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Eco-friendly natural dyeing using *Dillenia indica* leaf to produce functional silk fabric.

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Abstract Eco-friendly dyeing of fabrics with natural dyes is becoming more and more popular all over the world as a way to stop the harm that synthetic colors cause to the environment. This work used an ultrasonic extraction process to extract dyes from *Dillenia indica* (*D. indica*) leaves, which were then applied to silk fabric. The *D. indica* dyes were extracted and silk fabrics were dyed using the ultrasonic technique. In this case, *D. indica* powder was diluted at ratios of 1:15 and 1:10 liquid to produce the light and dark colors respectively. The dyed silk fabrics received a good rating based on an assessment of their color fastness under light, rubbing, and washing. The color strength of the dyed silk fabric was correctly represented by the spectrophotometric analysis, which also disclosed the L* (lightness or darkness), a* (redness or greenness), b* (yellowness or blueness), C* (chroma), h* (hue), R% (reflectance), and K/S (color strength) values. Several characterization techniques, such as Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), were used to better understand the relationship between *D. indica* dye and silk fibers. The results of the characterization process verified that the *D. indica* dyes were successfully deposited onto the silk fabric. Comparable testing results for silk fabrics, both dyed and undyed, anti-microbial analysis, and thermogravimetric analysis (TGA), were all within an acceptable range. The major result of this research was that chemicals were not used in the dyeing or extraction of silk fabrics. The fact that no chemicals were used in the extraction or coloring process makes this procedure absolutely chemical-free and environmentally beneficial. The natural dye that was produced from *D. indica* is therefore very promising and might be an appropriate option for the textile dyeing industry's sustainable silk fabric dying.

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Abbreviations

- SEM - Scanning Electron Microscopy
- TGA - Thermo-Gravimetric Analysis
- DSC - Differential Scanning Calorimetry
- FTIR - Fourier Transform Infrared Spectroscopy
- K/S - Kubelka-Munk theory
- CMC – Color Measurement Committee
- MI – Metamerism Index

INTRODUCTION

1.1 Purpose and Significance of the study

1.1.1 Natural Dyes, *Dillenia indica* & it's importance

For centuries, people have appreciated the sustainable and eco-friendly properties of natural dyes that come from plant sources. Tropical fruit trees such as *Dillenia indica*, also called Chalta or Elephant Apple, are indigenous to South and Southeast Asia. *Dillenia indica* has drawn interest for its potential as a natural dye source in addition to its culinary and medicinal uses.

Beautiful colours can be found all around us in nature, drawing our attention. Since ancient times, people have dyed their bodies, food, cave walls, textiles, leather, and everyday items with natural dyes. It has been determined that dyes and pigments can be extracted from a wide variety of plant, animal, insect, and mineral sources. Dyeing is an ancient art form that dates back to human civilization. The discovery of dyed textiles during archaeological digs in many locations across the globe demonstrates the existence of dyeing as a technique among prehistoric societies.

Natural dyes are important because they are environmentally friendly and do not cause the same problems as synthetic dyes. It's possible that *Dillenia indica* contains substances that can be used to extract dyes, offering the textile and craft industries a more environmentally friendly option.

1.1.2 Natural dyeing process (leaf, bark, plant) and their drawbacks

By using plant-based dyes, we can harness the rich botanical diversity for sustainable coloring techniques while simultaneously lowering the ecological impact. These dyes are often affordable, provide a large variety of colours, and have good fastness properties. However, they can also be toxic, allergic, or even carcinogenic, which puts people and the environment in hazards [1]. The significance of *Dillenia indica* goes beyond its potential use as a source of dye. Many parts of this adaptable plant are used medicinally in the area, giving it cultural and traditional significance. The incorporation of *Dillenia indica* into the natural dye industry is in line with sustainable practices

and emphasizes the value of recognizing and conserving the various applications of plants in various cultural contexts.

The process of natural dyeing entails removing colorants from plant components such as bark and leaves. To release the pigments, these materials are crushed and then boiled. To improve colorfastness, the fabric is prepared by mordanting it with metallic salts or minerals. Following its immersion in the dye bath, the fabric is dyed a variety of colors, ranging from earthy tones to vivid hues. This age-old, environmentally friendly method of dying ties art to sustainability and is in line with the growing interest in eco-friendly textile arts techniques.

The disadvantages of natural dyeing include a more muted color spectrum, limited color fastness, erratic colors, resource intensity, and environmental issues with mordants. In order to create a more viable and sustainable practice, ongoing research aims to address these limits.

Compounds in this plant may have applications as natural dyes in the future. To increase the variety of natural color sources available, researchers and craftspeople frequently investigate lesser-known plants for their ability to dye.

1.1.3 Plant selection and why it was used in this work

Selection of *Dillenia indica* may also be influenced by regional availability and cultural significance. When used for dye extraction, this plant is a sustainable and locally sourced product in areas where it is widely distributed. Krishnan et al. investigated the use of the leaf extraction of *D. indica* as a source of selenium nanoparticles with potential insecticidal and antimicrobial properties against vector mosquitoes and pathogenic microbes [2].

Furthermore, it's common practice to experiment with various plant species when using natural dyes because this leads to the discovery of special colors and qualities. Even though *Dillenia indica* isn't a typical choice, it might have been made in an effort to discover this plant's unrealized potential for natural dyeing and bring some novelty to the procedure.

Although *Dillenia indica* leaves have a high concentration of bioactive substances like flavonoids and tannins, they are used as natural dyes. These substances interact with textile fibers as natural colourants and have binding qualities that allow them to do so, producing a dye that is both long-lasting and useful. Furthermore, the growing desire for sustainable and environmentally friendly textile methods is in line with the eco-friendliness of natural dyes, such as those derived from *Dillenia indica* leaves.

Additionally, the usage of *Dillenia indica* for natural dyeing is in line with the textile industry's growing awareness of and demand for non-toxic, plant-based alternatives. This method not only produces colorful, useful textiles, but it also encourages the manufacturing of textiles in a way that is more ecologically friendly and sustainable.

The leaf extract of *Dillenia indica* yields a variety of colors, ranging from green to yellow, contingent upon the mordants included in the dyeing procedure. The distinctive and earthy tones produced by these natural dyes add to the allure of fabrics.

1.1.4 Silk fabric and its characteristics & functionalization

Silk fabric, known for its luxurious feel and natural sheen, is derived from the fibers produced by silkworms during the process of silk production. The intrinsic qualities of silk fabric can be further enhanced by functionalization techniques. This entails applying finishes, printing, and dyeing processes to alter characteristics like color, pattern, flame retardancy, and wrinkle resistance. Silk is infused with biological techniques, enzymes, and nanoparticles to impart antimicrobial, UV-blocking, or temperature-regulating properties. These techniques broaden the range of uses for silk, enabling it to be used in everything from fashion to medical textiles.

For natural dyeing, silk cloth is preferred for a number of reasons. First of all, when dyed with natural extracts such as those derived from plants, silk naturally absorbs and holds onto colour, displaying rich and vivid tones. Silk fibers have a smooth, densely woven structure that improves color adhesion and penetration during the dyeing process.

Second, silk binds natural dyes more effectively because of its protein-based makeup, which increases its receptiveness to different mordants. This makes the dye on silk more colorfast and long-lasting than it would be on some other materials.

In addition, the luxurious and glossy texture of silk combines well with the delicate colour shifts generated by natural dyes to provide visually appealing and distinctive textile results. All things considered; silk's inherent qualities make it a great option for anyone looking for premium, naturally colored textiles.

Due to the fact that silk is made mostly of proteins, it is thought to be an ideal fabric for natural dyeing with *Dillenia Indica* leaves. Better absorption of the natural dye is possible due to silk fibres' innate propensity for interacting with the bioactive chemicals found in *Dillenia Indica*. Silk textiles bear vivid and durable colours as a result. Silk's silky feel adds a sumptuous touch while

also improving the dyed fabric's overall appearance. However, silk is a popular material for natural dyeing techniques, such as those that use *Dillenia Indica* leaves, due to its resilience and wide colour spectrum.

1.2 Aim of the research

This thesis' main goal is to research sustainable textile dyeing techniques using the natural properties of *Dillenia indica* leaves on silk fabric. The research intends to provide an environmentally friendly substitute that complies with green chemistry principles in considering the growing concerns regarding the effects of traditional dyeing methods on the environment.

It's not just about giving the silk brilliant colours; it's also about giving the cloth practical qualities. The research aims to utilise the fundamental advantages of *Dillenia indica* leaves, such as enhanced durability, UV resistance, or antibacterial traits, to improve the performance of silk through a thorough exploration of these properties. This two-pronged technique takes care of the fabric's practical and aesthetic qualities.

The result is important not only for the textile sector but also for the larger paradigm change towards sustainable practices. The initiative aims to reduce the environmental impact of traditional dyeing methods by developing a methodology for eco-friendly dyeing utilising natural resources. The ultimate goal is to offer a concrete illustration of how methods inspired by nature may be used to produce useful and aesthetically beautiful silk fabrics, encouraging a more sustainable future for the textile sector.

1.3 Objectives

To illustrate the possibilities and advantages of using *Dillenia indica* leaves as a natural silk fabric dye, resulting in the creation of practical, eco-friendly textiles with less of an adverse effect on the environment.

- 1.To encourage environmentally friendly and sustainable dying techniques, look into the possibility of *Dillenia indica* leaves as a natural silk fabric dye.
2. Examine the silk fabric colored with *Dillenia indica* leaves for colour stability and fastness, making sure that vivid and durable colours are produced.
3. Research the eco-toxicological effects of dyeing with *Dillenia indica* on the environment with the goal of fully comprehending its ecological footprint.

