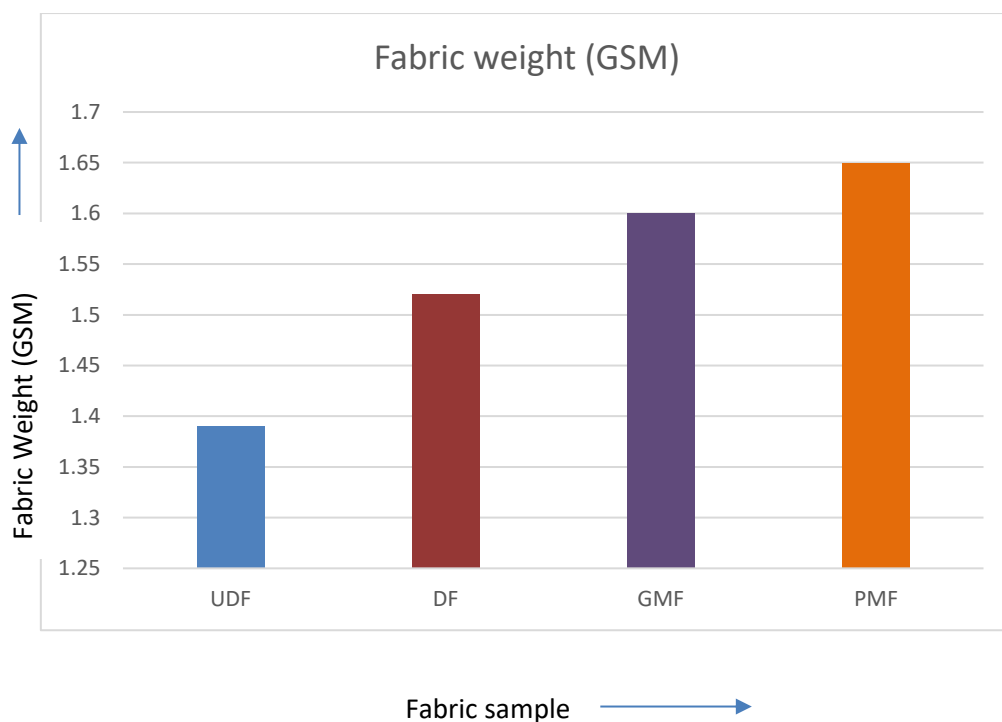


Figure-19 Fabric weight

4.6.2 Fabric thickness

The thickness of undyed, dyed and mordant dyed samples is analysed and presented in Table XX

Table XX

Fabric thickness

| S.no | Sample | Mean fabric weight (mm) | Gain or loss over original | % gain or loss over original | T- table |
|------|--------|-------------------------|----------------------------|------------------------------|----------|
| 1. | UDF | 0.25 | - | - | 0.035312 |
| 2. | DF | 0.35 | 0.05 | 16.66 | |
| 3. | GMF | 0.35 | 0.1 | 28.57 | |
| 4. | PMF | 0.34 | 0.09 | 26.47 | |

From Table XX. It is clear that the thickness of dyed and mordant dyed fabrics gains more thickness than undyed fabric, with gains of 16%, 28%, and 26%, respectively. Hence, the mordant-dyed fabric gains 0.35mm in thickness. The increases in thickness over dyed fabric

might be due to the treatment of the fabric with mordant, which increases dye uptake. The "t" result proves that the increase in fabric thickness found to be not significant.

