

**Enzyme Assisted Natural Dye Extraction From
Saraca Asoka Wood and Its Application
on Silk Fabric**

By

Anu, V

(13PTF002)

A Thesis submitted to
**Avinashilingam Institute for Home Science and Higher Education for Women
Coimbatore – 641 043**

In Partial Fulfillment of the Requirements for the

Degree of Master of Science

in

Textiles and Fashion Apparel

March, 2015

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Certified as Bonafide Research Work


27/3/15

Signature of the

Head of the Department



Signature of the Guide

ACKNOWLEDGEMENT

The investigator thanks **GOD ALMIGHTY** for giving strength, courage and ability to successfully complete this research study.

The investigator fondly remembers **Ayya Avl. Dr. T.S. Avinashilingam, Founder President – First Chancellor** and **AmmaAvl. Dr. (Mrs) Rajammal .P. Devadas, Former Chancellor**, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore.

The investigator records her sincere thanks to **Dr (Thiru).T.S.K.Meenakshi Sundaram, M.A., M.Phil.,Ph.D.**Chancellor, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, for providing the infrastructural facilities for the conduct of the study.

The investigator wishes to express her profound gratitude to **Hon. Col.Dr (Tmt) Sheela Ramachandran, M.Sc., P.G.Dip., Ph.D.**, Vice chancellor, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, for being a constant source of inspiration, motivation and guidance during the course of the study.

The investigator extends her sincere thanks to **Dr (Tmt) A.Venmathi, M.Sc., Dip. Ed., M.Phil., Ph.D.**, Registrar In charge, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, for extending all possible help for the smooth conduct of the study.

She expresses her respectful regards and sincere thanks to **Dr (Tmt) N.Vasugi Raaja, M.Sc., MBA., M.Phil., Ph.D.**, Dean, Faculty of Home Science and Professor and Head of the Department of Textiles and Clothing, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, for rendering all help needed for the successful completion of the study.

The investigator feels extremely privileged to have worked under the able guidance of her esteemed guide **Dr.(Tmt.) K.KALAIARASI, M.sc., M.Phil., Ph.D.**, Assistant Professor, Department of Textiles and Clothing, Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore, for her patience, effective guidance, untiring enthusiasm, meticulous efforts, abiding interest, useful

suggestions, innovative ideas and motivation for the successful execution of the study.

The investigator expresses her thanks to **all the staff members** in the Department of Textiles and Clothing for their help to complete this study.

The investigator owes her sincere thanks and gratitude to her beloved **FAMILY MEMBERS** and **sisters** for their enthusiasm, co-operation, love, care and support, constant encouragement, blessings, constant prayer, help rendered and moral support for the successful completion of the study. And Finally the investigator expresses her full gratitude to her **Friends** who helped her lot to finish this study.

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1. INTRODUCTION

Highly colored substances, known as dyes or colorants, are widely used to impart color to a boundless variety of materials described technically as substrates. The increased application of these dyes or colorants in dyeing industries and in efficiencies in dyeing result in a large amount of dyestuff being directly lost in the wastewater, which ultimately finds way into the environment. It is estimated that 10-35% of the dye is lost in effluent during the dyeing process. While in the present in the dye bath effluent moreover, some of these dyes contain potential colon carcinogens, which is possible hazard to humans when chronically exposed. Hence, there is a growing demand for eco-friendly non-toxic colorants, specifically for health sensitive application such as coloration of food and dyeing of child textile and leather garment (Sivakumar, 2007).

Natural dyes and pigments are emerged as an important alternative to potentially harmful synthetic dyes. Natural dyes comprise colorants that are obtained from animals or vegetable matter without any chemical processing. Natural dyes were used to color clothing or the textile. Dyes were extracted from plants, insects, leaves and roots of plants for thousands of years to impart color onto textile. Today natural dyes have almost no economical importance and are used in limited quantities (Kumar, 2009).

Natural dyes obtained from natural sources are aesthetically appealing and eco-friendly. Natural dyeing was commercially used for dyeing of textile from earliest time. For fulfillment of all needs man has looked towards nature. These were used for textile coloration (Sharma, 2011). These dyes have very little health hazards. No disposal problems and are expected to obtain creative natural finish to textile materials.

There are three primary sources for natural dyes plants, animals, and minerals. Regardless of the source natural dyes can be broken down into two categories. Substantive and adjective, substantive dyes, also referred to as direct dyes. Substantive dyes for green dyed garment it is preferable to use substantive natural dyes Adjective dyes also referred to as mordant dyes require an added

substance known as a mordant to make the dyes colorfast. Most natural dyes are adjective dyes. The types of mordant used in the dyeing.

Natural dyes are soft, gentle, safe, non-toxic and suitable for all fabric. Most natural dyes need mordants to fix the color to the fiber and increase light fastness, pollution problem can be controlled by natural dyes. The term mordant is from the natural dyes (Sundrajan, 2007). The synthetic dyes used in textile wet processing produce effluent which cause environmental pollution. Natural dyes are free from such problems. Moreover if steps are taken to commercialize the cultivation of plants from which most natural dyes are generated it will definitely assist in preserving the eco-balance (Rajani, 1999). These vegetable resources not only replace petrochemical resource of synthetic dyes. These have also been much interest recently in the pharmacological effect and possible health benefits of the use of natural dyes (Petal, 2000).

Enzymes are more stable catalysts than other chemical or biological molecules. Enzymes also work at low temperature and moderate p^H . This is not the case for most chemicals which perform the same processes. Enzymes do not become part of the final product of the biochemical reactions which they are catalyzing. (Herpes, 2008).

Silk is a way of life in India. Over thousands of years it has become an inseparable part of India culture and tradition. No ritual is complete without silk apparel. The Indian silks are known for their fine quality, lustrous sheen and traditional colors. Silk provides the much-needed employment in several developing and labour rich countries. Over 25 countries in Asia, Africa and South America are presently engaged in sericulture projects of varying dimensions (Ojha, 2004). Silk is one of most important natural fibers with merits of wearing comfort, handle, good, air, permeability and elegant appearances. (Kumara, 2012). To produce one pound silk it requires cocoons from about 2500 silk worms, accounting for the luxury price tag of this material (Shopper, 2013).

Asoka tree is a rain forest tree found in Indian subcontinent. Meaning of name Asoka is sorrow less. This tree is found in many areas in India mostly in Bengal, Uttar Pradesh and south India. Asoka tree is a beautiful tree with green leaves and

very attractive red flower. Its height is about 6-9 meters, leaves 15-25 cm long. Asoka tree is covered with red color flower and looks really very beautiful. The fruits are about 6-10 inches long, looks like broad beans containing 6-8 grey color seed like chest nut. Asoka tree leaves, flower and bark has antibacterial properties and can be used to cure bacterial infections. Saraca Asoka dried bark has been used for menorrhagia in India. Saraca Asoka dried bark as well as flower is given as a tonic to ladies in case of uterine disorders. Saraca Asoka stem bark also used to treat all disorder associated with the menstrual cycle (Kumar, 2011).

Traditionally most textile dyers use the aqueous extraction method to extract natural dyes from plant source. But, this method takes several hours of extraction time. It is therefore important to utilize most efficient extraction method. Hence the present study "Enzyme assisted natural dye extraction from Saraca Asoka wood and its application on silk fabric" aims with the following objectives.

- To extract dye from Saraca Asoka wood using cellulase enzyme.
- To optimize different parameters for dye extraction.
- To evaluate the dyed fabric for its physical and mechanical properties.

2. REVIEW OF LITERATURE

Review of literature for the present study on “**Enzyme Assisted Natural Dye Extraction From Saraca Asoka Wood and Its Application on Silk Fabric**” are discussed under the following headings:

2.1. Natural dyes

- 2.1.1 Introduction
- 2.1.2 History
- 2.1.3 Advantages
- 2.1.4 Disadvantages
- 2.1.5 Classification
- 2.1.6 Extraction of natural dye

2.2 Mordant

- 2.2.1 Types of mordant
- 2.2.2 Mordanting techniques

2.3. Enzymes

- 2.3.1 Cellulase
- 2.3.2 Application of cellulase in textile industry

2.4. Saraca asoca

- 2.4.1 Taxonomic classification
- 2.4.2 Botanical description
- 2.4.3 Uses

2.5. Silk

- 2.5.1 Introduction
- 2.5.2 History
- 2.5.3 Physical properties
- 2.5.4 Chemical properties
- 2.5.5 Advantages
- 2.5.6 Dyeing of silk with natural dyes

2.1. NATURAL DYES

2.1.1. Introduction

Textiles are dyed in many different colors and been for hundreds of years. They can be dyed with natural or synthetic dyes. Dyes are generally from an organic source, that is, plant or animal matter. Sources of natural dyes include berries, insects, flower, and other naturally occurring substances (Rita, 2012).

Dyes, which are much smaller molecules than pigments, do not adhere to the surface of a fiber or textile materials but rather bond with the fiber chemically and color the fiber throughout. Dyes allow fibers, yarns, or whole textile to be colored at any time during the textile production process. Since their application is so versatile, different effects can be created (Nandakumar *et al.*, 2014).

Natural dyes are aesthetically appealing, environment friendly, bio degradable, non toxic and cost effective. With world becoming more conscious towards ecology and environmental there is a greater need to revive our heritage (Sudhakar, 2011). Natural dyes give subtle rich warm colors that are unique. Natural dyes are believed of their nontoxic, non allergenic and biodegradable nature (Nagia *et al.*, 2007).

The ability of sensitizers in natural dye is linked to anthocyanin properties. Anthocyanin molecule in the form of carbonyl and hydroxyl which occurs naturally in fruit, leaf and flower is responsible to show different color in red –blue spectrum. Recent studies confirmed that the natural dyes can be good alternative to ruthenium based dyes (Hardin *et al.*, 2009).

Dyeing of textile with natural dyes is a multistep process involving the extraction of dyes, followed by mordanting and dyeing. Natural dyes can be extracted in an aqueous medium, in acidic or alkaline condition; depending on the stability of the dye at different pH. (Subramanian, 2014).

2.1.2 History

The actual date for the first textile dyeing is unknown. Textile work began in Mexico in 5000 BC. Some of the dyes used at this period stayed in until the beginning of the twentieth century. The earliest known dyeing utilizing the

complicated procedures dates to around 3000BC in India, where dye produced became the most advanced of the ancient world due in large part (Gordon, 2013).

During the first quarter of the 20th century a revival of interest in art and crafts led to experimentation with old methods of spinning, weaving and dyeing. This movement, plus efforts to encourage the continuation of southern mountain folk crafts inspired textile craftsmen throughout the country to explore the field of natural dyes once more (Adrosko, 2012).

The use of colorants dates back thousands of years in all societies around the globe. Even before people began to spin yarn and weave cloth, they applied colored earth, plant saps and juices directly to their skin .Perhaps the first step in the history of dyeing was the identification of potential dye plants. Our major dye plants, along with our staple food stuff and a vast dispensary of medicinal plants were all recognized thousands of years ago (Buchanan, 2012).

The history of dyestuffs, both natural and synthetic, invariably transcends the boundaries of science and technology, and cultural history. There are also close links with developments in chemistry, textile production, and conservation (Hessenbruch, 2013).The history of dyeing starts in antiquity, at least as far back as 2000BC.The chemistry of dyes and dyeing is a complicated subject and its history equally .The fact has been frequently used to produce patterned fabric by the application of different mordant in selected areas of the cloth which would then emerge differently colored from the same dye-bath (landing, 2012).

2.1.3 Advantages

Utilizing natural dyes to impart color onto fabric has a number of advantages over synthetic dyeing.

- Natural dyes are more eco-friendly since unlike their synthetic counterparts, they are all derived from natural sources.
- Almost all natural dyes are free of azoic compounds which are carcinogenic.
- Depending on the dyeing procedure and the type of mordant used, a variety of colors can be produced using a single natural dye source. Each

natural dye source provides an amazing diversity of shades. From one dye 5-15 varying colors and shades may be obtained.