4. Analysis how various mordanting methods affect the dyeing process to improve the silk fabric's capacity to retain color and withstand fading.
5. Assess the antibacterial qualities of silk fabric colored with *Dillenia indica* leaves, aiding in the creation of textiles that are both practical and health-promoting.
6. Examine the viability from an economic standpoint of adding *Dillenia indica* leaves to the silk dying process, taking into account the product's cost-effectiveness and possible market demand for environmentally friendly goods.
7. Examine whether *Dillenia indica* leaf dyeing is scalable in order to create a technique appropriate for mass-production of textiles.
8. In order to achieve optimum dying conditions, investigate the effects of different dyeing parameters (temperature, time, concentration) on the final qualities of silk fabric dyed with *Dillenia indica*.
9. Consider how using natural dyes derived from *Dillenia indica* can improve the environment by reducing waste production, energy use, and water consumption.
10. Create policies that encourage the use of *Dillenia indica* leaves in silk dying procedures, encouraging the adoption of environmentally and socially responsible methods by textile makers.

1.4 Research Questions

The research questions are given bellow:

- Why was *Dillenia indica* leaf utilized as a natural dye rather than a synthetic one?
- Which method of extraction was applied to the *Dillenia indica* leaf in order to extract the natural dyestuff?
- Which dyeing technique was applied to the silk cloth for the thesis project?
- What tools were utilized to extract and color the *Dillenia indica* leaf?
- What effects do the color fastness characteristics of a dyed fabric sample have on dyeing?
- What shade does L*, a*, and b represent in the CMC analysis of the colored fabric?
- What are the findings from the dyed fabric sample's SEM and FRIR analyses?
- What are the colored fabric's thermal characteristics, air permeability, and bursting strength analyses?
- What makes natural colors an acceptable substitute for synthetic ones?

- What alternatives exist for natural dyestuff in the event that enterprises produce huge quantities of them?

1.5 Research Limitations

The limitations of the research are given bellow:

- Standardizing an appropriate recipe for natural dyes is challenging because the ingredients and the colour component are both necessary for the natural dyeing process and the ensuing colour development.
- Typically, the process of extracting natural dyes can be costly and time-consuming.
- In the case of natural dyeing, there is a deficiency of specific scientific information regarding extraction and dying procedures.
- The availability of the equipment needed for the characterization of the colored cloth sample is also the primary constraint.

LITERATURE REVIEW

2.1 History of natural dyes

Natural dye is the most common and available ingredient in our environment for coloring textile materials. Natural dyes, discovered through the ingenuity and persistence of our ancestors, can resist brightly for centuries or millennia and may be found hidden in such diverse places as the roots of a plant, a parasitic insect and the secretions of a sea snail [1]. Only a few decades ago did the textile industry switch to synthetic dyes, but they were so effective at the time that natural dyes now make up only 1% of all dyes used globally [2]. And this is accurate despite the fact that employing natural colours has a long tradition in many countries. Whereas synthetic dyes were predominantly the domain of males, organic chemists, the interest in natural dyes in contemporary history seems to have been principally aroused by knowledgeable and adept women who continued and advanced home dyeing as a hobby - Furry and Viemont (1935) [3]. These women's books on home dyeing, which include detailed recipes, were published primarily in the USA. Natural dyes are only a viable solution for small-scale applications at the current state of scientific research, and they can only be used in conjunction with synthetic colors.

2.2 Dye Sources [4]

Five principal dyes are mentioned, which have been identified as:

- (a) archil (orchil), a purple colour derived from certain marine algae found on rocks in the Mediterranean Sea;
- (b) alkanet, a red colour prepared from the root of *Alkanna tinctoria*;
- (c) *Rubia tinctorum*, which generates red-coloured products;
- (d) woad (*Isatis tinctoria*), a blue colour obtained by a process of fermentation from the leaves;
- (e) and finally indigo from the leaves of the *Indigofera* species.

2. 3 Extraction Methods of Natural Dyes [5]

The main innovative extraction methods are listed as follows:

- Aqueous extraction,
- Alkaline or acid extraction,
- Extraction with microwave or ultrasonic energy,
- Fermentation,
- Enzymatic extraction,
- Solvent extraction, and
- Supercritical fluid extraction

2.3.1 Aqueous extraction

The traditional technique for extracting plants and other materials is called aqueous extraction. Firstly, colored materials are chopped, divided into small bits or powdered, and sieved to improve the extraction effect. Then, in stainless steel containers, colored powders are kept for a whole night or longer to allow the cell structure to relax. Stainless steel containers are employed when significant volumes of dye extraction are performed to purify powder materials, and the boiling procedure may shorten the wetting time.

2.3.2 Acid and Alkaline Extraction

Natural dyes usually include glycosides and can be extracted in diluted alkaline and acidic settings. For natural compounds having phenolic groups, alkaline extraction is appropriate [6]. Once extracted, natural colours can be precipitated using acids.

2.3.3 Ultrasonic and Microwave Extraction

These techniques employ microwave and ultrasonic radiation to increase the extraction process's effectiveness. Natural resources are processed as little as possible during microwave extraction, and the amount of solvent used is very minimal. Ultrasonic is the most effective dye extraction method where the liquor extracted by ultrasonic machine and then start the filtration process for overnight.

2.3.4 Fermentation

Enzymes made by microorganisms are utilized in this extraction process (Fig. 1). By using a fermentation technique, indigo dyes can also be extracted from other plants that contain indigo. The aqueous extraction process requires a high temperature, although the fermentation approach is similar to it. However, there are certain drawbacks to the fermenting process, including its lengthy duration, the transition phase that occurs right after harvest, and the microbial odor responses.

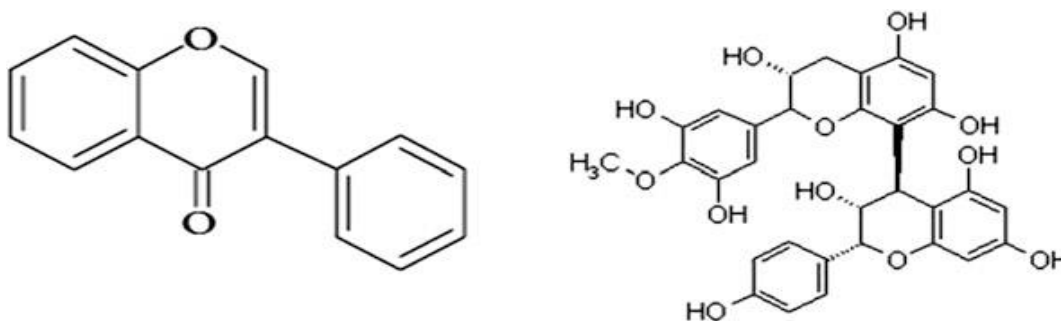


Fig. 1 Isoflavon and proanthocyanidin structure [7]

2.3.5 Enzymatic Extraction

Enzymes like cellulase, amylase, and pectinase can be used to extract the cellulose, starches, and pectin found in plant tissue, which is the basis for the extraction of natural dyes. In mild conditions, the extraction efficiency can be increased by using the enzymatic extraction method.

2.3.6 Solvent Extraction

Organic solvents like acetone, petroleum ether, chloroform, ethanol, methanol, and mixtures of alcohol and water may be preferred depending on the structure of natural coloring ingredients. The drawbacks of this approach are greenhouse gas effects and toxic residues. Problems arise when chlorophyll and waxy compounds are extracted simultaneously. (Fig. 2)



Fig. 2 Molded indigo dye [8]

2.4 Mordanting

A metal salt is referred to as a mordant in order to establish an affinity between the pigment or dye and the fabric. The mordanting procedure increases color fastness and affinity for dye and fiber molecules. Generally, there are three ways to apply the mordanting procedure

- a. Pre-mordanting
- b. Post-mordanting
- c. Meta-mordanting

2.5 Benefits of natural dye

Natural dyes come from sources such as plants, minerals, and animal products. Natural dyes were once used to color clothing, textiles, food, and other items. The following is a list of benefits of using natural dyes:

- ✓ Natural dyes are characterized and generally acknowledged as environmentally benign worldwide since they come from renewable sources.
- ✓ Natural dyes are safe for human skin; they don't cause allergies or cancer.
- ✓ When using the right mordants, natural dyes' fastness qualities can be tolerated.
- ✓ The waste from the natural dyeing process decomposes naturally.
- ✓ The source of synthetic dyes is non-renewable petroleum.
- ✓ In order for natural dyes to absorb UV radiation, many of them include strong UV protection.

2.6 Future of Natural Dyeing

At present, synthetic colors are taking the place of natural dyes due to growing environmental concerns worldwide. Adoption of natural dyes on a large scale is crucial for sustainability. Furthermore, with regard to fossil fuels, expanding the use of renewable raw materials is critical to safeguarding farmland and the life cycle. Synthetic pesticide use in organic agriculture is not the favored option. Natural dyes have not been able to meet the cost and technical demands of the twenty-first century, including reproducibility. Notwithstanding the drawbacks of natural dyes, it is important to ignore the growing array of environmental issues. organic colors lack the negative effects of synthetic colors, such as increased environmental pollution and health risks.

2.7 Importance of natural dyes

Dyes are the basic elements of dyeing textile material, among the natural and synthetic dyes, synthetic dyes are being used vastly for dyeing textiles worldwide. Because of using synthetic dyes in a large number, environment is getting polluted by the waste material produce from dye liquor. Above all, some of these dyes are toxic, carcinogenic and can cause skin and eye irritation.

To solve these problem natural dyes is the best substitute and can be commercially useable for long term uses of the material dyed using natural dyes. Dyeing using *Dillenia indica* leaves is the most beneficial and essential for all types of fabric, like cotton, silk, wool, viscos, nylon etc.

2.8 *Dillenia Indica* Leaf

Dillenia indica, commonly known as Elephant Apple or Chalta, is a tropical fruit tree native to Southeast Asia. Its leaves are large, glossy, and elliptical in shape. In traditional medicine, these leaves are used for various purposes, including treating digestive issues and skin conditions and dyeing with Textile materials. Additionally, the fruit of the tree is edible and sour in flavour, often used in culinary preparations and traditional remedies.