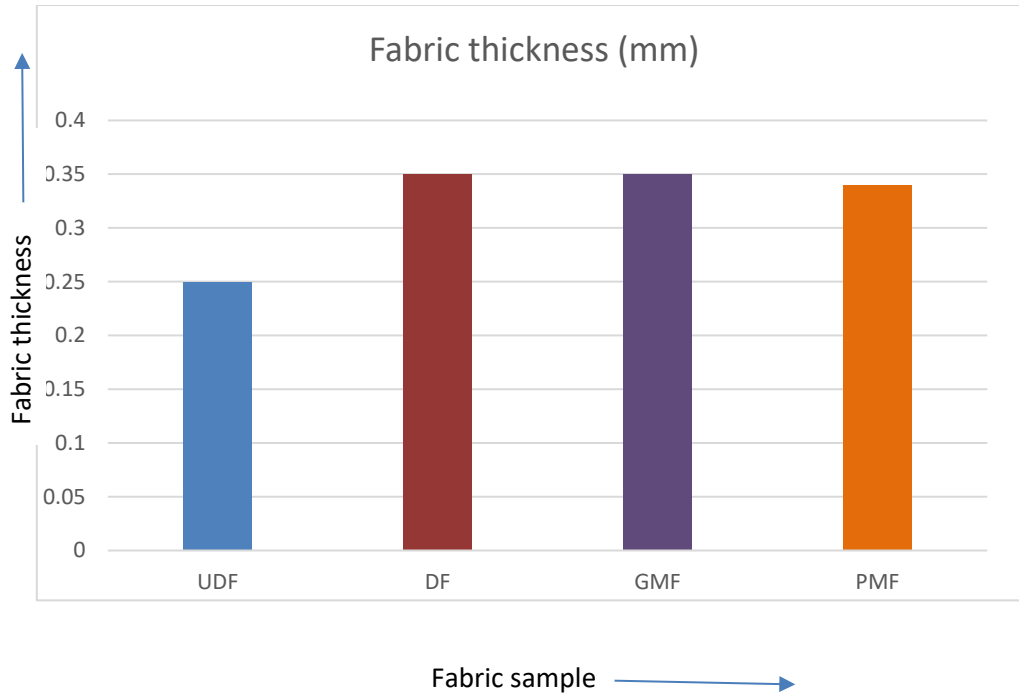


Figure-20 Fabric thickness

4.6.3 Fabric stiffness

Fabric stiffness warp direction

The stiffness of samples in warp direction are analysed and the results are presented in Table XXI

Table XXI

Fabric stiffness along warp direction

| S.no | Sample | Mean fabric weight (cm) | Gain or loss over original | % gain or loss over original | T- test |
|------|--------|-------------------------|----------------------------|------------------------------|----------|
| 1. | UDF | 2.72 | - | - | 0.067268 |
| 2. | DF | 2.88 | 0.13 | 4.51 | |
| 3. | GMF | 2.92 | 0.17 | 5.82 | |
| 4. | PMF | 2.4 | 0.35 | 14.58 | |

Table XXI , it shows that the undyed fabric has lower stiffness value in warp direction compared to both dyed and mordant dyed fabric. Dyed fabric showed increase of stiffness in warp direction the dyed fabric and lower than mordant dyed fabric. The "t" table result proves that the increase in fabric stiffness alone Warp direction is found to be significant.

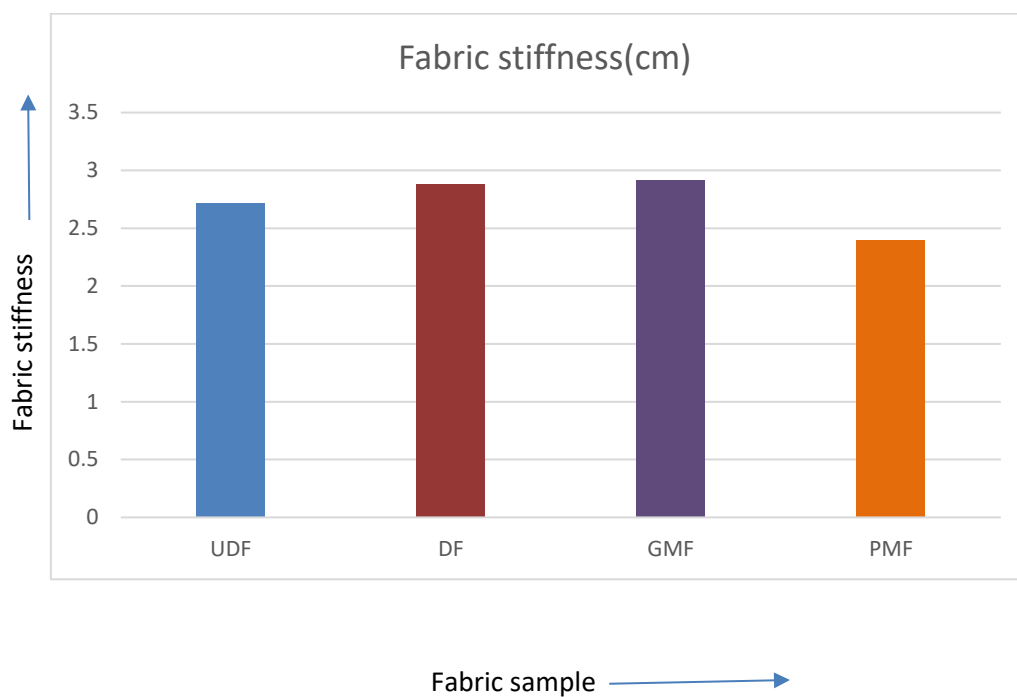


Figure-21 Fabric Stiffness

Fabric stiffness Weft direction

The stiffness of the samples along weft direction are analysed and values are presented in Table XXII