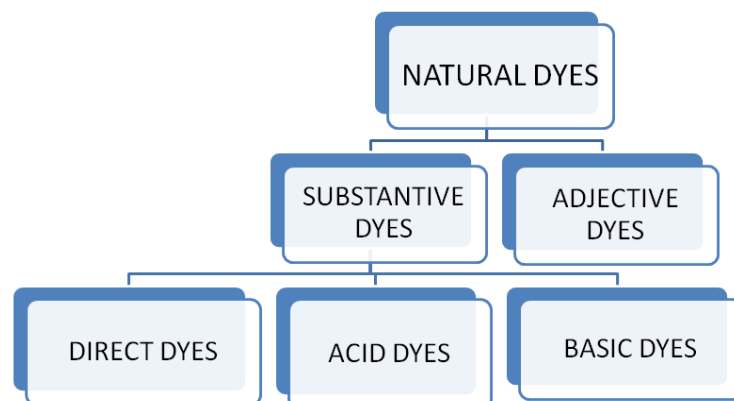
- Inconsistencies in color could be marketed as unique or one of a kind (Bhattacharya, 2006).

2.1.4 Disadvantages

The quality and effectiveness of natural dyestuff depends on many factors. Although there is a trend toward eco-friendly process, natural dyes have some disadvantages.

- Natural dyes have limited availability.
- It takes time to extract the color from raw materials.
- Unlike synthetic dyes that are created in a laboratory, natural dyes are obtained from plants and are dependent on growing seasons.
- Although natural dyes initially produce bright colors in a variety of shades, fabric tend to fade quicker than fabric colored with synthetic dyes when exposed to light and home laundering.
- Consistency of color is a challenge when dyeing with natural dyes. No two dyes are identical, due to impurities in natural dyes.
- Some mordant may present unacceptable levels of toxicity. Mordant, such as chromium and tin are more toxic and are required for the production of some of the brighter colors.
- Synthetic fibers usually cannot be dyed with natural dyes (Siobhan,2000)

2.1.5 Classification



(Jain, 2012).

Natural dyes can be classified by various ways. Early methods of classification were based simply on the alphabetical arrangement of dyes. Later on numerous other kinds of classification were adopted, which are as follows.

- Classification based on chemical structure
- Classification based on their origin or the sources from which they are obtained
- Classification based on their methods of application
- Classification based on their color

Classification based on chemical structure

Natural organic dyes and pigments belong to a wide range of chemical classes, such as indigoid, anthraquinonoids, naphthoquinones, polymethines, ketones, imines, quinines, flavones, flavanones and chlorophyll.

Classification based on their origin or the sources from which they are Obtained

Depending on their origin or the sources from which they are produced, natural dyes can be grouped into three distinct classes: those derived from vegetable sources; those derived from insect or other animal sources and those derived from mineral sources. (Forrester, 2007).

Classification based on their methods of application

- Direct dyes for cotton ,e.g. turmeric, pomegranate, annatto, safflower ,etc.
- Direct dyes for wool and silk, e.g. turmeric. pomegranate, annatto, safflower, etc.
- Acid dyes, e.g. saffron
- Basic dyes, e.g. Berberine (Kiekie et al, 2005).

Classification based on their color

Natural dyes are frequently categorized on the basis of the color that they impart to the fiber substrate.

- Natural yellow dyes
- Natural red dyes

- .Natural blue dyes
- Natural black dyes
- Natural brown dyes (paul et al, 2001).

2.1.6 Extraction of natural dye

As natural dye-bearing materials contain only a small percentage of coloring matter or dye along with a number of other plant and animal constituents such as water-insoluble fiber, carbohydrates, protein, chlorophyll, and tannins, among others, extraction is an essential step not only for preparing purified natural dyes. As natural coloring materials are not a single chemical entity and the plant. Extraction of natural dyes is a complex process. The nature and solubility characteristics of the coloring materials.

- Aqueous extraction
- Alkali or acid extraction
- Microwave and ultrasonic assisted extraction
- Fermentation
- Enzymatic extraction
- Solvent extraction
- Super critical fluid extraction (senthilkannan, 2014).

Aqueous extraction

Aqueous extraction was traditionally used to extract dyes from plants and other materials. The process of boiling and filtering to remove non-dye plant. As most of the dyeing operations are carried out in aqueous media, the extraction obtained by this method can be easily applied to the textile materials (Chakraborty et al., 2009).

Acid and alkali extraction process

Alkaline extraction is suitable for dyes having phenolic group as they are soluble in alkali, which improves the dye yield. dyes can be later precipitated by the use of acids. Dye from annatto seeds can be extraction by this technique. This process is also used to extract lac dye from lac insect secretions and red dye from safflower petals. The disadvantage of this process is that some coloring materials

may be destroyed under alkaline conditions considering the fact that some of the natural dyes are pH sensitive (Kamal et al., 2011)

Ultrasonic and microwave extraction

These are actually microwave –and ultrasound- assisted extraction processes where extraction is increased by the use of ultrasound or microwaves thus reducing the quantity of required solvent, time, and temperature of extraction. When the natural dye containing plant material is treated with water or any other solvent in the presence of ultrasound.

In microwave extraction, the natural sources are treated with a minimum amount of solvent in the presence of microwave energy sources (Siva, 2007).

Enzymatic extraction

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

Solvent extraction by Soxhlet

As the yield of traditional solvent method is very low, Soxhlet apparatus was used to extract the natural sources in organic solvent. Mainly the solvent used was methanol; ethanol etc. plant parts are cut into small pieces and were refluxed by the Soxhlet in solvent till it discharged color. The process takes 4-6 hours (Sara et al., 2004).

2.2 Mordant

A mordant is defined as a substance which aids the chemical reaction between the dye and the fiber in order for dye to be absorbed. Not all dyes need mordant to help them adhere to the fabric. Dyes needing no mordant, such as lichens, are called substantive (Mussel, 2009). However if a mordant is required for adherence, such dyes are termed adjective. Natural dyes are substantive, requiring mordant to fix to the fabric and prevent the color from either fading with exposure to light or washing out.

2.2.2 Types of mordants

Metallic mordants : metal salts of aluminum, chromium, iron ,copper and tin.

Tannins : myrobalan and sumach.

Oil mordants : These are mainly used in dyeing turkey red color from madder. The main function of oil mordants is to form a complex with alum used as the main mordant (Bharat,2010).

2.2.3 Mordanting techniques

There are three types of mordanting. They are premordanting, simultaneous mordanting & post mordanting.

Pre mordanting

Mordanting is done prior to the dyeing process and is called premordanting. The yarns are treated with the appropriate chemicals in a completely separate process from dyeing. After the yarns have been mordanted they can be used immediately for dyeing or dried and stored for future dyeing. Mordanted yarns may be stored wet in plastic bags for up to four weeks if kept in the refrigerator (krohn, 2001).

Post mordanting

In postmordanting method, the yarn is dyed first, and then mordanting was carried out. This method should be used only if mordants are not available at the time of dyeing and one has to use the dyestuff before it spoils. Not more than three weeks should elapse between dyeing and postmordanting so that the dye strength is not lost, Chrome and copper mordants are particularly suitable for postmordanting. Both appear to improve the color fastness to light and washing (Ashok, 2011).

Simultaneous mordanting

Simultaneous mordanting is a technique in which the mordant as well as the dye is mixed together and the fabric is soaked and boiled, in this mixture (Pantian, 2007).

2.3. Enzymes

Enzymes are biological molecule (proteins) that act as catalysts and help complex reactions occur everywhere in life. Enzymes are

- Very efficient catalysts
- Have high degree of selectivity
- Environmentally friendly
- Sustainable
- Biodegradable
- Require non toxic metals
- Operation at mild condition
- Can be reused (immobilized)
- Low energy consumption
- Can be produced in unlimited quantity (Prusti *et al.*2009).

Enzymes increase the rate of chemical reactions taking place within living cells without themselves suffering any overall change. They make biochemical reactions happen faster they would otherwise. Sometimes the essential reactions would not happen at all without the help of enzymes. Being catalysts also means that enzymes are not part of the final product. Enzymes are ready to catalyze a new biochemical reaction (Blackburn, 2005). Most industries have production waste .when using chemicals. These present a threat to nature. Enzymes can do the same job cheaper and do not threaten the environment .Enzymes are a part of nature and therefore fully biodegradable.

2.3.1. Cellulase

Enzyme that can degrade cellulose is called cellulase.It can be used to cut high molecular weight cellulose (wang, 2008). Although many cellulose –degrading microorganisms grow rapidly, only a few produce extracellular cellulases capable of crystalline converting cellulase into glucose. The cellulase activity is stable and may be stored for months under refrigeration or as tone extract powders, without any

significant loss of activity. cellulase enzyme is produced when cellulose is present in the medium ,as the cellulase is an inducible enzyme (Herbed ,2010).

Controlled hydrolysis by cellulase is used in textile processing to improve the surface properties and texture of cellulose –based fabric. Advanced hydrolysis is not desired since this could cause too great loss in fabric strength and weight. Using modern biotechnological tools different cellulase products having diverse cellulase profiles can be produced (Voutilainen *et al.*, 2004).

2.3.2 Application of cellulase in textile industry

Cellulase enzymes are produced by fungi, bacteria, protozoan, plants and animals. The catalytic modules of cellulases have been classified into numerous families based on their amino acid sequences and crystal structure (Bayer *et al.*, 2007). Enzyme treatments of textiles, typically cellulose materials such as cotton ,viscose or lyocel fabric ,have widely been used in the textile industry since 1980s. There are some advantages resulting from the use of enzymes for finishing cellulosic fibres and fabric (Blackburn,2005).

Cellulases are widely used in the textile industry for the manufacture and finishing of cellulose-containing materials. These enzymes are tools for improving basic processing steps in textiles manufacturing and creating new types of fabric (zhang *et al.*, 2006).

2.4 Saraca Asoka

Ashoka an ancient tree is an evergreen wild tree still found in Himalayas and across Indian sub –continent. It height is about 6-9 meters ,leaves 15-25 cm long and green in color .In spring season Asoka tree is covered with red color flowers and looks really very beautiful. The fruits are about 6-10 inches long ,looks like broad beans containing 6-8 grey color seed like chest nut (www.learningayurveda.com)

Table I

Different Names of Saraca Asoka

Languages	Name
Tamil	Ashogam
Hindi	Ashoka
Bengali	Oshok
Telugu	Vanjulamu
Oriya	Ashoka
Malayalam	Asokam
Kannada	Ashokadamara

2.4.1 Taxonomic classification

- Kingdom : plantae
- Division : Magnoliophyta
- Class : Mgnoliopsida
- Order : Fabales
- Family : Caesalpinaceae
- Genus : Saraca
- Species : Asoca (Gupta, 2007).

2.4.2 Botanical Description

Saraca indica or Saraca Asoka is a small evergreen tree of 7-10cm high .It occurs up to the altitude of 750 meters. Leaves are par pinnate 15-20 cm long and the leaflets 6-12 oblong and rigidly sub- coriaceous.Leaves are narrowly lance late, cork like at the base and stipules are intra- petiole (Ganpathy,2000).

Stem bark are irregular and rough due to the presence of rounded or projecting lenticels. Bark channeled smooth with circular lenticels and reversely ridged, sometimes cracked .Fracture splinting exposing striated, a thin whitish and continuous layer is seen below the cork. Flowers are fragrant. Flower is polygamous apetalous, yellowish orange (Sharma *et al.*, 2012).

2.4.3 Uses

By research of Asoka tree by many pharmacy companies it is found that it contains haematoxylin, tannin, iron, silica, potassium, magnesium, calcium and glycoside .Saraca Asoka dried bark as well as flower is given as a tonic to ladies in case of uterine disorders. (Anitha *et al.*, 2009). Asoka is used in many pharmacological activities like anti cancer , anti-hemorrhagic, anti - toxic, anti-microbial and have extended uses in ayurveda, unani and homeopathy. It is used to treat skin infections.