The purpose of this study was to use an ultrasonic extraction process to extract dyes from the leaves of the *Dillenia indica* (*D. indica*) tree for use on organic cotton garments. Both the *D. indica* dye extraction process and the dyeing of organic cotton garments were carried out using the ultrasonic method. Here, 5% and 6.67% of *D. indica* powder were employed to create the light and

dark shades, respectively. After the colour fastness of the coloured organic cotton clothing was assessed in respect to washing, rubbing, and light exposure, an exceptional grade was discovered. The spectrophotometric analysis, which also revealed the L* (lightness or darkness), a* (redness or greenness), b* (yellowness or blueness), C* (chroma), h* (hue), R% (reflectance), and K/S (colour strength) values, accurately depicted the colour strength of the dyed organic cotton.cotton textile. Several characterisation techniques, such as Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), were used to better understand the relationship between D. indica dye and organic cotton fibres.

The results of the characterisation process verified that the D. indica dyes were successfully deposited onto the organic cotton textiles. Comparable testing results for organic cotton fabrics, both dyed and undyed, including bursting strength, air permeability, and thermogravimetric analysis (TGA), were all within an acceptable range. One of the study's key conclusions was that organic cotton fabrics were extracted and dyed without the use of chemicals. Since no chemicals were used in the extraction or dyeing process, this method can be referred to as entirely chemical-free and environmentally beneficial. [9]

2.9 *Polyalthia longifolia*

Natural colours are nearly completely devoid of dangerous chemicals, eco-friendly, and biodegradable. The purpose of the study was to remove the natural dye from *Polyalthia longifolia* leaves. Finding the ideal circumstances for natural dye extraction is the aim of this investigation. Temperature, duration, and pH were the extraction parameters that were optimised according to their optical densities. Four different methods—aqueous, acidic, alcoholic, and alkaline medium—were used to extract the colour. The dyeing process decided to use alkaline extraction based on the dye yield % and their optical density. The best duration of 60 minutes at 90 degrees Celsius was determined for dye extraction; the dye output and M:L ratios were 18% and 1:1, respectively.

2.10 *Eucalyptus*

The evergreen hardwood genus that is native to Australia includes eucalyptus. Nine hundred species and subspecies are thought to exist. Additionally, eucalyptus has been successfully grown throughout the world, including the west coast of the United States, Asia, and southern Europe (Flint, 2007). One of the chief natural dye sources that produces yellowish-brown colourants is eucalyptus. According to Ali et al. (2007), eucalyptus colourant contains a significant amount of

natural tannins and polyphenols, ranging from 10% to 12%. The primary colouring agent found in eucalyptus bark is quercetin, an antioxidant. Its strong antioxidant qualities have made it useful as a food colouring (Vankar et al., 2006). Up to 11% of the major components of tannin (gallic acid [3,4,5-trihydroxybenzoic acid]) can be found in eucalyptus leaves. The minor components of tannin are flavonoids (quercetin [3,3',4',5,7-pentahydroxyflavone] and rutin [3,3',4',5,7-pentahydroxyflavone-3-rhamnoglucoside]) and ellagic acid [2,3,7,8-tetrahydroxy (1) benzopyrano (5,4,3-cde) (1) benzopyran-5,10-dione)] (Chapuis-Lardy et al., 2002; Conde et al., 1997).

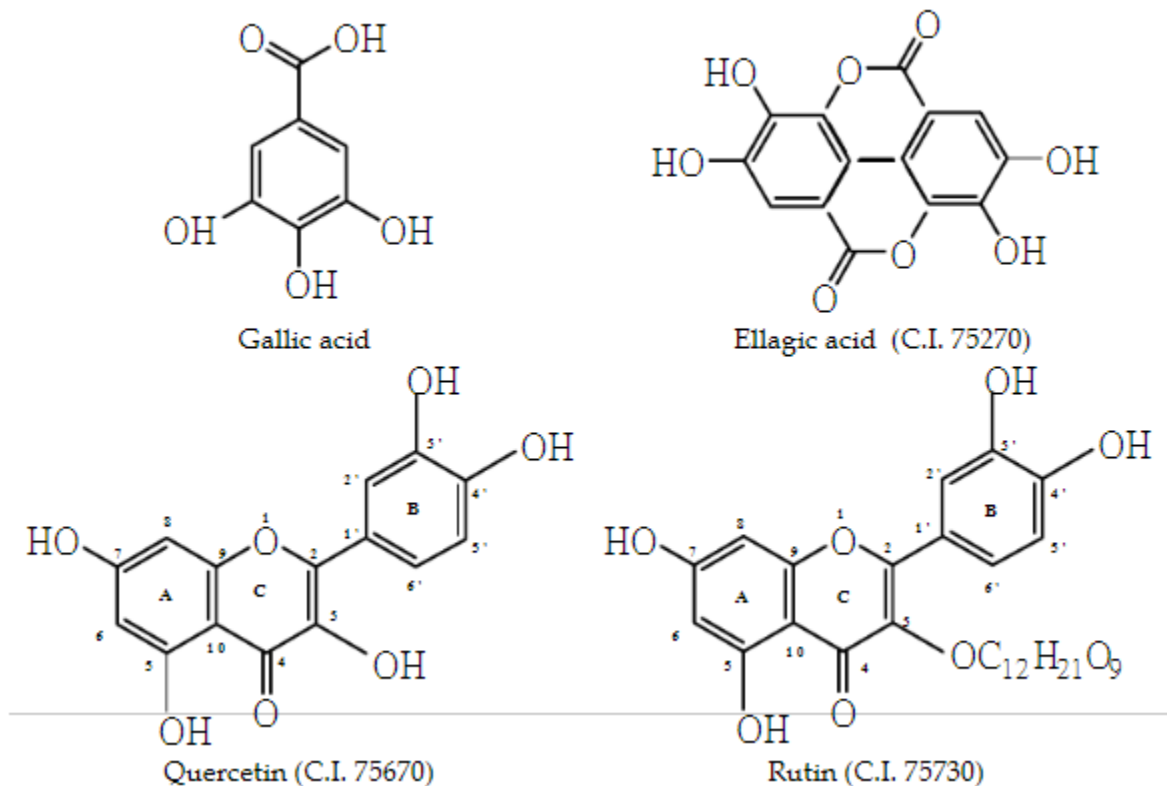


Fig. 3. Colour composition of eucalyptus leaf extract dye [11]

2.11 Dyeing with Tea leaves

It has been studied to dye cotton cloth using natural colourants derived from tea leaves. Several methods and solvents were used to carry out the dyeing process, which was then applied on Giza 86 Egyptian cotton fabric along with certain mordents and gelatin modification. Investigations were conducted into comparative study values and an evaluation of the dyeing properties on cotton fabric. The results obtained revealed that microwave technique using ionized water (DI) with ferrous sulphate as pre mordant after gelatin treatment gave the higher values compared with the other techniques, and the different solvents and mordants.

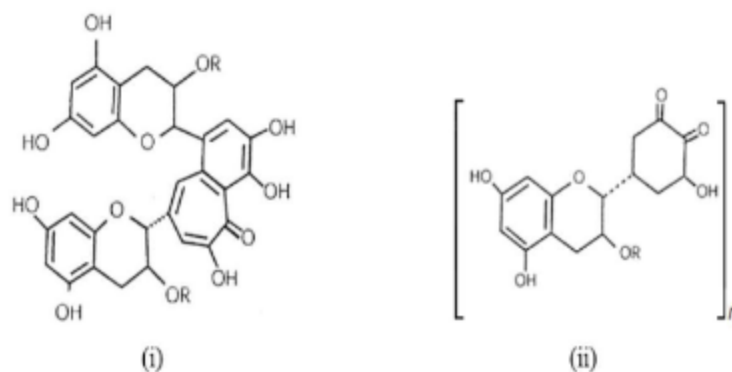


Fig. 4. (i) Theaflavins and (ii) Thearubigins - coloring component of tea (*Camellia sinensis*) [12]

2.12 Dyeing with Mango leaf

Beautiful colours can be found in many of the materials that nature provides. These days, one of the most crucial research topics for scientists is natural dyes. In this project, the dye was taken from mango leaves and used to dye polyester, cotton, and silk. The extraction medium was established by extending the pH value range of 3 to 12. It was discovered that pH 10 was the greatest relative colour strength of the isolated dye liquid. It was discovered that the ideal dye extraction parameters were 98 °C, 60 minutes, and a material-to-alcohol ratio of 1:10, in that order. Several kinds of mordants are employed in fixing. Since mordanting produces a more effective hue when natural dyes are used for dyeing. In this case, silk produces finer tints when various mordants are used, but deeper shades are produced when ferrous sulphate>alum, alum>tin>tannic acid. Compared to silk, cotton and polyester produce mediocre results and milder hues. However, utilising alum>tin Cotton produces darker colours than other mordants; polyester produces somewhat darker colours than other mordants when ferrous sulphate, alum, and tin are used.