Table XXII

Fabric stiffness along weft direction

| S.no | Sample | Mean fabric weight (cm) | Gain or loss over original | % gain or loss over original | T-test |
|------|--------|-------------------------|----------------------------|------------------------------|----------|
| 1. | UDF | 2.25 | - | - | 0.093361 |
| 2. | DF | 2.30 | 0.05 | 2.17 | |
| 3. | GMF | 2.37 | 0.12 | 5.06 | |
| 4. | PMF | 2.63 | 0.38 | 14.44 | |

From Table XXII, it is clear that the undyed fabric showed lower stiffness value in weft direction. Among the dyed fabrics, PMF showed highest stiffness which might be due to increased dye uptake followed by GSM. Also, mordant fabric shows increase in weft direction compared to both undyed and dyed sample. The "t" table result proves that the increase in fabric stiffness alone Weft direction is found to be significant.

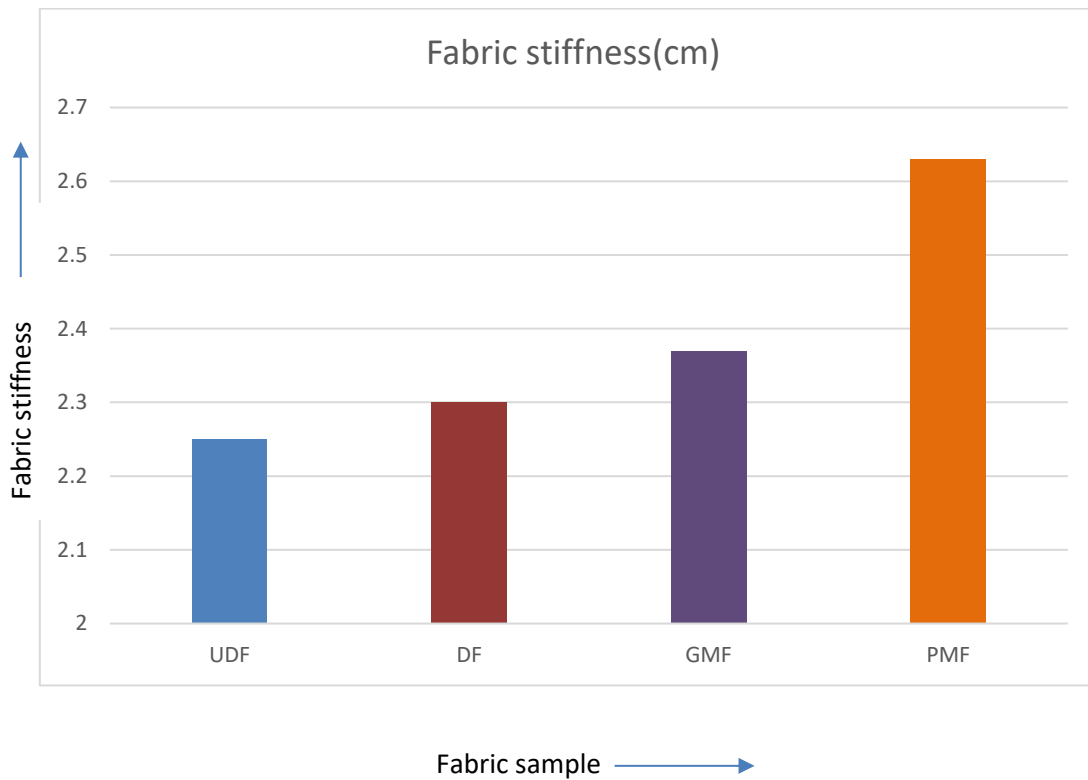


Figure-22 Fabric stiffness-weft

4.6.4 Absorbency

4.6.4.1 Sinking time

The results of fabric sinking time of undyed, dyed and mordant dyed fabric are presented in Table XXIII

Table XXIII

Sinking time

| S.no | Sample | Mean fabric sinking time (seconds) | Gain or loss over original | % gain or loss over original | T-table |
|------|--------|------------------------------------|----------------------------|------------------------------|----------|
| 1. | UDF | 10 | - | - | 0.063778 |
| 2. | DF | 12 | 2 | 16.66 | |
| 3. | GMF | 15 | 5 | 33.33 | |
| 4. | PMF | 11 | 1 | 9.09 | |

From Table XXIII, it is clear that the time taken for sinking of dyed fabric increased when compared to their undyed and mordant dyed fabric. The sinking time of undyed and mordant dyed fabric was found to be decreased than dyed fabric. From above absorbency test, it can be concluded that absorbency was found to be increased in all dyed fabrics. The "t" table result proves that the increases in fabric sinking is found to be significant.