The bark of Asoka tree has medicinal value and it is reported to have a stimulating effect on the endometrial and ovarian tissue. The bark is useful in all cases of uterine bleeding where ergot is indicated (www.ashoka-bark-saraca-indica-uses).

2.5 Silk

2.5.1 Introduction

Silk fabric is the strongest natural fabric in the world and is practical for many uses. Silk was discovered in China around 2600 B.C., and China continues to lead the world in silk production , followed by Japan, Brazil, and India. The united states is the leading manufacturer of silk products ,while France is and the primary manufacturer of silk fabric. Because of its strength beauty and luxurious feel, silk fabric is used for many different things (Babu, 2013).

The silk fiber is strong, translucent and possesses a uniquely dry crush feel. It is a protein fiber made from silkworm cocoons. These cocoons are unraveled into a fine strand called filament silk which is then plied into thread .In comparison spun silk are carded and spun .The filament silk are capable of the highest sheen while spun silk tend to be matte or nearly matte in finish (Michler,2003).

2.5.2 History

The history of silk development spans through centuries and can be traced around the world's very ancient trade route called silk road. A UNESCO inspired team trekked this obscure yet historical caravan tract called fabulous silk from China and India were carried to Europe through this 6,400 km silk road. Silk road originated in Sian, followed the Great Wall of China to the north-west (Datta, 2007).

Sericulture entered Europe during 140-86 BC. India has equally long 3,000 year history before industrial revolution but later with perinea disease destroying the crop, took to silk processing and weaving where it excelled (Anita *et al.*, 2012). Silk follows the store of this remarkable through 5,000 years, reproducing hundreds of fabric, furnishings and garments from all parts of the world. From ancient China, where the secret of reeling silkworm cocoons was first discovered (Philippa, 2001).

Reeling silk and spinning were always considered household and were carried out in workshops as well as the home. In every silk producing province the technique and process of sericulture were guarded secrets and closely controlled by Chinese authorities (www.silk-road.com).

2.5.3 Physical properties

Length: silk is the only natural filament where each filament measures about 900-1200 meters sometimes even up to 2759m.

Diameter: silk is very fine with a diameter of 9-11 microns.

Luster: silk has soft luster with an occasional sparkle. The triangular cross section and smooth surface are responsible for this, Wild silk have a duller luster due to irregular surface (Parameswaran, 2011)

Strength: Silk is one of the strongest natural fibers but loses up to 20 per cent of its strength when wet.

2.5.4 Chemical properties

Silk emitted by the silkworm consists of two main proteins, sericin and fibroin. Fibroin being the structural centre of silk, and sericin being the sticky material

surrounding it. Silk is resistant to most mineral acids, except for sulfuric acid, which dissolves it and is yellowed by perspiration.

2.5.5 Advantages

- Silk is highly absorbent and like wool, can take in much perspiration and atmospheric moisture and not feel damp.
- Silk does not pill and has moderate abrasion.
- Silk is naturally somewhat wrinkle resistance.
- It has a poor resistance to the effect of ultra violet (Mondelson, 2005).

2.5.6 Dyeing of silk with natural dyes

The extracted natural dyes were applied to the dyeing of silk fabrics using different methods, including or excluding a mordant. It was found that mordant had a significant effect on the color of dyed silk fabric. The silk fabric dyed with the pre-mordant using potassium sulphate as a mordant showed a bright shade (Elsevier, 2013).

Silk was boiled in water containing the ashes of lacin tree fruits. Extraction was followed by the stage of processing –bleaching. Silk was placed in vessels where it soaked then it was covered in powder. When the powder sank silk was taken out and shaken. Black color was obtained with the help of vitriol. Indigo leaves were the most popular for dark blue (Kumaresan, 2011).

3. EXPERIMENTAL PROCEDURE

Experimental procedure pertaining to the study “**Enzyme Assisted Natural Dye Extraction From Saraca Asoka Wood and Its Application on Silk Fabric**” are discussed under the following headings:

3.1 Extraction of natural dyes

- 3.1.1 Selection and collection of dye source
- 3.1.2 Processing of natural dye
- 3.1.3 Conventional extraction of dye from Asoka wood
- 3.1.4 Enzyme assisted extraction

3.2 Optimization of various parameters for dye extraction

- 3.2.1 Solvent
- 3.2.2 Dye concentration
- 3.2.3 Time
- 3.2.4 pH
- 3.2.5 Temperature
- 3.2.6 Extraction of dye under optimized conditions

3.3 Dyeing

- 3.3.1 Selection of fabric
- 3.3.2 Pre-treatment of the fabric
- 3.3.3 Degumming
- 3.3.4 Selection of mordant
- 3.3.5 Selection of mordanting techniques
- 3.3.6 Dyeing parameters

3.4 Dyeing of Selected Fabric using Dye Solution Extracted by Conventional and Enzyme Assisted Methods

3.5 Evaluation of Dyed Fabrics

- 3.5.1 Subjective evaluation – Visual inspection

3.6 Objective evaluation

- 3.6.1 Fabric weight
- 3.6.2 Fabric thickness
- 3.6.3 Abrasion resistance
- 3.6.4 Fabric strength and elongation
- 3.6.5 Fabric stiffness
- 3.6.6 Drape test

3.7 Wetability and absorbency test

- 3.7.1 Drop test
- 3.7.2 Sinking test
- 3.7.3 Capillary rise test

3.8 Color fastness tests

- 3.8.1 Fastness to crocking
- 3.8.2 Fastness to sunlight
- 3.8.3 Fastness to wet and dry crocking
- 3.8.4 Fastness to washing

3.9 Statistical analysis

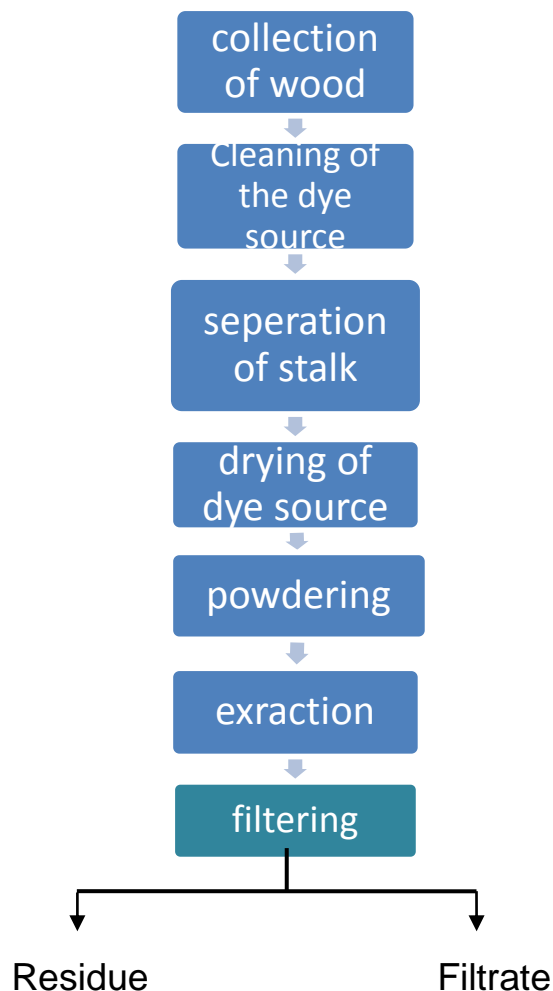
3.1 Extraction of Natural dye

3.1.1 Selection and collection of Dye Source

Natural dyes can be defined as those colorants obtained from animal or vegetable matter with no processing involved (Clark, 2011). The bright yellow corolla tubes of the Saraca Asoka wood contain a coloring substance nyctanthin. Asoka wood was selected for the present study as natural dye source. It is non-carcinogenic in nature and so does not affect human body and environment. Hence it was selected for the study. Asoka wood was collected from the Avinashilingam University compous.

3.1.2 Processing of natural dye

The flow chart for extraction of dye components from plant sources are as follows:



3.1.3 Conventional Extraction of dye from Asoka wood

To six grams of Asoka wood powder, 100 ml of water was added and allowed to boil for one hour at 90°C. The resulting liquid was filtered and used as dye solution. The optical density of the dye solution was measured using a spectrophotometer at 440 nm. (Maximum absorbance wavelength).

3.1.4 Enzyme Assisted Extraction

A one ml of solution of cellulase was sprayed on 6 g of Asoka wood powder and left overnight. After that 100ml of Water was added to it and placed in water bath for one hour. Color intensity was determined using spectrophotometer at 440 nm.

3.2 Optimization of various parameters for dye extraction

3.2.1 Solvent

To determine the suitable solvent for extraction, 6 g of Asoka wood powder was crushed with 100ml of different solvents such as water, ethanol, and NaOH individually and placed in water bath for one hour at 90°C. The resulting dye extract was filtered and the color intensity was measured at 440 nm using spectrophotometer. The solvent which showed higher absorbance was selected as the suitable solvent and used for further study.

3.2.2 Dye concentration

To determine the optimum concentration of the dye source, Asoka wood at various concentration such as (1, 2,3,4,5,6,7,8,9,10 %), was taken individually and placed in water bath for one hour. The optical density of the dye solution was analyzed spectrophotometrically at 440 nm. The concentration at which the absorbance was maximum was fixed for the subsequent study.

3.2.3 Time

To determine the optimum extraction time, the dye extraction was carried out at different time intervals such as 30-180 min at an interval of 30min. The dye solution was analyzed spectrophotometrically at 440 nm. The time at which the absorbance was maximum was selected as optimum time.

3.2.4 pH

To determine the optimum pH for dye extraction, the pH of the solvent was adjusted to 1, 3, 5, 6, 7, 8, 9, 10, 12, using 1N HCl or 1N NaOH. Optical density of the dye solution was determined.

3.2.5 Temperature

To determine the optimum temperature the dye extraction was carried out at different temperatures such as 30-90°C at an interval of 10°C. The optical density of the dye solution was analyzed spectrophotometrically at 440 nm. The temperature at which the absorbance was maximum was selected optimum temperature for dye extraction.

3.2.6 Extraction of dye under optimized conditions

Dye was extracted using suitable solvent under optimized conditions of pH, time, concentration and temperature. The dye extract was filtered and used as dye solution.

3.3 Dyeing

3.3.1 Selection of fabric

The silk is characterized by its good insulation properties warm in winter, cool in summer, good elasticity, resilience, absorbency, luster, stronger, and also reflects light, have good affinity to dye, it is very comfortable for clothing, 100 per cent silk fabric was selected for the study.

3.3.2 Pre- treatment of the fabric

The aim of the preparatory process is to improve the quality by removing impurities and foreign matters thoroughly and uniformly from the fabric and makes the fabric suitable for follow up process. (Anthappan et al., 2006)

3.3.3 Degumming

Degumming removes accompanying substances like fats, oils, natural's pigments and mineral components (choudhury, 2006)

Recipe:

Weight of the material	-100gms
MLR	-1:10
Wetting agent	-0.5 g/l
Natural soap	-20g/l
Soda ash	-2 g/l
Temperature	-90-95°C
Time	-90-120min

Procedure

The selected material was degummed in a bath containing 20gm of natural soap, 2g of soda ash and 0.5 g of wetting agent, with MLR of 1:10 at 90-95°C for 1^{1/2}-2 hours. Cool down to 70°C and then switch off the bath. After degumming, the material was washed thoroughly with warm and cold water successively.

TABLE 2**Nomenclature of the Dyed Sample**

Samples	Code
Original silk Fabric	OS
Fabric dyed with Conventional Extraction	CDS
Fabric dyed with Enzyme assisted dye extract	EDS

3.3.4 Selection of mordant

The mordant is the chemical link that fixes the dye to a substrate by combining with the dye pigment to form an insoluble compound (www.earthues.com).