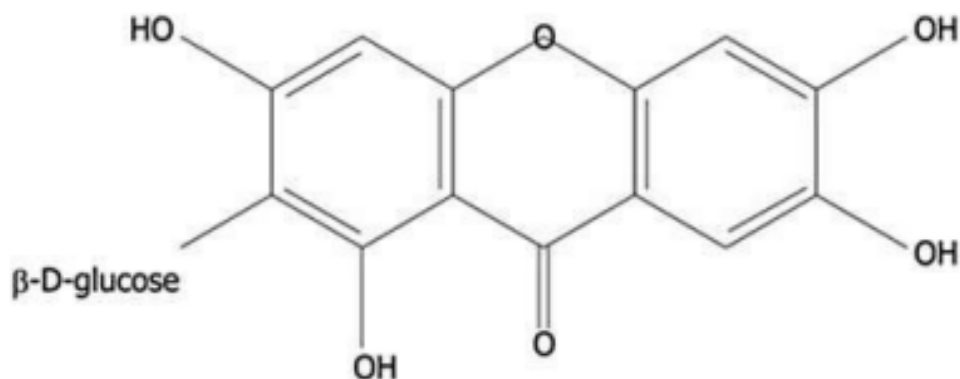


Fig. 5. chemical structure of mangiferin leaves (1, 3, 6, 7- tetrahydroxyanthrone-c-2-β- Dglucoside) [13]

2.13 Dyeing of silk with natural dyes

Eco-friendly dyes have gained much importance in dyeing of textiles, especially silk fabrics. The possibilities to extract dyes from plants and to optimize various dyeing variables of these dyes for dyeing of silk have been studied. .. The following natural dyes were chosen for the study: walnut, euphorbia, hamelia, jatropa, lantana, and kilmora. Firstly, silk was degummed before dyeing in order to remove sericin from the cloth, which prevents the colours from penetrating the fibre. The ideal dye concentration, extraction and dyeing times, concentration of the mordant, and mordanting techniques were all determined through a series of trials. Jatropa, Lantana, Hamelia, and Euphorbia dyes showed the highest % absorption in acidic conditions, although Kilmora and Walnut dyes performed well in alkaline media. A standard recipe for dyeing silk with each dye source is optimised based on the findings of several tests. [14]

2.14 Silk Dyeing with Natural Dye Extracted from Spice

In the textile processing sector, textile dyeing is crucial. Recent studies' findings indicate that hazardous substances such formaldehyde, pentachlorophenol, benzidine, aryl amine, lead, cadmium, zinc, mercury, and halogen carriers may be present in the chemicals used to make synthetic dyes. Such dyes are linked to allergies, skin irritation, lung issues, and skin cancer in addition to posing a risk to human and other living things' health when used in textiles.. Study in

the field of textile dyeing have focused on developing synthetic dye substitutes because of the negative impacts of synthetic dyes. Natural dyes don't harm the environment or cause adverse skin reactions. The purpose of this project is to extract natural dye from spices and apply it to textile materials. Acidic media was used to extract the dye. Fabric was mordanted using alum mordant to improve the dyeability of the textile substrate. The cloth in the experiment was mordanted using pre, post, and simultaneous methods. An investigation of the general colour fastness characteristics of coloured textile material has been conducted. [15]

2.15 Dyeing of silk using *Madhuca longifolia* as natural dye source

The possibility of dried *Madhuca longifolia* leaves as a natural silk dye source has been assessed. The dye has been extracted under ideal circumstances, including a pH of 10, a duration of 60 minutes, and a temperature of 95°C. After applying the extracted dye to the silk materials, several techniques were used, either with or without the use of mordants, to achieve a range of hues. It is discovered that dyed silk materials' colour is significantly influenced by the presence of mordants.

The dyed samples underwent routine wash, light, and rub fastness tests as well as colour measurements. Additionally, the extracted dye is examined using GC/MS and atomic absorption spectrophotometry for a few eco-parameters. The test results are compared with set standards to determine the eco-friendliness of natural dye. Their concentrations are found to be lower than the stipulated limits. The dyed samples are also tested for antimicrobial activity against Gram-positive and Gram-negative bacteria.

The silk materials that have been dyed exhibit satisfactory fastness characteristics and are discovered to have antimicrobial capabilities. According to the findings, *Madhuca longifolia* leaves have promise as a natural colourant. This could lead to the development of a new line of environmentally friendly textile material dyes. [16]

2.16 Indigofera Leaves as Natural Dyes for Silk Fabrics

The leaves of the *Indigofera* plant are used to make the naturally occurring blue dye indigo, which is neither carcinogenic nor hazardous to the environment. The purpose of this study was to evaluate the fastness of rubbing indigo-dyed silk materials with various fixators against sun heat, dry, wet, and acid. Along with determining the effects of hydrochloric acid concentrations and aeration duration on the amount of indigo, it also aimed to determine the reduction potential value of molasses and brown sugar as reducing agents. The Indian glycosides present in *Indigofera* leaves were degraded with hydrochloric acid to yield indoxyl and glucose, which were subsequently

utilised to extract the indigo pigment. Next, indoxyl was converted to indigo by oxidising it with molasses, a polysaccharide component. The concentration of 0.01 M hydrochloric acid produced the maximum indigo content, 26.83 ppm, according to the data, and 4 hours was the ideal aeration time to make indigo with a concentration of 22.32 ppm.

Different colours were produced by the fixator types' effects. A bright blue colour was created by alum, a light blue colour by limestone, and a dark blue-green colour by the ferrous sulphate fixator. The grey selling value of 4-5 indicated a slight fading, which was also supported by the fastness test

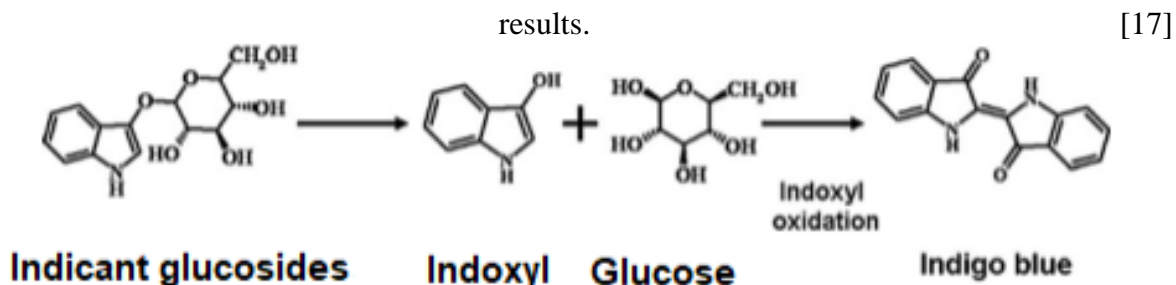


Fig. 6. Indigo color formation reaction

2.17 Indigoid Dyes

Blue indigoid colours made from the tropical *indigofera tinctoria* L. plant that grows throughout India. *Isatis tinctoria* L. and *Indigo suffraticosa*, two indoxyl molecules with identical chemical structures, combine to form the aromatic compound that makes up indigoid dyes [18]. In addition, *Isatis tinctoria* L., commonly known as "isatis tinctoria," is grown across Anatolia and Europe.

2.18 Quinone Dyes

Quinones are aromatic monocyclic and polycyclic components that are converted into cyclic ketones. Quinone groups are responsible for the production of color-quality chemicals. Quinones may be divided into three main categories such as benzoquinones, naphthoquinones, and anthraquinones [19].

2.19 Flavonoid Dyes

Flavonoids, sometimes referred to as colorific groups in botany, are substances that plants synthesize with a phenolic structure. According to the results, the dye extracted from venetian sumac is in fisetin structure and the color of the dye which is of antioxidant and antimicrobial quality is yellow-brown [20]. Citrus contains flavanones, the main class of flavonoids. The flavanone's chemical structure is provided in (Fig 2)

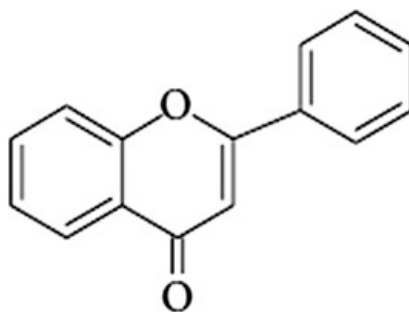


Fig. 8 Luteolin structure

2.20 Carotenoid Dyes

Annatto and other plant seeds contain the chemical structure of carotenoids. Moreover, carotenoid chemical groups are involved in the blooms of parijat (*Nyctanthes arbor-tristis*) [21].

2.21 Indigo Blues

One of the oldest and most widely used dyestuffs ever discovered by humans was indigo blue [22]. Despite being extremely costly to transport, it was valued for its quality as a dye throughout the major civilizations of Egypt, Greece, and Rome [23].

2.22 Ancient Purple

The prominence of those who wore purple fabrics in the past could never be matched by any other colour, artificial or natural. Both the Roman emperors and the powerful Catholic Church, who later represented the growing power, used the colour purple as a status symbol.

Three Mediterranean purple mollusks, namely *Murex brandaris*, *Murex trunculus*, and *Purpura haemastoma*, have had their names altered. They are now known as *Bolinus brandaris*, *Hexaplex trunculus*, and *Stramonita haemastoma*, respectively. The names were therefore changed to remove any reference to *Murex* and *Purpura*'s historical significance as dyes. [24]

CHAPTER THREE

RESEARCH DESIGN

3.1 METHODOLOGY

Dellenia indica leaves collected from our rural area. Leaves are dried under sunlight at least for 15-20 days. After that leaves are processed into powder by using blender. Then powder is poured into strainer and pure fine powder is collected from it. In 10 gm powder, maximum 100-150 ml distilled water is used in here such as 1:10/ 1:20/1:30/ 1:40/1:50. As example 1:10 & 1:50 ratio is taken for producing dark & light shade. Ultrasonic machine is used in here as dye extraction. And in here, time is taken 1-2 hours & temperature is taken 60-80°C.



Fig 1: Process step of dye extraction procedure from *dillenia indica*

To employ a specialised procedure to extract the dye from its source. Natural dyes are becoming more and more popular for eco-friendly textile dyeing as a way to reduce the harm that synthetic

color does to the environment. This work concentrated on using an ultrasonic extraction process to extract dyes from *Dillenia indica* leaves and apply them to silk fabric.

Both the *D. indica* dye extraction process and the dyeing of organic cotton garments were carried out using the ultrasonic method. Here, 5% and 67% of *D. indica* powder employed to create the light and dark shades, respectively. An outstanding rating was found after color fastness of the colored silk was examined in relation to washing, rubbing, and light exposure.