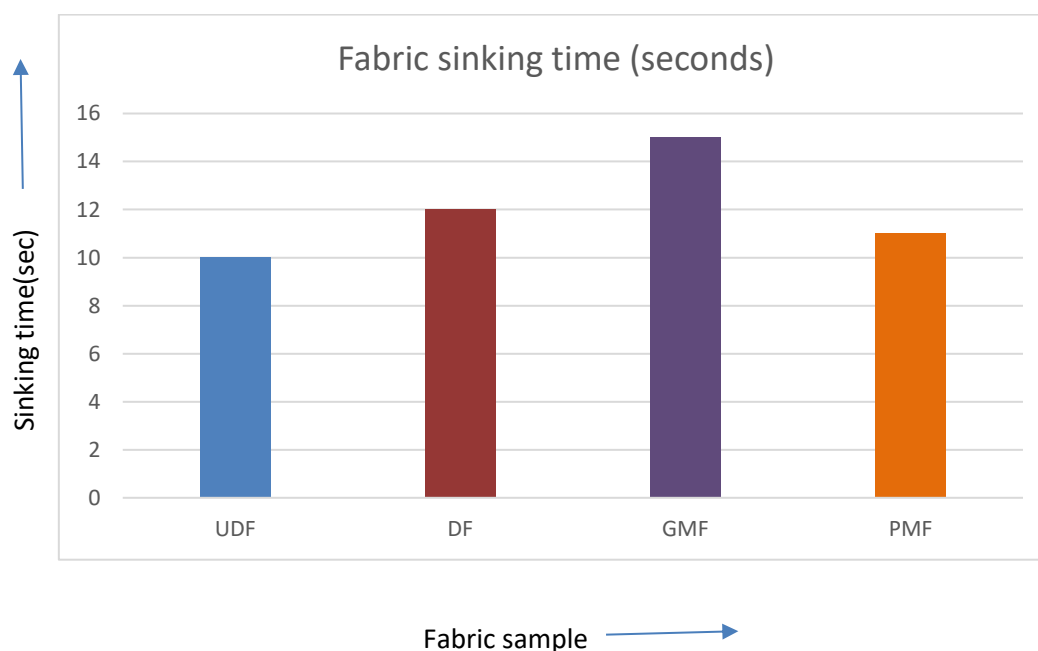


Figure-23 Sinking

4.6.4.2 Wicking

Table XXIV

Wick ability of fabric

| S.no | Sample | Mean fabric wick ability (cm) | Gain or loss over original | % gain or loss over original | T-table |
|------|--------|-------------------------------|----------------------------|------------------------------|----------|
| 1. | UDF | 1.8 | - | - | 0.079056 |
| 2. | DF | 2.2 | 0.4 | 18.18 | |
| 3. | GMF | 2.5 | 0.7 | 28 | |
| 4. | PMF | 6.5 | 4.7 | 72.30 | |

From Table XXIV, it is evident that the dyed sample increased when compared to undyed fabric. Mordant dyed fabric exhibited higher absorbency when compared to dyed and undyed fabric. The "t" table result proves that the increase in fabric wick ability is found to be significant.