Gehring, (2011) defines mordant as chemicals that help to keep dyes from fading, changing color, washing out or rubbing off. Keeping the above points in mind the investigator selected mordant such as pomegranate rind powder, amla powder, orange peel powder and alum.

Among the natural mordant, selected pomegranate rind gave better color when compared with other mordants. Hence pomegranate rind powder as mordant was selected for this study.

3.3.5 Selection of mordanting techniques

Mordanting is a process of impregnating textiles with mordants usually salt or acid to faster the dye stuff, which is applied before (or) after the dye stuff. The three ways of mordanting are pre-mordanting, post mordanting and simultaneous mordanting.

In the pre-mordanting technique the sample is mordanted and then dyed. In post -mordanting technique the sample is first dyed and then mordanted. Simultaneous mordanting is which mordanting and dyeing are carried out at the same time (Jacob, 2006).

Among these three mordanting techniques, the pre-mordanting technique which gave better results when treated with pomegranate rind power, which is a natural mordants was selected for this study.

3.3.6 Dyeing parameters

Dyeing parameters namely dye concentration, dyeing time, mordant concentration and mordanting time were optimized. To determine the optimum dye concentration, different percentage of dye extract such as 20, 30, 40, 50, 60, 70, 80 and 100 % was used for dyeing. The optimum dye concentration was selected based on the shade produced on the fabric. To evaluate the optimum dyeing time, dyeing was carried out at different time (30-180). Dyeing temperature was determined by dyeing the selected fabric at different temperatures (30-100). The optimized parameter for dyeing the selected silk fabric is as follows.

Material: liquor ratio	:	1:20
Dyeing time	:	1 hour
Dyeing temperature	:	100° C
Mordant soaking time	:	1 hour
Mordanting temperature	:	100°C

3.4 Dyeing of selected fabric using dye solution extracted by conventional and enzyme assisted methods

The dye solution extracted by conventional and enzyme assisted method was filtered. The selected silk fabric was dyed with dye solution obtained by conventional and enzyme assisted methods. Dyeing was carried out for one hour at 100°C with a M: L ratio of 1:20

3.5 EVALUATION OF DYED FABRICS

3.5.1 Subjective evaluation – Visual inspection

The fabric samples dyed with Asoka wood was evaluated visually by the panel members comprising of 20 PG students specializing in the field of Textiles and Clothing, evaluated the fabric for general appearance, brilliancy of shade and evenness of dyeing.

3.6. Objective evaluation

Textile testing is the process of inspecting, measuring and evaluating characteristics and properties of textile materials. Textile testing and valuable aid to those engaged in the production, distribution and consumption of textile if the instruments and techniques are used effectively. The bleached silk fabric and the dyed samples with mordant's were tested by using the sample pieces from the same relative portions of the material for their respective laboratory tests namely, fabric, thickness, strength, wet ability and absorbency tests and color fastness property.

3.6.1 Fabric weight

Fabric weight as the relative weight of the fabric and expressed as the weight of a particular size of piece as gram/ square meter or ounces /square yard. Fabric weight of the original and dyed sample was determined using GSM cutter. It is a device to cut circular specimen of 100 square centimeters of a fabric very accurately. It has four blades that cut the fabric when the hand wheel is rotated by applying light pressure. The samples were cut and weighted accurately using digital balance having 0.01 sensitivity. the value in grams multiplied by 100 gives/ square meter of the fabric.

The samples were weighted for five times and the mean value was calculated and recorded.

A sample of 10cm X 25cm was cut using a GSM cutter. and electronic weighting balance was to find out the weight of the samples. The inference obtained is calculated using the formula

$$\text{Grams per square meter (GSM)} = \frac{\text{Weight of the fabric x Square meter}}{\text{Area of square}}$$

Weight of the fabric = X (g)

Square of the fabric = 100cmX 100cm = 10000cm²

Area of square = length X breath square unit

The same procedure was followed to find out the fabric weight of original and bleached, dyed fabrics are carefully recorded and the mean value was calculated.

3.6.2 Fabric thickness

Fabric thickness is defined as the distance between lower and upper surface of the material measured under a standard pressure, using Shirley thickness tester with an accuracy of 0.01 mm (stocker et al., 2005). Fabric thickness gauge foot. Pressure was given at the foot to make the gauge zero. The samples were placed between the cleaned pressure foot and anvil. The reading shown by the dial was noted for each samples at five different places away from two inches of the selvedge.

3.6.3 Abrasion resistance

Abrasion is defined as the wearing a way of any part of the fabric by rubbing against another surface. Fabrics are subjected to abrasion during their lifetimes and this may result in wear, deterioration, damage and loss of performance. However, the abrasion resistance is only one of several factors contributing to were performance or durability. Abrasion can occur in many ways and can include fabric to fabric rubbing when sitting, fabric to ground abrasion during crawling (Hu, 2008).

For an abrasion test, the specimen is mounted on a motor-driven rotating lower plate where it is abraded uniformly in all directions by the motion of the effect rotating abrading mounted strictly parallel above it. Both specimen and abrading

rotate in the same direction with approximately the same angular velocity, 250 rpm, each about its own axis. The axis are space 1 in apart and are parallel. The abrading is located with a standard weight. A count records the number of revolutions (panda, 2010).

3.6.4 Fabric strength and elongation

Breaking strength is the measure of the fabric to a tensile load or stress in both warp and weft direction. Elongations when measures the extent of deformation along the axis of a material under a tensile stress and expressed in units of length of the fabric when loaded.

The original and dyed fabrics were tested for tensile strength using eureka cloth tensile strength tester. 12 inches × 12 inches specimen from each samples were cut in warp and weft direction of the fabric, 2 inches apart from selvedge. The specimen was placed between the upper and lower clamp. The dial reading was set to zero by adjusting the pendulum over the quadrant scale. The elongation pointer was checked for its position zero. Before starting the machine the pendulum lock was released and the machine was stated to run. A certain point the fabric starts to break, the machine was switched off and the dial reading in lb was taken.

Elongation reading was noted from elongation scale. The specimen was removed and the machine positioned back to original and the five specimens of both directions from each sample were tested and readings were noted.

3.6.5 Fabric stiffness

Fabric stiffness indicates the resistance of the fabric to bending and it is a key factor in the study of handle and drape (angappan and gopalakrishnan, 2000). Shirley stiffness tester was used to test the stiffness of the fabric. Sample was placed on the platform with template at the top of it, so that the leading edges coincide. Both were slowly pushed forward until the leading edges of the sample and template project beyond the edge of the platform. The sliding of the sample was stopped when it cut both the index lines. Then the bending length of the sample read from the scale opposite a datum line engraved on the side of the platform. Four reading were taken.

3.6.6 Drape test

Drape is the term used to describe the way a fabric hands under its own weight. It is one of most important fabric characteristics. Since it shows how good a garment looks in use. In the drape test, the specimen deforms with multi-directional curvature and consequently the results are dependent to a certain amount upon the shear properties of the fabric, but are mainly dependent on the bending stiffness (song, 2011)

The most commonly used instrument for measuring the drape value is cusick drape meter. A circular piece of fabric was held by two small circular plates, so that its free edges down under their own weight. For ordinary textile fabrics, a satisfactory spread of difference in drape behavior is obtained, when the diameter of fabric specimen is 30cm and the diameter of the disc is 18cm.

A value known as drape co-efficient F1 is determined by considering the following ,

A_D – the area of the specimen

A_d - the area of the supporting disc

A_s - the actual projected area of the specimen

The drape co-efficient is given by

$$F = \frac{A_s - A_d}{A_d} \times 100$$

Thus, ten readings were taken from original and all finished samples. Then their mean value was calculated and tabulated.

3.7 Wettability and absorbency test

The wettability and absorbency tests include drop test, sinking test and capillary rise test.

3.7.1 Drop Test

A burette filled with distilled water was clamped in a stand was mounted in an embroidery frame and placed at the base of the stand. The distance the sample and the burette nozzle was kept constant. The nozzle of the burette was opened just to

allow a drop of water to fall on the samples. The stop watch was started simultaneously and it was stopped when the drop of water fully sank into the material. The time taken for this was noted. The same procedure was repeated for ten times for the original, bleached and dyed samples and then means value was calculated and recorded.

3.7.2 Sinking test

A 100ml beaker was filled with distilled water. The samples were dropped on the surface of the water from a standard height. The stop watch was started the fabric struck the surface of water and stopped when the last corner sank below the water surface and time required for the sample to sink was noted. The same procedure was repeated for all samples. The mean value was calculated (Choudhury, 2006).

3.7.3 Capillary rise test

The samples were cut into sizes of 15cm length and 25cm width from the original and dyed samples. One end of the samples strips was pasted with a glass red which was placed on heavy wooden blocks and at the other end, two grams weight was attached to keep the samples straight. At the weighed end 2cm of the samples was allowed to immerse in a tray of distilled water. The rate of the water level in the sample was measured after 60 seconds (MacDonald,2008).

3.8 Color fastness tests

Dye fiber interactions are varied and their strength or combined strength determines both the outcome and performance of the dyeing.

Color fastness properties, of the dyed textile material depend upon the nature of the, dyeing procedure, washing methods and after treatment methods. The grey scale employed for color fastness test is 1-5 grades in this scale one means poor and 5 represents excellent color fastness (smith, 2006)

Three color fastness tests were carried out. They are color fastness to sunlight, washing and crocking.

3.8.1 Fastness to crocking

Sasmira crock meter was used for ascertaining the fastness of dyed textile to wet and dry crocking. It has two metal blocks. The base block was stationary, while the upper block had an arrangement to move to and from the base by means of a rotating handle. There was a finger knob attached to the upper block to hold the silk material with ring (XXII). The samples were cut into pieces with size of 20cm X10cm, the sample was fixed on the base block with longer side in the direction of rubbing the white desized original material (5cmX 5cm) was fixed on the finger knob of upper movable block with a ring. The number of rubs given was standardized and fixed as ten rubs.

The white material was rubbed to and fro against the dyed samples along a track of 10cm with pressure of 900 grams on the finger. The color transfer from the dyed sample to the white material was assessed with AATCC grey scale for staining. The same procedure was repeated for all the samples of the dyed material. For wet crocking, the damp white material was used and the same procedure was repeated for all the dyed samples. The color staining of each dyed material to dry and wet crocking carefully observed and recorded carefully (Fung, 2002).

3.8.2 Fastness to sunlight

The color fastness of textile material to day light is very important property. Color fastness determines the resistance of a material to change its color characteristics as a result of exposure to light source (AATCC, 2007).

To test the fastness to sunlight the specimens of 16 cm × 5 cm were cut from each sample of dyed fabric and divided into 8 equal parts measured a 20 cm each, the specimens were covered with black chart. For the successive 7 days the specimens were exposed to direct sunlight. First day first portion of the specimens were exposed and accordingly seven portions were exposed to sunlight. The first portion was exposed for 7 days and the 7th portion was exposed for a day. The last portion was not exposed to sunlight and considered as standard. Comparisons were done using grey scale and the specimens were rated.

3.8.3 Fastness to wet and dry crocking

Crocking is the rubbing fastness of dyes. Crocking is the transfer of colorant from the surface of the colored fabric to an adjacent area of sample fabric or to another surface, principally by rubbing action. Fastness to crocking is important in both apparel as well as upholstery. Crocking test determines the extent to which color may be transferred from the surface of the dyed fabric to another by rubbing.

Sasmira crocking meter was used to determine the fastness to crocking. Each of dyed samples was cut in the measurement of 25 cm × 20 cm and mounted on flat base. The desized white silk fabric was mounted in a ring on rubbing finger. Each sample was given to rubs based on standardization. The procedure adopted was same as the of crocking. The color transfer from the dyed samples to the white material was assessed using grey scale.