3.2 Methods

The fact that no chemicals were used in the extraction and coloring of silk fabrics is one of the study's key conclusions. Since no chemicals were used in the extraction or dyeing process, this method can be referred to as entirely chemical -free and environmentally beneficial. Both the *D. indica* dye extraction process and the dyeing of silk were carried out using the ultrasonic method. Have to apply a specific procedure to extract the dye from its source. Some techniques for removing natural colors from the sources are as follows:

The methods of extraction include aqueous, solvent, acid and alkali, supercritical fluid, ultrasonic-assisted, and enzyme-assisted. A solute is moved from one phase to the next throughout the extraction process in order to keep it apart from contaminants or unreacted starting components. Additionally, extraction is utilized to help isolate a solute from a reactive solvent such as a solvent such as a solvent with a high boiling point that is challenging to evaporate.

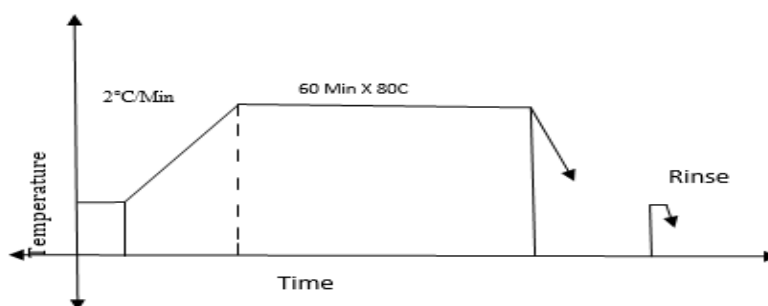


Fig 3 Dyeing Curve.

While it can withstand 7-47°C, *Dillenia indica* thrives best in climates with yearly daytime temperatures between 30 and 40°C. Although it can handle 2000-5500mm, it favors a mean annual rainfall in the range of 3000-4000mm. Thrives in soil that is rich and slightly acidic and prefers a pH within the 5.5-7, allowing the seeds sprout within the fruit, which is left behind on a

riverbank, frequently partially filled with muck. It provides a germination friendly substrate. It favors a sunny weather and sandy loam with good drainage.

3.3 Materials and Equipment

Dellenia indica leaves collected from various places like Cumilla, Dhaka, Bangladesh & also from Sylhet, Dhaka, Bangladesh. Here *Dellenia indica* leaves is used because of its some properties such as anti-inflammatory, antimicrobial & antioxidant. These leaves are collected from aversely grown-up trees which are near about 10 years old. Only leaves are collected from removing stalks. Silk fabric is dyed for experimental purpose. The special purpose of using silk is its luxurious feel, natural sheer, breathability, hypoallergenic, moisture absorption, versatility, durability, natural temperature regulator, biodegradability, cultural significance. Silk fabric had bought from Soura silk, Dhanmondi, Dhaka. No chemical is used for the experiment. Only lemon juice is used as mordant which is help to increase efficiency of the dye absorbency.



Fig 4 Ultrasonic machine

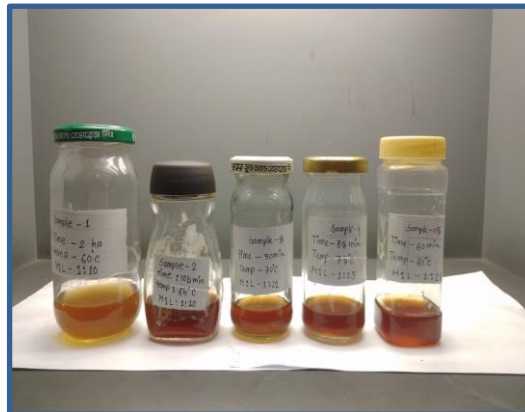


Fig 5 Extracted dye liquor

The potential of employing enzymes and a bio mordant to naturally dye silk with *Dillenia indica* has been investigated. This textile preparation is environmentally safe and does not involve the use of metal mordanting. Silk fabric was using the aqueous extract. Silk was treated with a bio mordant or an enzyme. The resultant dyed fabric exhibited fading resistance. Ultimately, the wash and light fastness characteristics of each dyed specimen were examined, establishing *Dillenia indica* as a competitive substitute for artificial dyes.

3.4 Experimental Details

3.4.1 Color Fastness Analysis:

Color fastness is a crucial component in the textile industry that ensures fabric color stability and keeps colors from fading or shifting readily under various use and care situations. An analytical method for determining how well a textile material retains color under different conditions is called color fastness analysis.

3.4.2 Washing Fastness

Under ideal circumstances, the color fastness to wash of the dyed Silk fabric swatch was measured using the ISO 105-CO6 A2S (2013) method. The materials were washed in a conventional soap solution with a 30:1 liquor to cloth ratio for 20 minutes at 60 °C. In textile materials, "color" refers to any pigment or dye that is sprayed on the fabric to give it a specific pattern or hue. These dyes or pigments have a propensity to smudge over time and after several washings. Therefore, it is essential to confirm the fastness of any material that has been dyed or printed in order to assess the quality of the dye or pigment being utilized.

3.4.3 Rubbing Fastness (Crocking):

The ISO 105 X12 (2002) method was used to determine the dyed Silk fabrics' dry and wet rubbing fastness. Ten times, the materials were rubbed in a crock meter. A fastness is a place, such as a fortress, that is considered secure because it is hard to get to or has an easy defense against attack. Finding out how much color can be transferred from a colorful fabric's surface to a specific test cloth—which could be wet or dry—is the goal of the color fastness to rubbing test.

3.4.4 Perspiration Fastness:

The perspiration test is widely used to assess the colorfastness and durability of textiles and dyed materials when they are exposed to simulated human perspiration. This test ascertains whether the color of a material will bleed or alter in contact with perspiration. Color fastness is the completed textile's ability to maintain its original color through any challenges it may encounter in its lifetime

of use. For a textile material to be deemed appropriate for its intended purpose, it must exhibit a high degree of colorfastness. Exposure to light, dye loss during washing, rub fastness—a measurement of the amount of color retained on the fabric surface after mechanical rubbing—and thermal stability—at temperatures that could be encountered, for example, when ironing—are the most common "challenges" for clothing.

3.4.5 Spectrophotometric Analysis:

In the textile industry, spectrophotometric analysis refers to the measurement and examination of a material's response to light at different wavelengths using a spectrophotometer. The textile sector mostly uses spectrophotometry for color characterisation and measurement. Spectrophotometric analysis is a versatile and vital technology in the textile industry that aids in the production of textiles with accurate and consistent color properties. Data color machine was applied to the silk fabrics. There is a 30mm aperture. The parameters that correspond to different characteristics of color and material attributes include K/S (R), $L^*a^*b^*$, $L^*c^*h^*$, ΔE , and MI.

K/S (R): This metric describes the strength of color absorption of a substance. K/S is an acronym for "absorption coefficient divided by scattering coefficient" in the Kubelka-Munk theory. Reflectance is commonly represented by R. Use K/S (R) to calculate the color intensity of a sample.

$L^*a^*b^*$ Color Space: The CIE Lab* color space is a three-dimensional color model based on human perception. Lightness (L^*):* Shows the brightness (0–100), ranging from dark to light.

a (green to red): * Positive values signify red, and negative values represent green.

b (blue to yellow): * Positive values signify yellow, whereas negative values represent blue.

Color Space $L^*c^*h^*$: * Though it represents color using hue angle (h^*), chroma (c^*), and lightness (L^*), this color space is also derived from CIE Lab*.

ΔE (Delta E): Delta E, or ΔE , is the color difference measurement unit. It calculates the difference between two colors in a color space. The perceived difference between two colors can be assessed with the use of ΔE values. As the ΔE value decreases, the color difference becomes less apparent.

Melt index, or MI, is a statistic that polymer scientists use to characterize the flow properties of polymer melts. It measures the rate at which a polymer extrudes at a specific load and temperature.

Grams per 10 minutes (g/10 min) is the unit of measurement used to express the viscosity of the polymer melt.

3.4.6 Scanning electron microscopy (SEM) measurement:

Using electron beams to analyze sample surface morphology at high magnification, Scanning Electron Microscopy (SEM) is a potent imaging technique. SEM is an essential tool in many scientific and industrial fields because it generates detailed, high-resolution three-dimensional images. The signals from the SEM analysis, when paired with the EDS feature, provide a two-dimensional image that offers information on the sample's material orientation, external morphology (texture), and chemical composition. The system's EDS component is utilized in tandem with SEM analysis to identify components inside or on the surface of the sample for qualitative information. Along with coatings on metal and non-organic foreign materials, it may also determine the elemental makeup for semi-quantitative results.

3.4.7 Thermogravimetric (TGA) analysis:

A thermal analysis method called thermogravimetric analysis (TGA) calculates how a sample's weight changes in response to temperature or time. This technique is especially helpful for researching a material's thermal stability and decomposition behavior. By observing weight variations, the device generates a thermogram that illustrates processes like volatilization or breakdown. With applications in polymer analysis, pharmaceuticals, catalyst studies, and environmental studies, TGA is used to investigate the thermal stability, decomposition behavior, and composition of materials. The method is useful for research and quality assurance in a variety of sectors. The analysis was carried out up to a temperature of 500 °C. The heat was applied at a rate of 10 °C.

3.4.7 Antimicrobial test

To assess or test an extract's or a pure compound's in vitro antimicrobial activity, a range of scientific techniques can be applied. The most popular and fundamental techniques are the broth or agar dilution and disk-diffusion methods. Other techniques, including the poisoned food method, are specifically employed for antifungal testing.