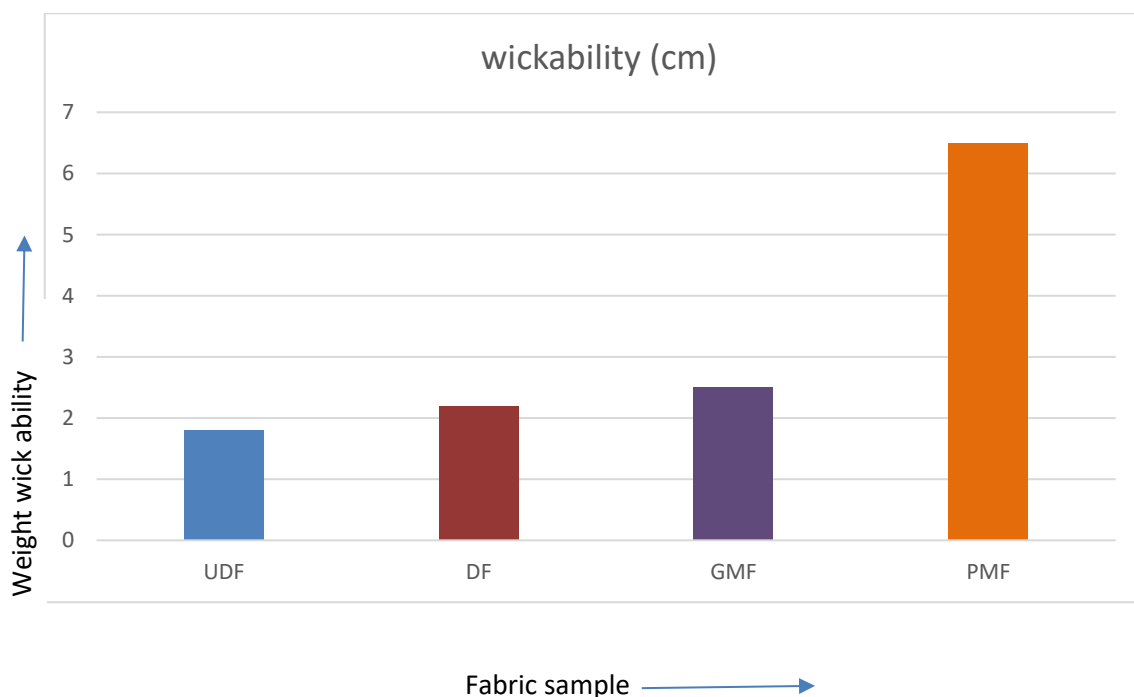


Figure-24 Wickability

4.6.5 Colour fastness to washing, sunlight and crocking

Natural dyed cotton fabric colour fastness properties are assessed to washing, sunlight and crocking and results are presented in Table XXV

Table XXV

Colour fastness to washing, sunlight and crocking

| S.no | Sample | Washing | Sunlight | Crocking | |
|------|--------|---------|----------|----------|-----|
| | | | | Dry | Wet |
| 1. | DF | 2 | 3 | 4 | 3 |
| 2. | GMF | 3 | 4 | 4 | 3 |
| 3. | PMF | 3 | 5 | 5 | 3 |

5- Excellent, 4- Good, 3- Moderate, 2- Poor, 1- Very poor

From Table XXV, it is evident that the of dyed and mordant dyed fabric showed good fastness properties. However un-mordant natural dyed samples showed poor to fair fastness properties. Mordant treated fabric showed good fastness properties to washing and rubbing than dyed fabric. Dyed and mordant dyed fabric exhibits fair to good fastness properties to rubbing.