3.8.4 Fastness to washing

Major loss of color from the fabric is due to washing and results in staining over the adjacent fabric. Test sample of the dyed fabric measuring 5× 10 cm size were cut. Each of them was sandwiched between the un dyed white cloth which was desized well. Specimen were completely soaked in the soap solution about 5 g /1 for 30 minutes at 40°C, after that the sample were removed rinsed in cold water, squeezed well and dried. Evaluations of staining on the white adjacent fabric were found using a grey scale. The same procedure was carried out for other dyed samples.

3.9 Statistical Analysis

The results of laboratory tests were analysed statistically using tests for analysis of variance (ANOVA).

PLATE 1

Selected Dye Source – ASOKA Wood Powder

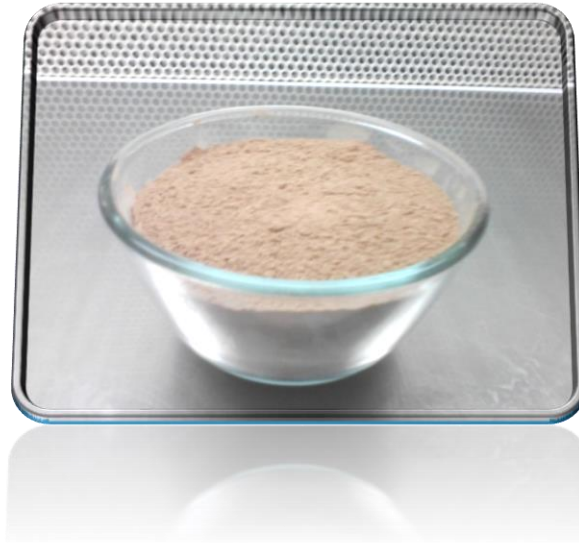
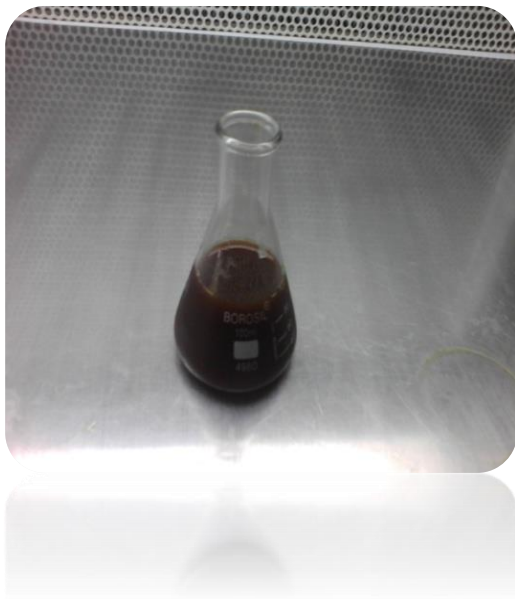


PLATE 2

Dye Extract

**Enzyme assisted
Dye Extract**



Conventional Dye Extract

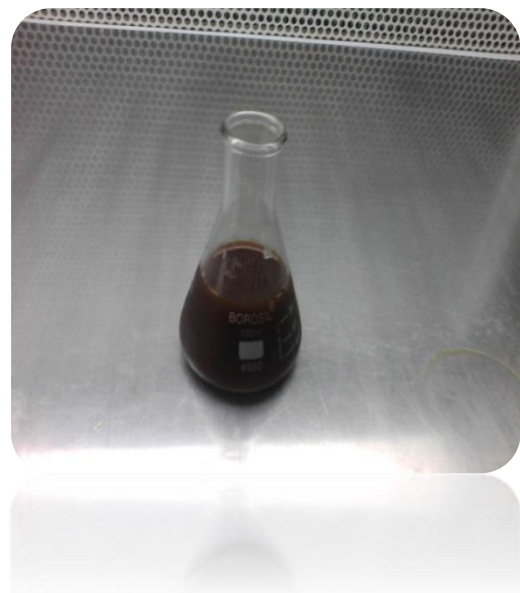
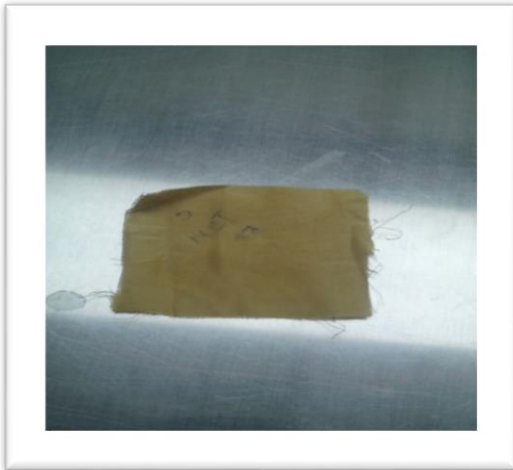


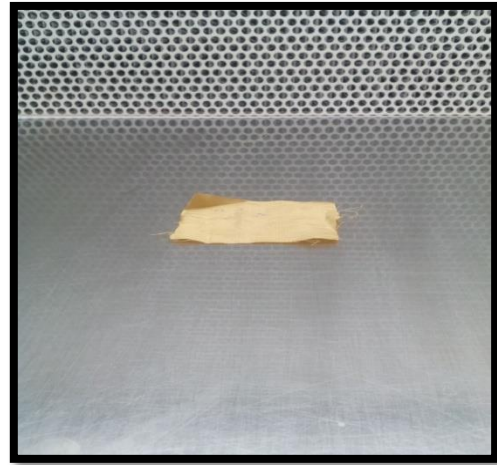
PLATE 3

Fabrics Dyed with Different Mordants

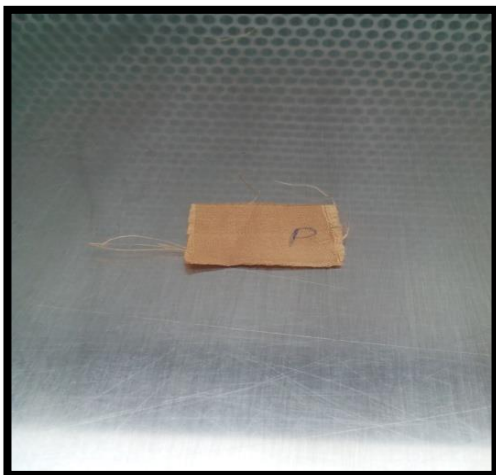
ORANGE PEEL



MYROBOLAN



POMEGRANATE PEEL



ALUM

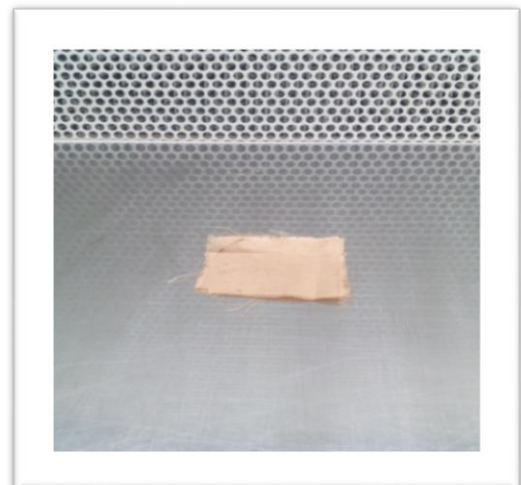
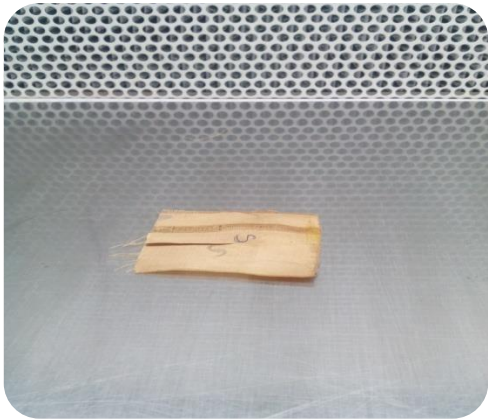


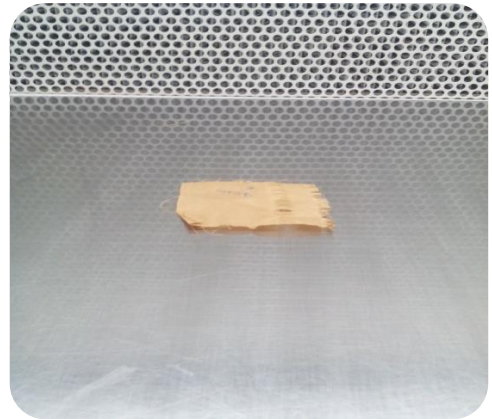
PLATE 4

Mordanting Techniques

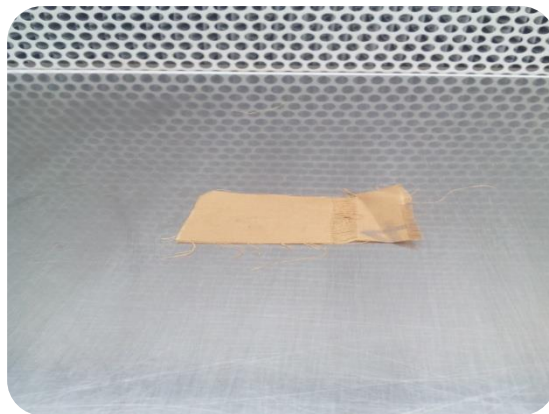
SIMULTANEOUS MORDANTING



POSTMORDANTING



PRE MORDANTING



4. RESULTS AND DISCUSSION

The results of the study on “**Enzyme Assisted Natural Dye Extraction From Saraca Asoka Wood and Its Application on Silk Fabric**” presented are discussed as follows.

4.1. Optimization of Various Parameters for Extraction of Natural Dyes

- 4.1.1. Solvent
- 4.1.2. Dye Concentration
- 4.1.3. Time
- 4.1.4. pH
- 4.1.5. Temperature

4.2. Extraction of Natural Dye under Optimized conditions

4.3. Evaluation of dyed fabric

4.3.1. Subjective Evaluation – Visual inspection

4.3.2. Objective Evaluation

- 4.3.2.1. Fabric Weight
- 4.3.2.2. Fabric Thickness
- 4.3.2.3. Fabric Strength- Warp and Weft
- 4.3.2.4. Fabric Elongation – Warp and Weft
- 4.3.2.5. Fabric Stiffness
- 4.3.2.6. Crease Recovery – Warp and Weft
- 4.3.2.7. Sinking Test
- 4.3.2.8. Drop Test
- 4.3.2.9. Drapability Test
- 4.3.2.10 Color Fastness Test

Color Fastness to Sunlight Washing,
Pressing, Drying and Crocking.