RESULTS AND DISCUSSION

4.1 Shade Analysis

Silk fabric was dyed using *Dillenia indica* leaf to produce functional fabric in different material liquor ratio. Darker shade was produced by mixing 10gm of dry *Dillenia indica* leaf powder with 100ml distil water. For lighter shade 10gm of powder were added to 150ml fresh distil water to get 1:15 material to liquor ratio. After shade analysis under standard day light (D-65) dyed silk fabric (1:15) were lighter, more greenish and more yellowish than the sample were dyed by 1:10 liquor ratio. After dyeing the silk fabrics shade was evaluated under standard day light (D-65), Fig 1 shows the raw silk we have collected for dyeing. Fig 2, Fig 3 confirm the silky appearance of the fabric which were dyed by 1:15 and 1:10 liquor ratio.



Fig 1: Raw Silk Fabric



Fig 2: Dyed Silk (Lighter)



Fig 3: Dyed Silk (Darker)

4.2 Color Fastness to Rubbing

Color fastness to rubbing of dyed silk fabric were determined using Crock meter. Darker and lighter dyed fabric were tested under dry and wet condition to get appropriate result. Dyed silk fabric was abraded by rubbing paper and compared the paper to the standard grey scale. Dyed darker shade fabric shows excellent rubbing fastness.

Condition	Rating (darker shade)	Rating (lighter shade)
Dry	5	5
Wet	5	4/5

Table 1: Rubbing test ratings for darker and lighter shade

In Table 1, its clear that all the rubbing fastness for dyed darker or lighter shade were excellent. Darker shade (1:10 liquor ratio) exhibit outstanding rubbing fastness properties with the rating of 5 out of 5 both the wet and dry condition. Lighter shade shows excellent fastness properties too with the rating of 4/5 in wet condition. So, after all its proved that dyed silk fabric using *Dillenia indica* leaf shows excellent rubbing fastness properties.

4.3 Color Fastness to Perspiration

Dyed silk fabric shows excellent perspiration fastness under acidic and basic condition. The result was determined under woven at 38°C (Body temperature) temperature for 6 hours. No color change found after the experiment.

Multi-fiber / Sample	Acidic condition		Basis condition	
	Dry	Wet	Dry	Wet
Acetate	5	5	5	5
Cotton	5	5	5	5
Nylon	5	5	5	5
Polyester	5	5	5	5
Acrylic	5	5	5	5
Wool	5	5	5	5
Color change	5	5	5	5

Table 2: Color fastness to perspiration test

Following drying, evaluate each specimen color change using the grey scale, and evaluate each component staining on the nearby multi-fiber cloth using the same grey scale. Acidic and alkaline sweat must have a level 3-4 color fastness (color change staining). Dyed silk fabric shows excellent fastness properties with the rating 5 (outstanding) out of 5 both the acidic and alkali conditions under wet and dry state.

4.5 Spectrophotometer Analysis

Lighter and darker dyed fabric were compared to each other for spectrophotometric analysis using data color machine. Standard day light D-65 considered as first light source, whereas TL84-10 and A-10 deg. selected as 2nd and 3rd light source. The dyed fabric (light shade) was more yellowish (Fig. 2) than the darker shade. Reflectance and K/S value determined to find out the color strength of the fabric. L^* stands for lightness, C^* for chroma, and h for hue angle in this color space showed in the Table 3. The a^* and b coordinates in $L^*a^*b^*$ are used to compute hue and chroma. Positive (+) or negative (-) deltas can be found for hue (ΔH^*), chroma (ΔC^*), and lightness (ΔL^*). The brighter is denoted by L^* , the red/green coordinate by a^* , and the yellow/blue coordinate by b^* , as illustrated below on Table 3. In color space $+a$ indicates the red color where $-a$ refers the greenish color. The tested dyed sample were more greenish as you can see on the table 3 given below under the standard light source D-65 (Day light), TL-84, and 10 deg observer. On the other hand, $+b$ refers the yellowish color on the coordinate under the same light source. Lightness 1.16 indicates the darkness of the tested fabric. Hue, chroma, and lightness are re-presented by the semi-axes of an ellipsoid that is mathematically defined around the standard color by the CMC calculation. There is no metamerism found for the tested sample under standard light source compare to the darker sample. But under 2nd light source there is a little bit metamerism found (0.04).

Spectrophotometer Shade Analysis

Ill/Obs	DL*	Da*	Db*	Dc*	DH*	CMC DE	Metamerism index
D65 10 Deg.	1.16	-2.20	1.15	0.60	2.41	3.44	-
TL84-10	1.13	-2.20	1.16	0.62	2.41	3.24	0.04
A 10 Deg.	0.98	-2.05	0.70	-0.17	2.16	3.26	0.53

Table 3: Spectrophotometer test result

DL* = Lightness

Da* = difference on redder/greener

Db* = difference on yellow/blue

Dc* = difference in chroma

DH* = difference in hue

DE* = total colour difference

3.6. Color Strength Analysis

Color strength of a dye is the intensity of colour and the ability of dye to impart colour to other materials. Reflectance (R%) value is determine using Kubelka-Munk theory to determine K/S value.

$$K/S = (1-R)^2/2R.$$

Here

K/S = Color strength

R = Reflectance %

Standard Sample or Darker Shade

Wavelength	Reflectance (R%)	Color strength (K/S)
400 nm	27.21	12.623
410 nm	34.00	16.014
420 nm	38.74	18.382
430 nm	42.11	20.066
440 nm	44.51	21.266
450 nm	46.51	22.265

Table 4: Reflectance & K/S value for darker shade

Tested Sample or Light Shade

Wavelength	Reflectance (R%)	Color strength (K/S)
400 nm	29.03	13.532
410 nm	34.89	16.459
420 nm	39.30	18.662
430 nm	42.49	20.256
440 nm	44.83	21.426
450 nm	46.98	22.500

Table 5: Reflectance & K/S value for light shade

For darker shade in the wavelength 400 nm, under reflectance 27.21% the k/s value found 12.623 but as the same wavelength for the lighter shade under the reflectance 29.03% the color strength was 13.532. As the wavelength increase the k/s gradually increase.

4.7 Tensile Test Result

Maximum strength that a material can withstand before breaking is known as tensile strength. The dyed fabric was torn on warp (length wise) direction to get the maximum strength. Dyed silk fabric using *D. indica* leaf shows less breaking strength (Table 6) than the grey fabric.

Specimen	Maximum Force	Elongation at max. force (%)	Extension at 30.00 N (%)
Dyed sample	120.54	14.59	7.765
Bleached sample	177.02	10.07	3.767
Mean	148.78	12.33	5.766

Table 6: Tensile test result

The tested results show the tensile strength of the dyed and raw silk fabric. Maximum force to break down the dyed sample was 120.54 N, Elongation at maximum force was 14.59% where the grey sample exhibit more strength than the dyed silk fabric with the elongation at maximum force of 10.07%. there is a little change in the strength of the dyed sample.

4.8. TGA Analysis & DSC Analysis

Thermo-gravimetric analysis is one of the most important physical tests to determine the change in weight of the substance as a function of temperature or time upon a controlled environment. DSC is a thermal analysis device that measures temperature and changes in a sample's physical characteristics over time. In another way, the apparatus is a thermal analysis tool that measures the temperature and heat flow related to material transitions as a function of temperature and time. The sample is heated in a standard or given atmosphere at a controlled rate. Differential Scanning Calorimetry (DSC) is a test of a material that shows (Fig. 2) how physical properties changes along with temperature changes and its compared to TGA result. Thermogravimetric analyzer which, via a single analysis, can identify many parameters at user-defined temperatures and atmospheres, including moisture, volatiles, and ash. Operating at temperatures as high as 5000 °C, the thermogravimetric analyzer TGA Thermostep can analyze up to 19 samples at once, each weighing up to 5 g.

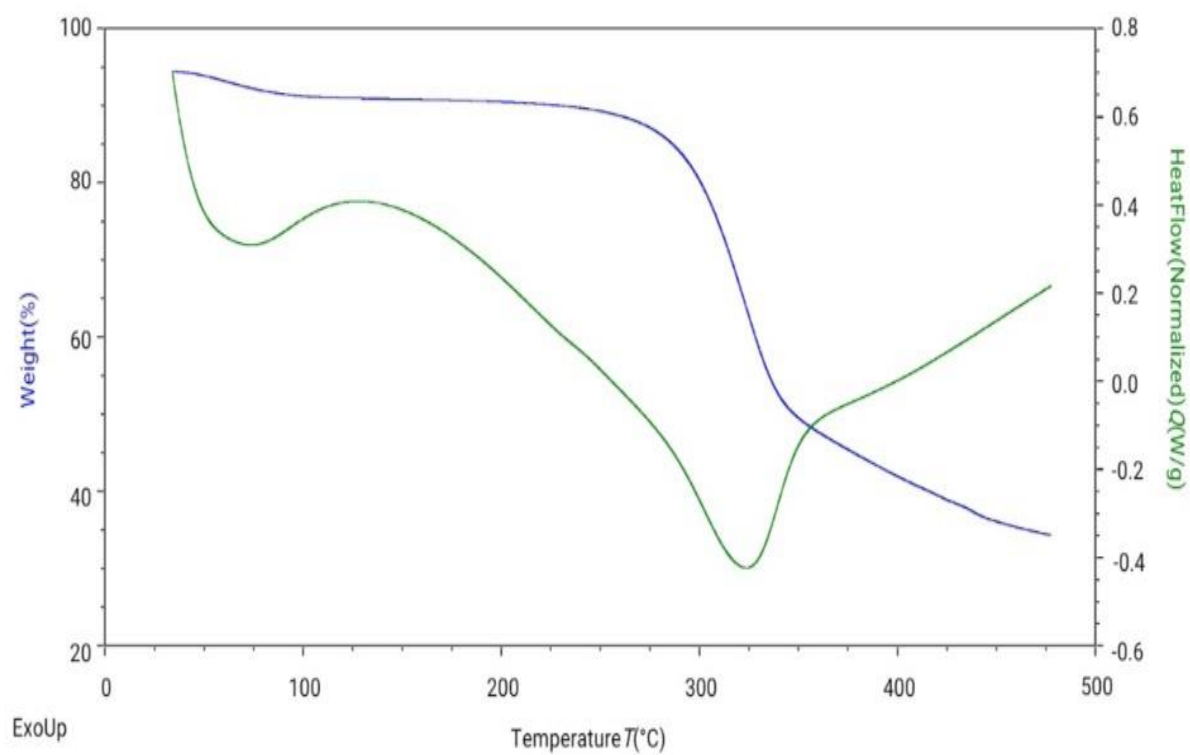


Fig. 2: TGA and DSC analysis graph

4.9 SEM Analysis

Scanning Electron Microscopy (SEM) images shows the surface morphology of dyed silk fabric. SEM is a powerful analytic technic for wide range of material that gives a microscopic view with an electron beam to produce magnified image of a sample. The images confirm the excellent dye up take by silk fabric using *D. indica* leaf provides outstanding golden colour along with functional properties. From fig. 3 this is confirmed that more the microscopic view of the fabric shows higher lustered. Darker shade fabric was tested as a stander for SEM analysis for different microscopic views. After the analysis is confirmed that dyed silk fabric (300 μm to 10 μm) shows more silky and lustrous appearance. Microscopic view of silk fiber shows the floating fiber on the surface after dyeing, A little bit of mechanical damage on the surface due to the dyeing process. In the warp direction its noticed that the fiber becomes less lustrous and curly.