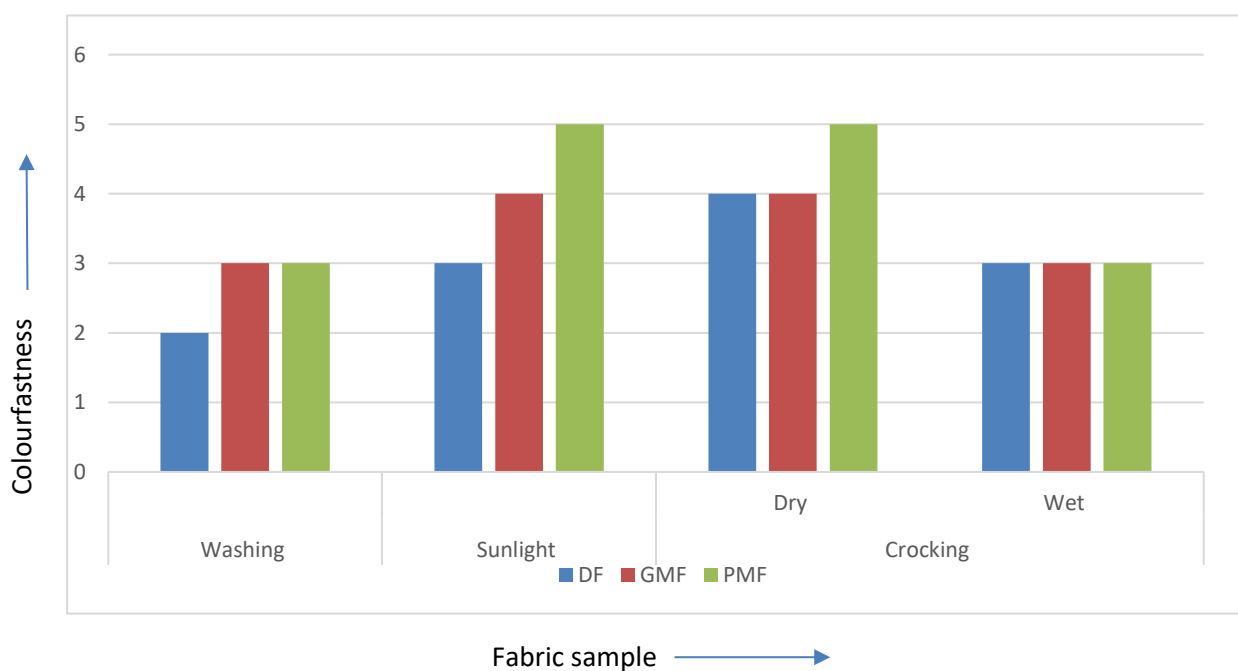


Figure-25 Colour fastness

4.7 Assessment of functional properties

4.7.1 UPF Analysis

The UPF rating and percentage of transmission for the control fabric, dyed cotton fabric with and without mordant using *Borassus flabellifer L.* extract, are presented in Table XXVI, Cotton fabric dyed with fruit extract shows a moderate UPF rating.

Table XXVI

UPF Analysis

| SAMPLE | COTTON FABRIC | | | GRADE |
|--------|---------------|-------|------------|-----------|
| | UV-A % | UV-B% | UPF RATING | |
| UDF | 96.21 | 95.84 | 22 | Good |
| DF | 96.74 | 97.30 | 35 | |
| PMF | 97.58 | 97.81 | 44 | |
| GMF | 98 | 98.27 | 55 | Excellent |

Due to its superior light resistance compared to other materials, the textile material is used to provide UV protection. UV-A (320–400 nm), UV-B (290–320 nm), and UV-C (200–290 nm) radiation are the three types of ultraviolet radiation, and the fabric's high surface area functions as a screen against them. UV-A and UV-B are responsible for determining UV protection and ultra-protective factors.[Faizan Shafiq, 2021] Dyeing using *Borassus flabellifer L.* in the presence of guava and pomegranate as mordants greatly increases the UPF protection and transmission percentage due to the presence of bio active molecules present in the source

4.7.2 Antibacterial activity

Antibacterial activity of *Borassus flabellifer L.* fruit extract finished fabric against *Staphylococcus aureus* and *E. coli* was assessed. The zones of inhibition of undyed fabric, dyed fabric, mordanted fabric, and fabric dyed using both pomegranate rind and guava leaf are presented in Table XXVII

Table XXVII

Zone of inhibition against bacterial isolates

| Samples | Zone of inhibition(mm) | |
|---------|------------------------|---------|
| | S. aureus | E. coli |
| GMDF | 14 | 15 |
| PMDF | - | - |
| DF | - | - |
| GMF | 16 | 20 |
| PMF | 18 | 18 |
| UDF | - | - |

The antibacterial potential of the dyed fabric is evaluated by measuring the diameter of the zone of inhibition against pathogens.

In antibacterial studies, the zones of inhibition produced by GMDF was 14 mm and 15mm. GMF was found to have a moderate zone of inhibition for S. aureus (16mm) and a maximum for E. coli (20mm). The PMF exhibits maximum antibacterial activity against S. aureus (18mm) and E. coli (18 mm). No zone of inhibition was recorded in PMDF, DF and UDF against the selected bacterial isolates.

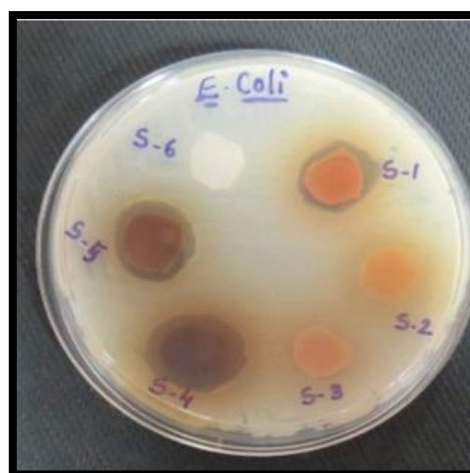


Plate-10,11 Fabric sample tested against S.aureus and E.Coli

5. SUMMARY & CONCLUSION

Product safety knowledge and awareness among consumers are growing. (Waraphorn,2016; Verma,2016). In recent years, there has been a growing effort to switch to more ecologically friendly reagents and catalysts in place of hazardous and polluting ones. (Chia,2019; Narayanaswamy,2013)

The demand for textile goods has grown recently, which has led to an increase in the usage of synthetic dyes in the textile sector. Because of their benefits, including higher substantively, improved durability, and a wide range of applications, synthetic dyes are frequently used. Synthetic dyes because a number of human ailments when released into the environment (air, soil, plants, and water) in untreated or only partially treated forms.