4.1 OPTIMIZATION OF VARIOUS PARAMETERS FOR EXTRACTION OF NATURAL DYE

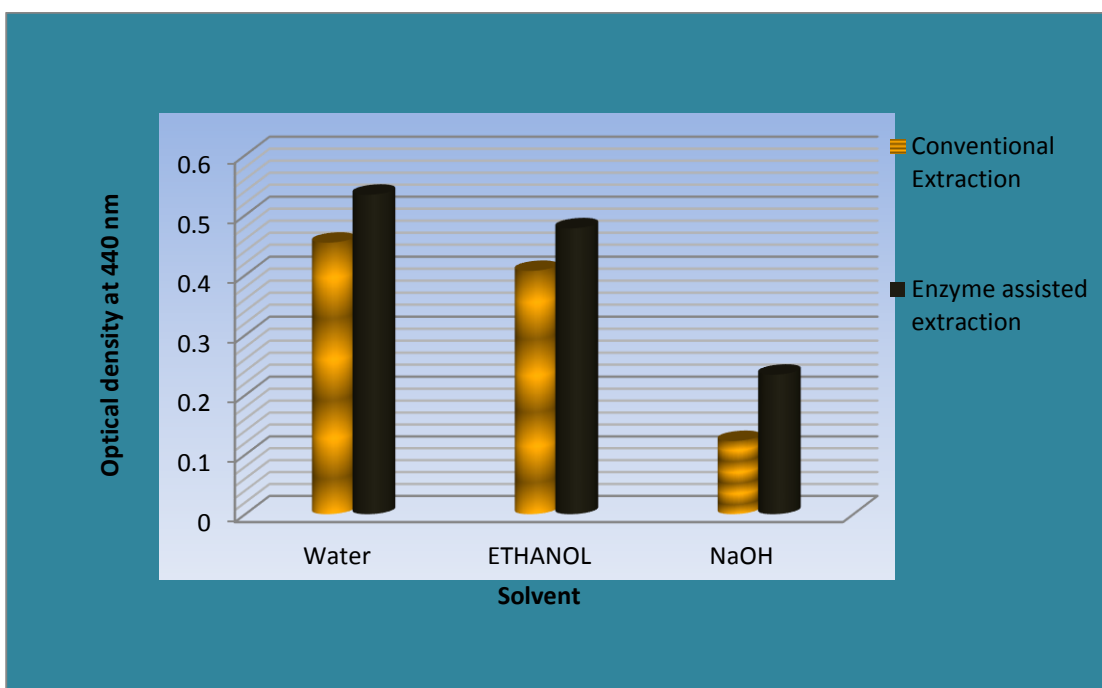
4.1.1 Solvent

The effect of various solvents on dye extraction was studied and the results are presented in Table 3 and Figure 1.

Table 3
Selection of Solvent

Solvents	Optical density at 440 nm	
	Conventional Extraction	Enzyme-assisted Extraction
Water	0.454	0.534
Ethanol	0.407	0.478
NaOH	0.123	0.234

Figure 1
Selection of Solvent



Various solvents such as water, NaOH and ethanol were used separately as extraction medium and the color intensity was measured. From Table 3, it is clear that maximum color yield was obtained with water as extraction medium, followed by NaOH, ethanol. Hence, water was selected as extraction medium for the extraction of dye from Asoka wood.

4.1.2 Dye Source Concentration

The effect of various concentration of Asoka powder was studied and the results are presented in Table 4 and Figure 2.

Table 4
Dye Source Concentration (%)

Concentration of Asoka powder (%)	Optical density at 440 nm	
	Conventional Extraction	Enzyme- assisted Extraction
1	0.142	0.184
2	0.217	0.267
3	0.335	0.356
4	0.338	0.337
5	0.407	0.492
6	0.450	0.523
7	0.589	0.558
8	0.553	0.626
9	0.541	0.634
10	0.576	0.612

Figure 2
Dye Source Concentration

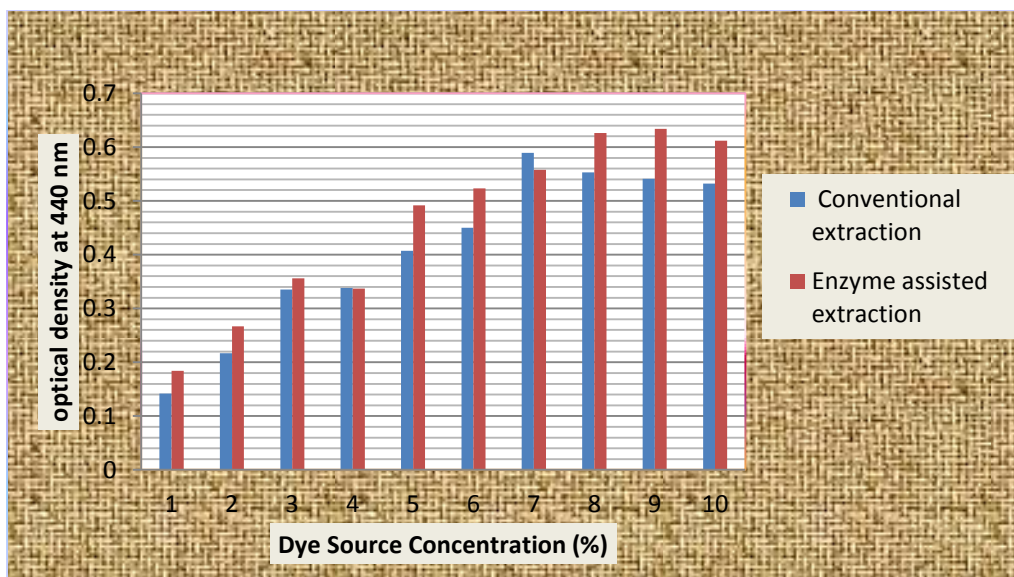


Table 4 and Figure 2, clearly indicates that the optimum dye source concentration is 7 per cent for conventional extraction with the color intensity of 0.589 and 9% for enzyme-assisted extraction (0.634). Hence on optimum dye concentration of 9% for enzymatic and 7% for conventional method was fixed for farther studies.

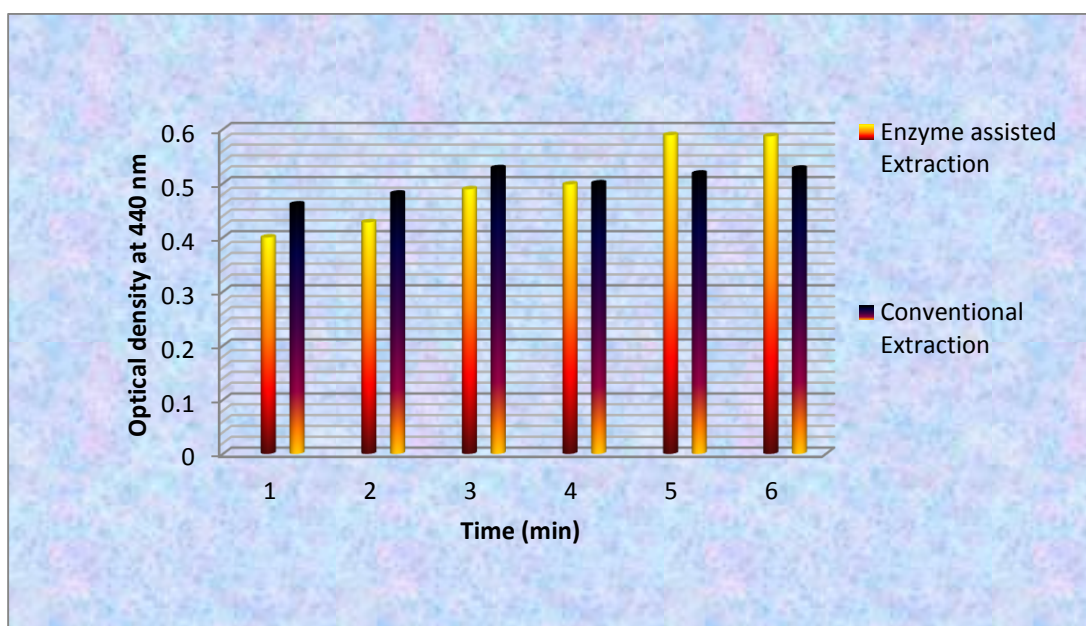
4.1.3 Time

The effect of time on the extraction of dye was determined and the results are shown in Table 5 and Figure 3.

Table 5
Contact Time

Time(min)	Optical density at 440 nm	
	Conventional Extraction	Enzyme-assisted Extraction
30	0.461	0.400
60	0.481	0.428
90	0.528	0.489
120	0.500	0.498
150	0.518	0.589
180	0.527	0.587

Figure 3
Contact Time



In extraction process, time is very important parameter because long and short time during constant heat for a long time decomposition of the dye material might occur while short time causes incomplete extraction (Agarwal, 2008). From Table 5 and Figure 3, it is evident that there was an increase in color yield with increase in time from 30- 90 mins in conventional method and 30 – 150 min in enzymatic extraction methods. Hence, optimum time for extraction of dye was selected as 90mins and 150 min for conventional and enzymatic extraction respectively.

4.1.4 pH

The effect of pH on dye extraction was studied and the results are presented in Table 6 and figure 4.

Table 6

Optimization of pH

pH	Optical density at 440 nm	
	Conventional extraction	Enzyme-assisted Extraction
1	0.158	0.162
3	0.166	0.176
5	0.241	0.280
7	0.465	0.303
9	0.499	0.235
10	0.325	0.377

Figure 4

Optimization of pH

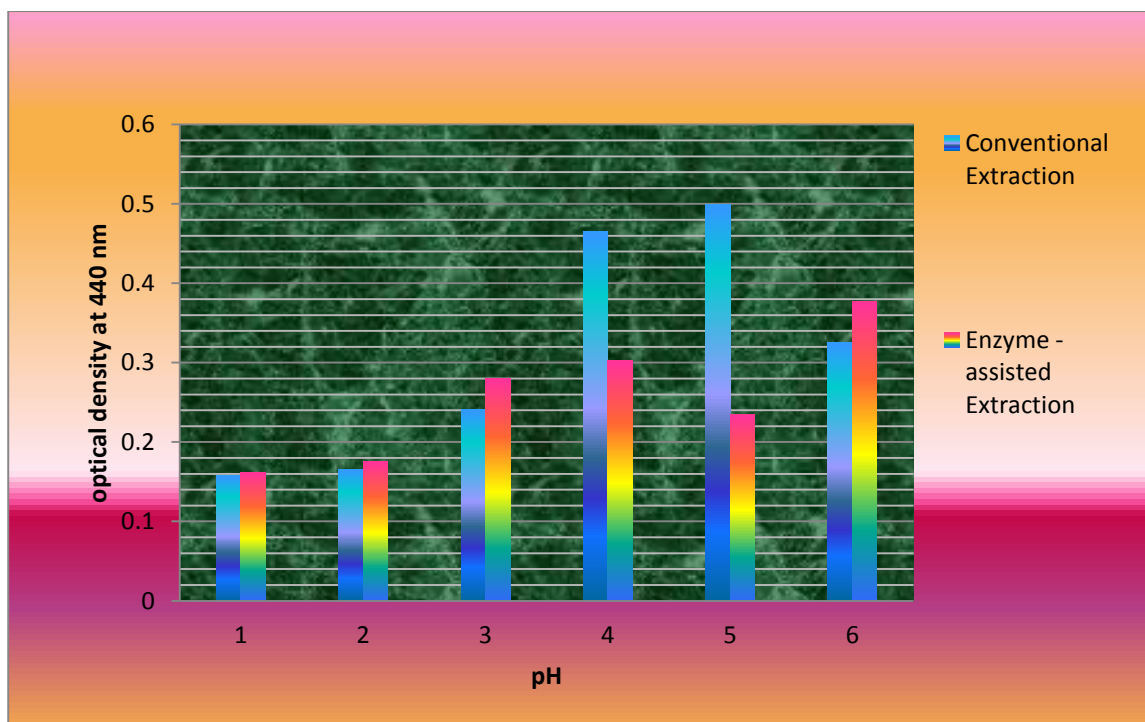


Table 6 and Figures 4 clearly indicates that there was an increase in dye extraction up to pH 9, in conventional extraction and further increase in pH decreases the color yield. Hence pH of 9 was selected as optimum pH for extraction of dye from Asoka wood by conventional method. The pH of extraction solution is an important process parameter because it has great impact on the stability of natural dyes. Hence the initial pH of the extraction medium was maintained at 9 for conventional extraction and pH 10 for enzymatic extraction.