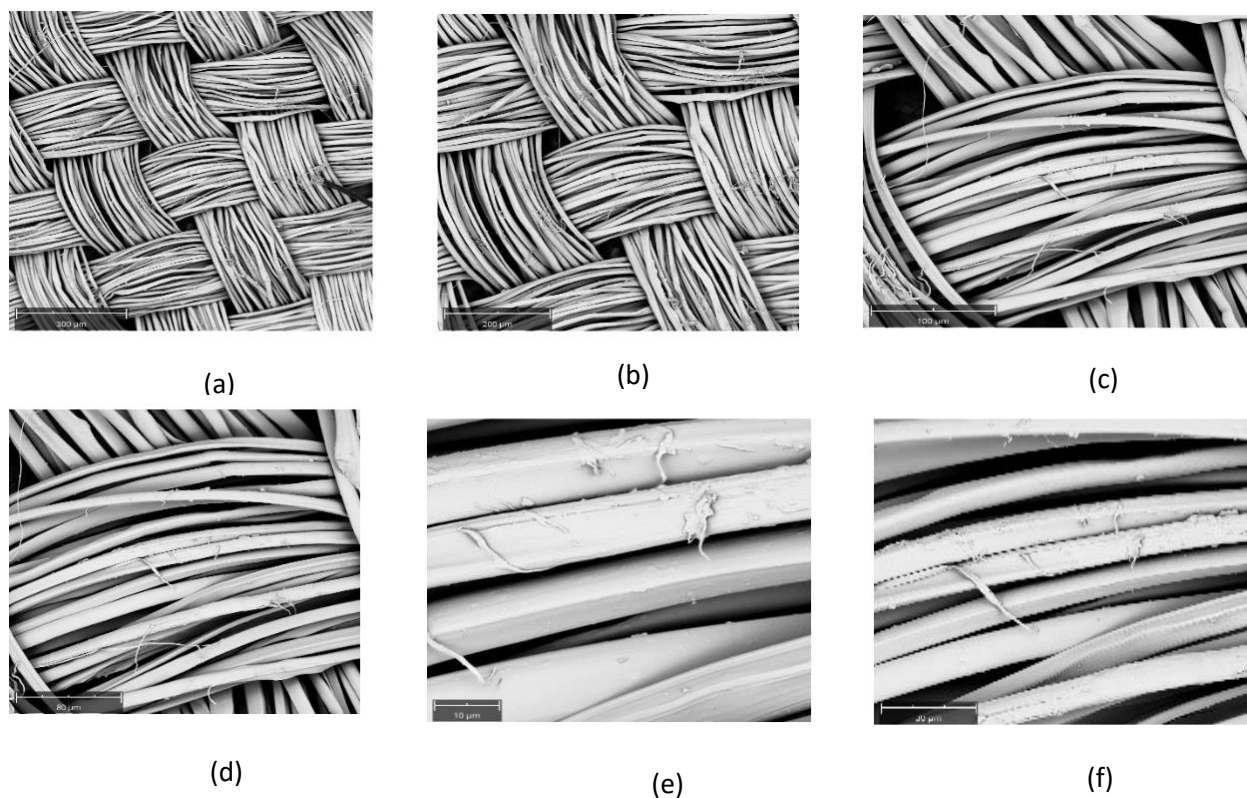


Fig. 3: SEM image ((a)300 μm , (b)200 μm , (c)100 μm (d)80 μm , (e)30 μm , (f)10 μm).

4.10 Antimicrobial Test Analysis

The anti-microbial characteristics of dyed silk fabric are demonstrated by the anti-microbial test. The most popular and basics methods are the broth or agar dilution and disk-diffusion methods. Other techniques, including the poisoned food method, are specifically employed for antifungal testing. Here, the silk textiles were tested using the Kirby-Bauer disc diffusion test.

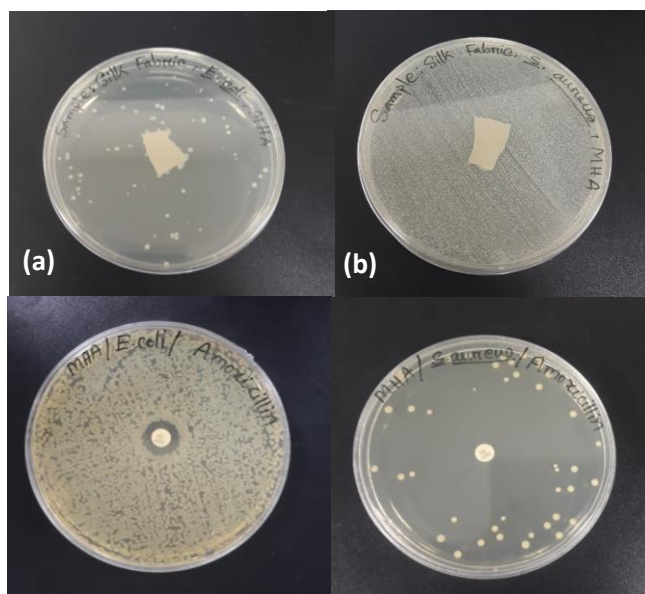


Fig 4.2: Anti-microbial test image (a) *E. coli*, MHA (b) *S. aureus*, MHA (c) *E. coli* Amoxicillin (d) *S. aureus* Amoxicillin

SL	Test Parameters	Test Results	Reference Test Method
1	Antimicrobial Sensitivity (<i>E. coli</i> ATCC: 11775)	No ZOI	Kirby-Bauer disk diffusion test
2	Antimicrobial Sensitivity (<i>Staphylococcus aureus</i> ATCC: 6538)	NO ZOI	Kirby-Bauer disk diffusion test

Table 7: Antimicrobial Test

Bacterial cell concentration: 1.5×10^5 CFU/ml

ZOI: Zone of inhibition

Disk size: 6mm×6mm

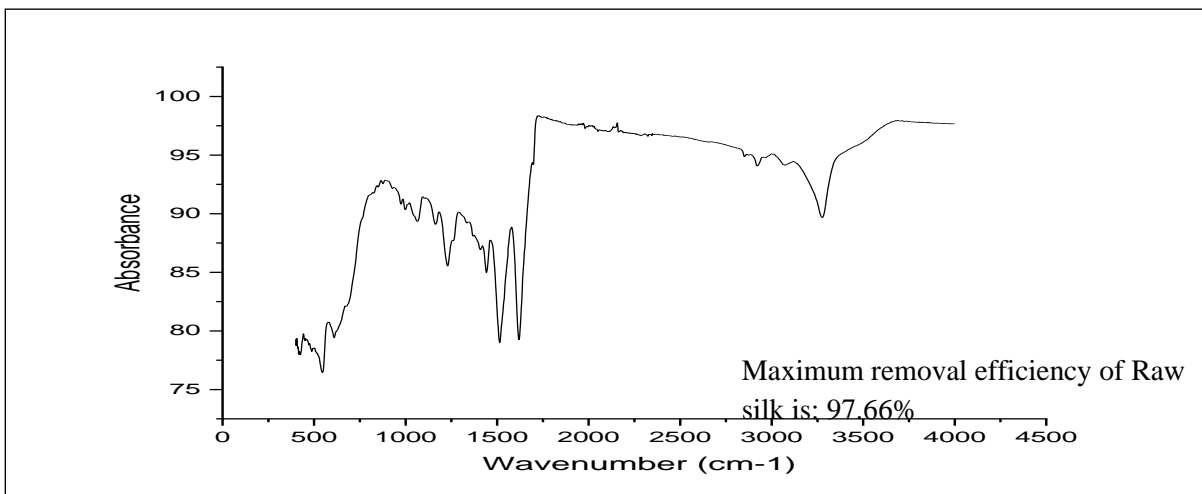
The antimicrobial test's negative result could be caused by a number of factors:

1. **Insufficient Concentration:** *Dillenia indica* extract may not have enough concentration to have a noticeable antibacterial effect. Raising the concentration might make it more effective.
2. **Extraction Technique:** The antimicrobial compounds from *Dillenia indica* leaves may not have been extracted using the best possible technique. Different extraction techniques can produce bioactive compounds at different concentrations.
3. **Particular Microorganisms to Aim for:** *Dillenia indica* has the potential to be antimicrobial against particular kinds of microbes. Testing against a wider range of fungi or bacteria could show which strains it works well against.
4. **Dyeing Process Interference:** The antimicrobial qualities of the *dillenia indica* extract may have been changed or lessened by the dyeing procedure itself. It is important to make sure that the functional properties are not adversely affected by the dyeing process.
5. **Interaction with Silk:** The antimicrobial qualities of silk fabric may be impacted by the interaction between *dillenia indica* extract and silk. It might be required to look into possible interactions and improve the fabric treatment procedure.