Alternatively, natural dyes are seen as environmentally friendly because they are biodegradable and renewable, and they also offer a rich variety of dyestuffs. They are gentle on the skin and may benefit the wearer's health. Textiles, beverages, food, pharmaceuticals, cosmetics, printer ink, home goods, and other commodities are all coloured using natural dyes. Without using any chemicals, a range of textile fibres was coloured using natural colourants originating from plant, animal, mineral, and microorganism sources. (Muthu, 2017; Kumar,2018)

Other than the colourant, the phytochemicals present in the dye have functional properties such as antibacterial, UV protection, antioxidant, and anticarcinogenic that are present in plants. Natural dyes have some limitations: they are difficult to reproduce colour, require skilled labour for dyeing, are expensive, and have poor colour fastness. Moreover, processes for the extraction and dyeing of fabric using natural colourants are time-consuming.

Mordants are used to improve the dye uptake of natural dye. Mordants help to increase the physical qualities of the fabric, such as colour brightness, fastness to light, washing, and rubbing. Without mordant, dyed fabrics have low fastness properties because they have a low affinity for the fabric. Mordants are used to create a wide range of hues similar to or different from the dye source.

The market impact of environmentally friendly dyes is anticipated to grow in emerging nations like India over the next few years in both the industrial and small-scale segments. (Baliarsingh

et al., 2012). Without sacrificing the quality and quantity of dyeing, the appropriate extraction method and dyeing technique should be followed to establish natural dye at the industrial level. (Kasiri et al. 2013). In a standardised dyeing technique, conditions for dye extraction and dyeing are optimised based on variables including time, temperature, and dye source concentration. These are referred to as "standardisation techniques (Chakrabarti et al., 2011). Standardising the extraction process and maximising the extraction variables are important from a technical and commercial point of view in terms of colour yield, extraction process cost, and dyeing process cost (Mansour et al., 2019).

Hence, the present study, "Eco-friendly natural dye from *Borassus flabellifer L.* fruit for coloration on cotton fabric," was designed with the following objectives:

- To extract dye from the *Borassus flabellifer L.* fruit
- To optimise dye extraction and dyeing conditions
- To evaluate the dyed fabric for physical and functional properties

Experimental procedure

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

- The fabric 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 was carried out as a pre - treatment step to remove starch and other impurities from cotton fabric.

- The dye source selected for the study was *Borassus flabellifer L.* fruit commonly known as Palmyra Palm fruit. The dye was extracted from *Borassus flabellifer L.* fruit by alkaline extraction.

- The mordant selected for the study was pomegranate rind and guava leaf powder.

- Various parameters such as solvent (water, NaOH, ethanol), dye source concentration (2%10%), time (15- 75 min) and temperature (30°C - 100°C) were optimized for dye extraction.
- Optimization of dyeing conditions such as time (15min – 75min),, temperature (30°C- 100°C), material liquor ratio (1:10 – 1:50) were done.
- Colour yield of the optimized dye extraction was measured using UV-Visible spectrophotometer.
- Fabric was treated with selected mordant with various mordanting techniques such as pre mordanting, simultaneous mordanting and Post mordanting.Dyeing was done at optimized conditions.
- Colour strength of the optimized dyed fabric was measured using premier colour scam spectrophotometer.
- Physical properties of control fabric dyed with mordant and fabric dyed without mordant, fabric thickness, weight, stiffness and absorbency through wicking and sinking was assessed for all the dyed fabrics.
- Functional properties of the fabric such as Ultraviolet Protection, Antibacterial and Antioxidant property were studied
- Colour fastness properties of fabric to washing, crocking and light were analysed.