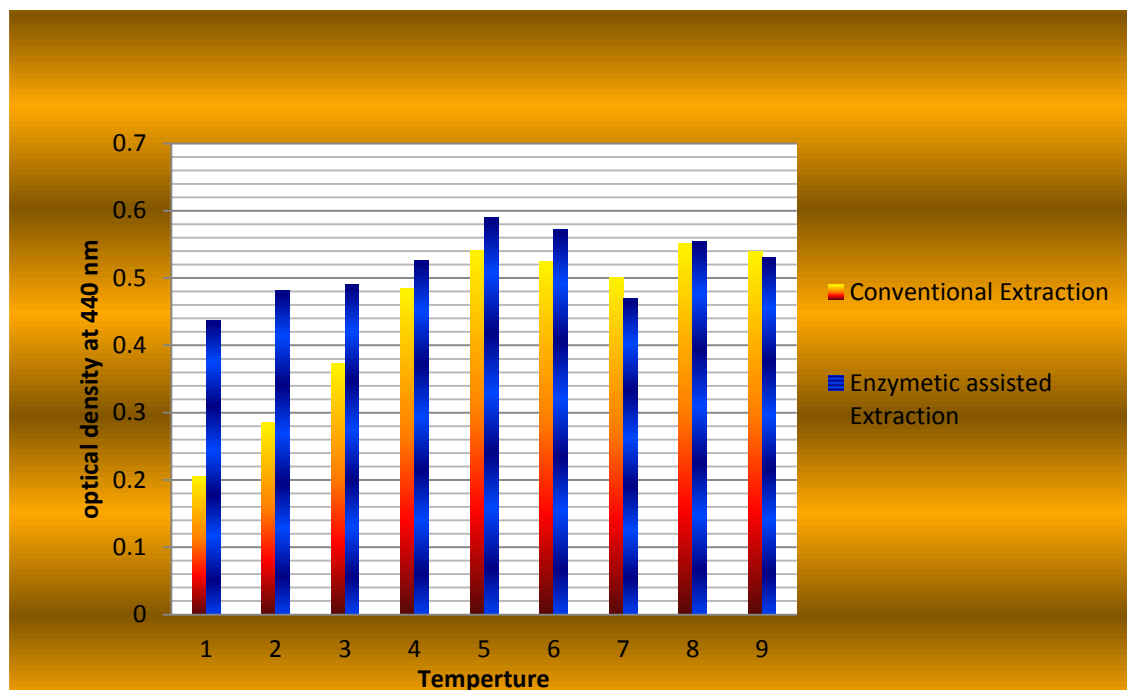
4.1.5 Temperature

To determine the optimum temperature for the effective extraction of natural dye, extraction was done at different temperatures from 30- 100°C and results are shown in Table 7 and Figure 5.

Table 7
Temperature

Temperature (°C)	Optical density at 440 nm	
	Conventional extraction	Enzyme-assisted extraction
30	0.205	0.437
40	0.285	0.481
50	0.373	0.490
60	0.485	0.526
70	0.541	0.590
80	0.525	0.573
90	0.501	0.469
100	0.552	0.555

Figure 5
Temperature



The temperature has greater influence on the natural dye extraction as natural dyes are temperature sensitive. Thus, the temperature was considered as an important factor for the extraction of natural dye. Temperature influences the extraction yields both positively and negatively (Samantha, 2007). Table 7 and Figure 5 clearly indicate that a rise in temperature increases dye extraction and reaches a maximum at 70°C for enzyme-assisted extraction, whereas for the conventional method, it reaches a maximum at 100°C. Hence, 100°C was fixed as the optimum temperature for the extraction of dye by the conventional method and 70°C for enzyme-assisted extraction.

4.2 EXTRACTION OF NATURAL DYE UNDER OPTIMIZED CONDITIONS

Various parameters such as solvent, time, temperature, and concentration were optimized, and results are presented in Table 8.

Table 8**Extraction of Natural Dye Under Optimized Conditions**

Parameters	Conventional Extraction	Enzyme – Assisted Extraction
Solvent	Water	Water
Time	150 mins	150 mins
Temperature	100 ^o c	70 ^o C
Concentration	7%	9%

The dye was extracted under optimized conditions. The natural dye was extracted from Asoka wood using H₂O as solvent at 150° for 2.30 hour.

4.3 EVALUATION OF DYED FABRIC**4.3.1 Subjective evaluation – Visual inspection**

Subjective evaluation of fabrics dyed with conventional dye extract and enzyme assisted dye extract was carried out for appearance, brightness, and evenness of dyeing and the results are presented in Table 9.

Table 9**Subjective Evaluation**

S. No	samples	Presented in percentage						Evenness of Dyeing	
		General Appearance			Brightness of shade				
		Excellent	Good	Fair	Bright	Medium	Dull	Even	Uneven
1.	CDS	78	18	2	90	7	3	99	1
2.	EDS	96	3	1	92	7	1	98	2

CDS – Fabric Dyed with Conventional Extract

EDS – Fabric Dyed with Enzyme Assisted Dye Extract

From Table 9, it is clear that the EDS sample was rated as excellent in general appearance by 96% per cent of the judges and bright by 92% of the judges and even by 98% of the judges. In conventional dyed sample, 78% of the judges rated appearance as excellent. With regard to brightness, 90% of the judges rated CDS as bright and 99% rated the sample as evenly dyed.

Hence it could be concluded that the general appearance, brightness of shade and evenness of dyeing was better in enzymatic assisted dyed sample than when compared with conventional dyed sample.

4.3.2 Objective evaluation

4.3.2.1 Fabric weight

The fabric weight and analysis of variance of the samples OS, CDS and EDS are given in Table 10 and Figure 6.

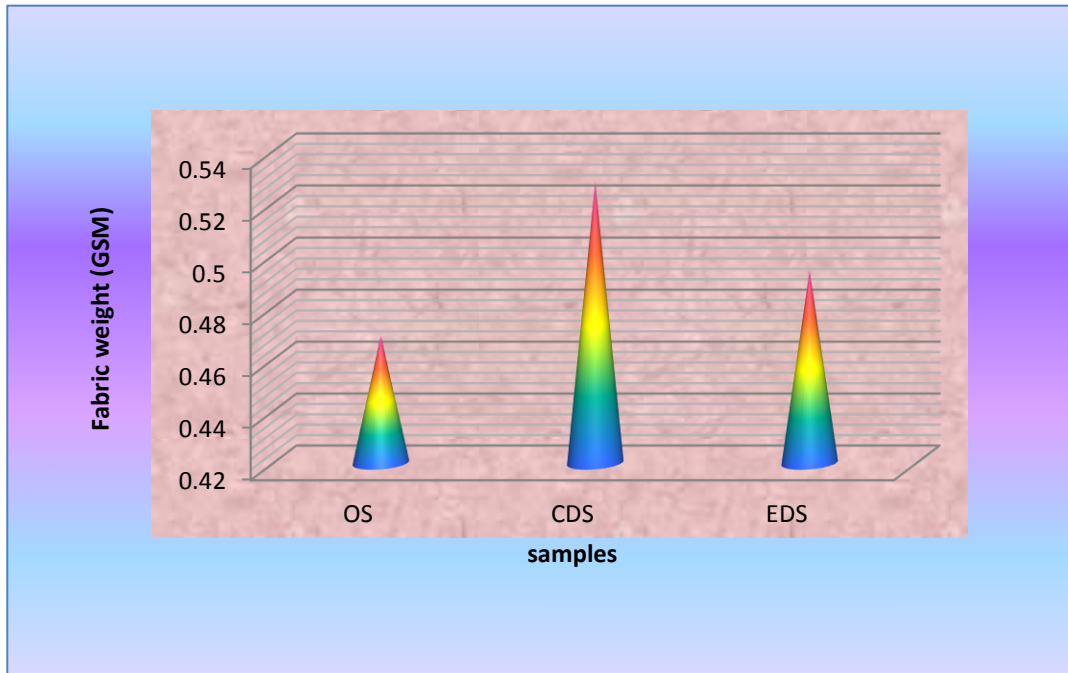
Table 10
Fabric Weight

S.No	Samples	Mean value (gsm)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	0.469	-	-	113.569 ^{**}
2.	CDS	0.528	0.059	12.57	
3.	EDS	0.494	0.025	5.33	

** - Significant at 1% level

Figure 6

Fabric Weight



From the Table 10 and Figure 6, it is clear that, the fabric weight of all the samples increased when compared to original. The increase in fabric weight is maximum in CDS sample. This results indicates that the dye absorption is maximum in conventional method. The increase in fabric weight when compared to the original was found to be significant at one per cent level.

4.3.2.2 Fabric thickness

The fabric thickness and analysis of variance of the samples OS, CDS and EDS are shown in Table 11 and Figure 7.

Table 11

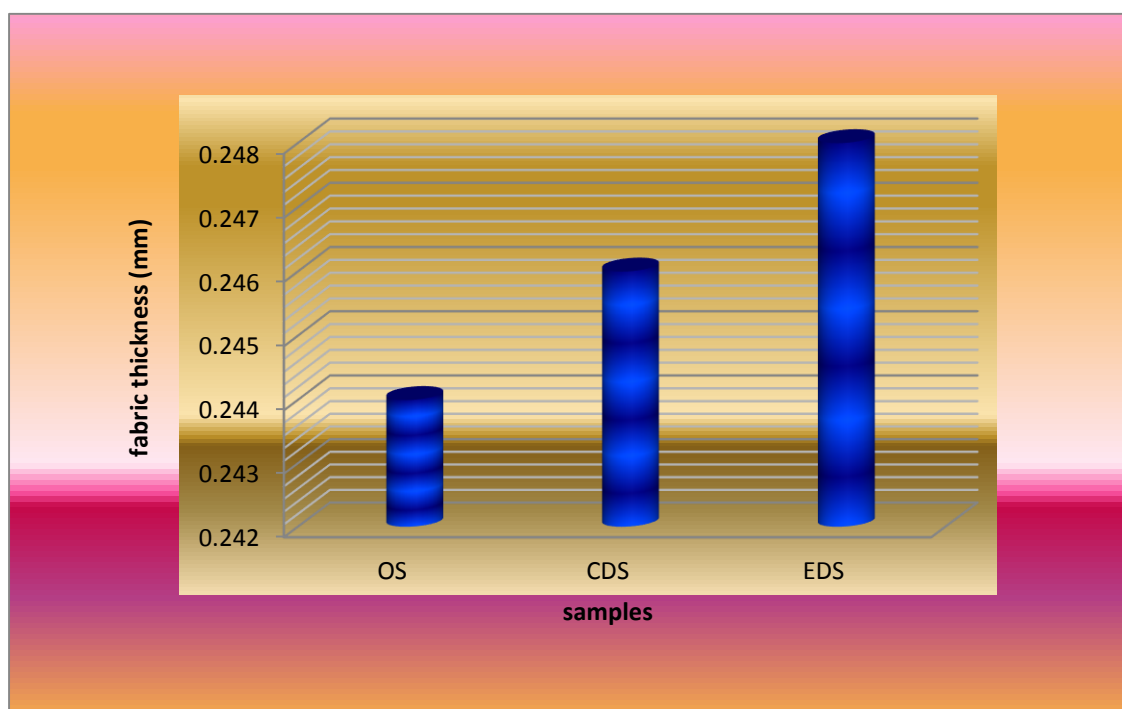
Fabric Thickness (mm)

S.No	samples	Mean value (mm)	Loss or gain over Original	% loss or gain over original	'F' Value
1.	OS	0.248	-	-	0.093 ^{NS}
2.	CDS	0.246	0.002	0.80	
3.	EDS	0.244	0.004	1.61	

Ns- Not significant

Figure 7

Fabric Thickness



From Table 11 and Figure 7, it is clear that the thickness of the samples CDS, EDS decreased when compared to the control. The decrease in fabric thickness was maximum in sample CDS. The decrease was 0.80 per cent in CDS sample and 1.61 per cent in EDS. The results indicate that the dye absorption was maximum in CDS sample. The decrease in fabric thickness was not significant when compared with the original.

4.3.2.3 Fabric strength (warp)

Fabric strength and analysis of variance of the samples OS, CDS and EDS in warp direction are presented in Table 12 and Figure 8.