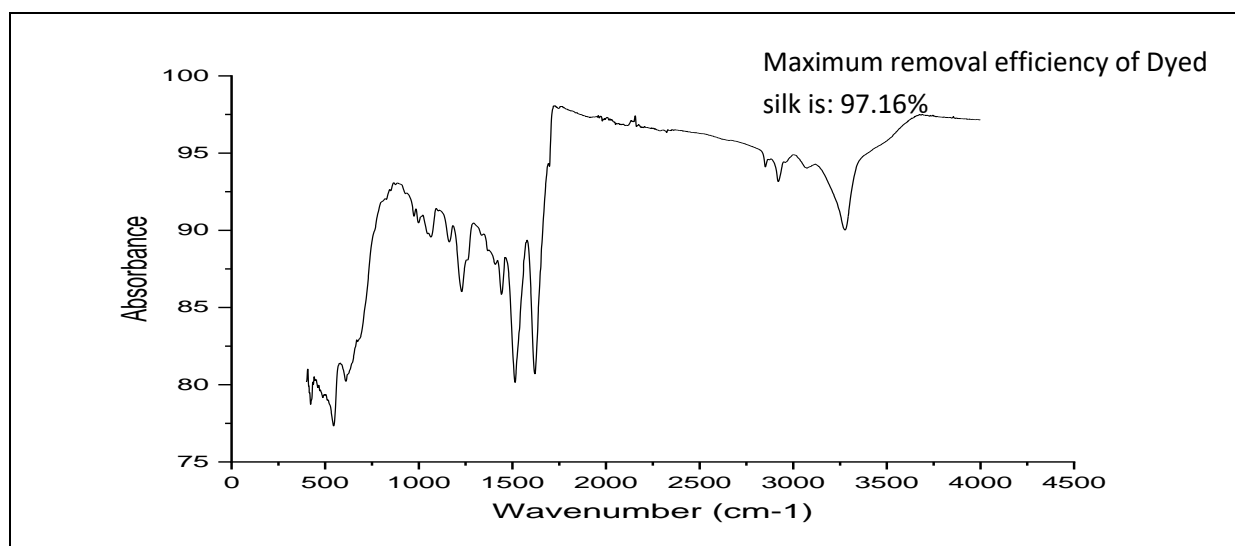
By systematically experimenting and adjusting these factors, it may be possible to determine the causes of the lack of antimicrobial activity.

4.11 FTIR Test Analysis

Fourier Transform Infrared Spectroscopy measures the chemical bonds presence in a molecule by producing an infrared absorption spectrum. A silk polymer system is held together by van der Waals forces, hydrogen bonds, and salt linkages. Silk filament dissolves easily in the alkaline solution because the alkali hydrolyzes these interpolymer forces of attraction. At first, silk dissolution means only the separation of polymers from each other.



Graph 1: FTIR for Raw Silk Fabric



Graph 2: FTIR for Dyed Silk fabric

Functional Group	Frequency Range (cm ⁻¹)
C-O	2353
-COOH	2260-2100
SO ₃	2918-2954
Phenol	3587
Amide	3675
NH ₂	2322

The frequency range and functional group of the silk fabric were obtained from the FTIR test.

CHAPTER

FIVE

CONCLUSION

In this Concluding chapter, we provided an overview of our conclusions and arguments in this concluding chapter and consider the advantages and disadvantages of the theoretical and methodological techniques we have used. This work is innovative because it uses an eco-friendly natural dye made from *Dillenia Indica* leaves and dyes silk fabric using an ultrasonic process. For this process, we used aqueous extraction process. In this thesis work, it is demonstrated that the ultrasonic approach is an excellent alternative for extraction and coloring. The primary objective of our research is to investigate eco-friendly methods of textile dyeing that make use of leaves' inherent qualities on silk fabrics. Our primary goals are to ensure that vibrant and long-lasting colors are produced by testing the silk fabric dyed with *Dillenia indica* leaves for color stability and fastness. Analyzing the effects of various mordanting methods on the dyeing process to improve the color retention and fade resistance of silk textiles. We looked into the scalability of *Dillenia Indica* leaf dyeing in order to devise a process appropriate for large-scale textile production. We gathered leaves of *Dillenia indica* in our rural communities. Leaves are sun-dried for a minimum of 15 to 20 days. The leaves are then ground into a powder using a blender. One of the main findings of the study is that no chemicals were utilized in the extraction or coloring of the silk fabric. This procedure can be referred to as completely chemical-free and environmentally helpful because no chemicals were used in the extraction or dyeing process. This process is entirely chemical-free and beneficial to the environment because it uses no chemicals- were used in the extraction or dyeing process. We have performed a variety of tests, including tensile, shade, color fastness to rubbing, color fastness to perspiration, spectrophotometer, TGA & DSC, SEM, antimicrobial, FTIR, and more. The fastness qualities of natural dyestuffs were often poor, but we

have discovered that they can range from very good to exceptional. The improved dyeing performance of the dyed fabric sample is revealed by its characterization. The dyed sample exhibits a reddish yellow tone or tint, according to its CMC value. Future research will examine the commercial viability, scalability, and prospective uses of eco-friendly natural dyeing with *Dillenia Indica* leaves for functional fabric in sectors such home furnishings, textiles, and sustainable fashion. Enhancing colorfastness, evaluating the environmental impact, and refining dye extraction techniques are areas of potential research. Working together with textile and fashion designers can produce eco-friendly goods that are both inventive and commercially viable, thereby promoting sustainability and social responsibility within the industry.

REFERENCES

1. Melo, Maria J. "History of natural dyes in the ancient mediterranean world." *Handbook of natural colorants* 8 (2009): 1.
2. Gulrajani, M. L.: Present status of natural dyes. *Indian Journal of Fibre and Textile Research* (2001), 26.1(2): 191-201
3. Furry, M. S., Viemont, B. M.: Home dyeing with natural dyes. U. S. Dept of Agriculture (1935), (reprint. by Thresh Publications)
4. Ahmed, H. E. (2023). History of Natural Dyes in North Africa_Egypt. *Handbook of natural colorants*, 33-41.
5. Merdan, N., Eyupoglu, S., & Duman, M. N. (2017). Ecological and sustainable natural dyes. *Textiles and clothing sustainability: sustainable textile chemical processes*, 1-41.
6. Liu WJ, Cui YZ, Zhang L, Ren SF (2009) Study on extracting natural plant dyestuff by enzyme-ultrasonic method and its dyeing ability. *J Fiber Bioeng Info* 2(1):25–30
7. http://www.health.com/health/gallery/0,,20588763_15,00.html. 09 May 2016
8. <https://www.pinterest.com/pin/264727284316589787/>. 09 May 2016
9. Burhan Uddin Banna, Rony Mia, Md. Mahabub Hasan, Bulbul Ahmed, Mohammad Abul Hasan Shibly (2023). *Ultrasonic-assisted sustainable extraction and dyeing of organic cotton fabric using natural dyes from Dillenia indica leaf*. <https://doi.org/10.1016/j.heliyon.2023.e18702>
10. <https://www.researchgate.net/publication/354352116> PHYTOCHEMICAL SCREENING AND EXTRACTION OF NATURAL DYE FROM LEAVES OF POLYALTHIA LONGIFOLIA AND ITS APPLICATION ON SILK FABRIC
11. <https://www.researchgate.net/publication/221919686> Natural Dye from Eucalyptus Leaves and Application for Wool Fabric Dyeing by Using Padding Techniques
12. <https://www.researchgate.net/publication/338123459> Extraction of Natural Dye from Tea Leaves and its Application on Giza 86 Egyptian Cotton Fabric
13. <https://doi.org/10.5281/zenodo.3519880>
14. <https://www.researchgate.net/publication/289907170> Dyeing of silk with natural dyes

15. Renu Singh, Sangita Srivastava (2015). *Silk Dyeing with Natural Dye Extracted from Spice* [https://www.academia.edu/22677078/Silk Dyeing with Natural Dye Extracted from Spice](https://www.academia.edu/22677078/Silk_Dyeing_with_Natural_Dye_Extracted_from_Spice)
16. V Narayana Swamy, a, K N Ninge Gowda & R Sudhakar (2014). <https://core.ac.uk/reader/229215390>
17. Erdawati, Hayyun Lisdiana and Ganjar Saefurahman (2014) *Utilization of Indigofera Leaves as Natural Dyes for Silk Fabrics* <https://iopscience.iop.org/article/10.1088/1755-1315/1187/1/012014>
18. Merdan, N., Eyupoglu, S., & Duman, M. N. (2017). Ecological and sustainable natural dyes. *Textiles and clothing sustainability: sustainable textile chemical processes*, 1-41.
19. Chairat M, Rattanaphani S, Bremner JB, Rattanaphani V (2008) Adsorption kinetic study of lac dyeing on cotton. *Dyes Pigm* 76:435–439
20. Han KH, Okada TK, Seo JM, Kim SJ, Sasaki K, Shimada KI, Fukushima M (2015) Characterisation of anthocyanins and proanthocyanidins of adzuki bean extracts and their antioxidant activity. *J Funct Foods* 14:692–701
21. <http://www.emrath.de/pigmente.htm>; November 2015
22. Ferreira E, Quye A, McNab H, Hulme A (2003) LC-ion trap MS and PDA–HPLC—complementary techniques in the analysis of different components of flavonoid dyes: the example of Persian berries (*Rhamnus* sp.). *Dyes Hist Archaeol* 18:13–18
23. Melo, M. J. (2009). History of natural dyes in the ancient mediterranean world. *Handbook of natural colorants*, 8, 1.
24. J. Balfour-Paul, *Indigo*, British Museum Press, 2000. <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC9024665/>