Findings of the study

- Alkali was found to be suitable solvent for dye extraction.
- Concentration of 6% (Borassus flabellifer L. fruit extract) was found to be optimum concentration for dye extraction.
- Optimum dye extraction time was found to be 45 min.
- Dye extraction was found to be maximum at 100°C

- The optimum material liquor ratio selected for dyeing cotton fabric was 1:20
- Optimum dyeing time and temperature was found to be 60 min & 100°C respectively.
- The dye uptake was found to be increased after using mordant (pomegranate rind powder and guava leaf).
- The maximum uptake of dye was found in pre-mordanting technique.
- UV protection analysis showed good protection for dyed fabric without mordant and Moderate for the mordant dyed fabric.
- The antioxidant level for dye extract was excellent
- GMF and PMF shows good antibacterial against *S. aureus* and *E. coli*
- Weight of the fabric was found to be increased in mordant dyed fabric (MDF).
- The thickness of the mordant dyed fabric (MDF) was 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 regard to colour fastness tests to washing, sunlight and crocking, the mordant dyed fabrics showed good fastness properties whereas the fabric dyed without mordant showed moderate fastness properties.
- In absorbency test, the wicking ability of mordant dyed fabric (MDF) was found to be increased. The sinking time was found to be increased in dyed fabric (DF).

Conclusion

From the study, it can be concluded that cotton fabric dyed with *Borassus flabellifer* L. fruit extract can be a potential natural dye source for textile dyeing. The processes of extraction and dyeing of colourants and mordants used are environmentally friendly, cause minimum environmental pollution. The dye exhibited good fastness properties. The study reveals that the extraction and dyeing parameters have a significant influence on the colour characteristics and quality of the fabric. Based on the UV-Vis spectrometer, the optimised extraction conditions were: solvent as alkaline, dye source concentration of 6%, extraction time of 45 min, and temperature of 100°C, while the most suitable dyeing parameters were selected based on k/s value: MLR of 1:20, dyeing time of 60 min, and temperature of 100°C was found to be optimum. Excellent fastness properties were achieved with the use of the pre-mordant technique. Other than the colourant, *Borassus flabellifer* L. has high medical value and other functional properties like antibacterial, antioxidant, and UV-protective properties. This work clearly indicates that the extract from *Borassus flabellifer* L. fruit extract could be used as a source for natural dyes and for dyeing cotton fabric.

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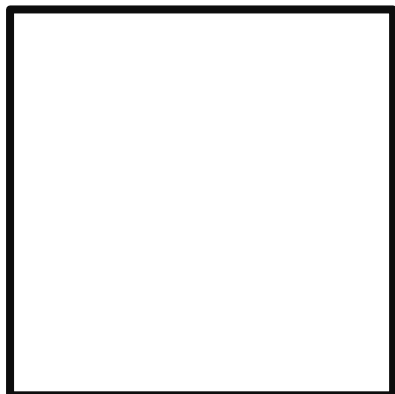
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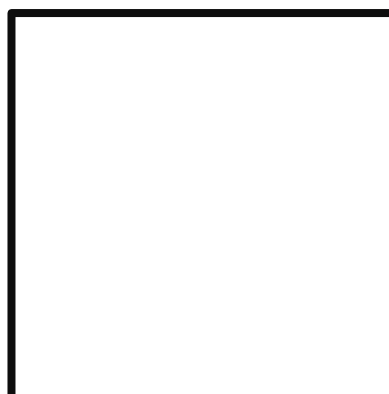
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APPENDIX-I

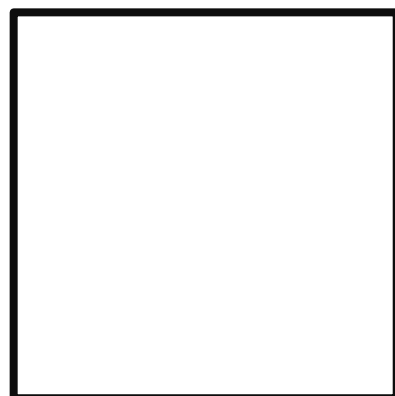


Desized cotton fabric

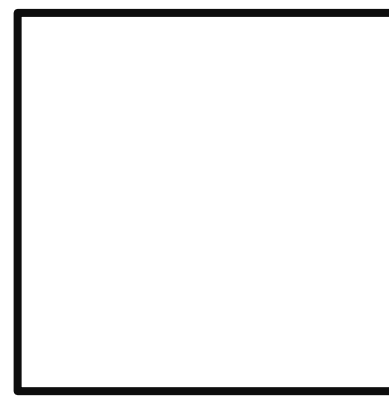


Fabric dyed using *Borassus flabellifer*

L. Fruit extract



**Dyed fabric with mordant-
Pomegranate rind**



**Dyed fabric with mordant-
Guava leaf**