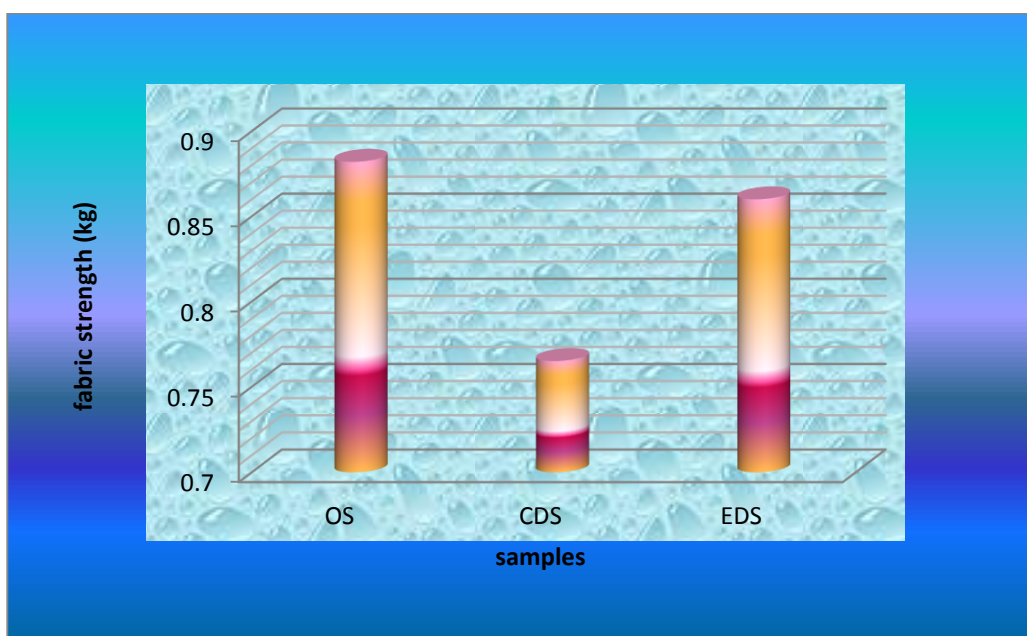
Table 12
Fabric Strength (warp)

S.No	Samples	Mean value (kg)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	0.889	-	-	113.359 **
2.	CDS	0.226	0.656	74.37	
3.	EDS	0.86	0.022	2.49	

** - significant 1% level

Figure 8

Fabric Strength (warp)



From Table 12 and Figure 8, it is clear that the strength of CDS fabric decreased by 74.37 per cent and EDS fabric by 2.49 per cent, when compared with original fabric. The fabric strength was found to be higher in EDS when compared with CDS sample. The result clearly indicates that the uptake of dye was higher in

EDS than CDS. The increase in fabric strength when compared to the original sample was found to be significant at one per cent level.

Fabric strength (weft)

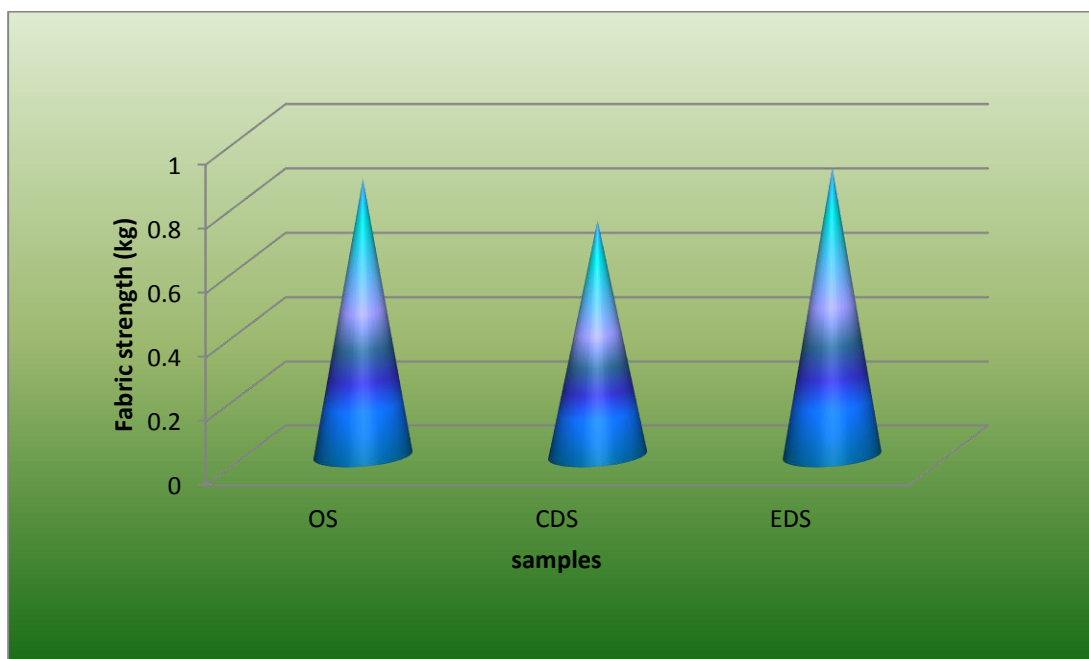
Fabric strength and the analysis of the variance of the samples OS, CDS and EDS in the weft direction are shown in Table 13 and Figure 9.

Table 13
Fabric strength (weft)

S.No	samples	Mean value (kg)	Loss or gain over Original	% loss or gain over original	'F' value
1.	OS	0.864	-	-	113.359**
2.	CDS	0.634	0.23	26.62	
3.	EDS	0.898	0.03	3.47	

** - significant at 1% level

Figure 9
Fabric strength (weft)



From Table 13 and Figure 9, it is evident that the fabric strength increased in EDS sample. The fabric strength was higher in EDS when compared to CDS fabric in both warp and weft directions. The increase in fabric strength when compared with original was found to be significant at one per cent level.

4.3.2.4 Fabric elongation (Warp)

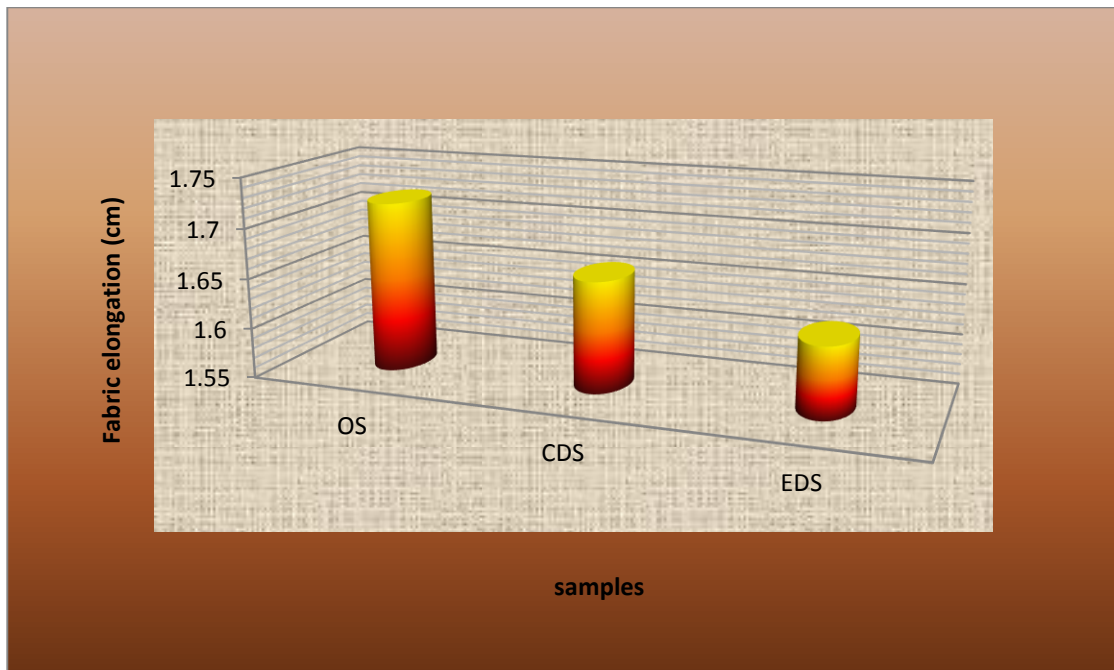
Fabric elongation and analysis of variance of the samples OS, CDS and EDS in warp direction are shown in Table 14 and Figure 10.

Table 14
Fabric Elongation (warp)

S.No	samples	Mean value (cm)	Loss or gain Over original	% loss or gain over original	'F' value
1.	OS	1.7	-	-	0.559 ^{NS}
2.	CDS	1.66	0.04	2.35	
3.	EDS	1.62	0.08	4.70	

Ns - Not significant

Figure 10
Fabric Elongation (warp)



From Table 14 and Figure 10, it is clear that the elongation of CDS and EDS sample decreased by 2.35 and 4.70 per cent respectively. The decrease in elongation is not significant when compared with the original fabric.

Fabric Elongation (weft)

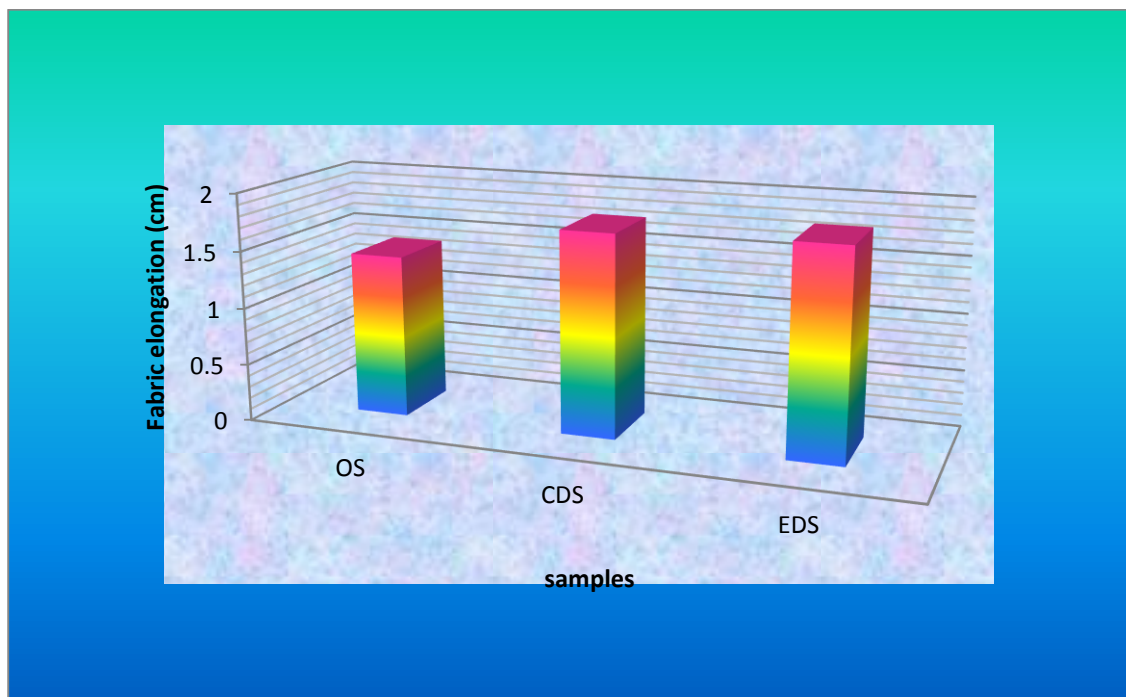
Fabric elongation and analysis of variance of the samples OS, CDS and EDS in weft direction are shown in Table 15 and Figure 11.

Table 15
Fabric Elongation (weft)

S.No	Samples	Mean value (cm)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	1.42	-	-	4.414 ^{NS}
2.	CDS	1.76	0.34	23.94	
3.	EDS	1.8	0.38	26.76	

NS – Not significant

Figure 11
Fabric Elongation (weft)



From Table 15 and Figure 11, it is clear that the fabric elongation in weft direction is increased by 26.76 per cent and 23.94 per cent for EDS and CDS respectively. The difference in fabric elongation when compared with original was found to be not significant.

4.3.2.5 Fabric stiffness (warp)

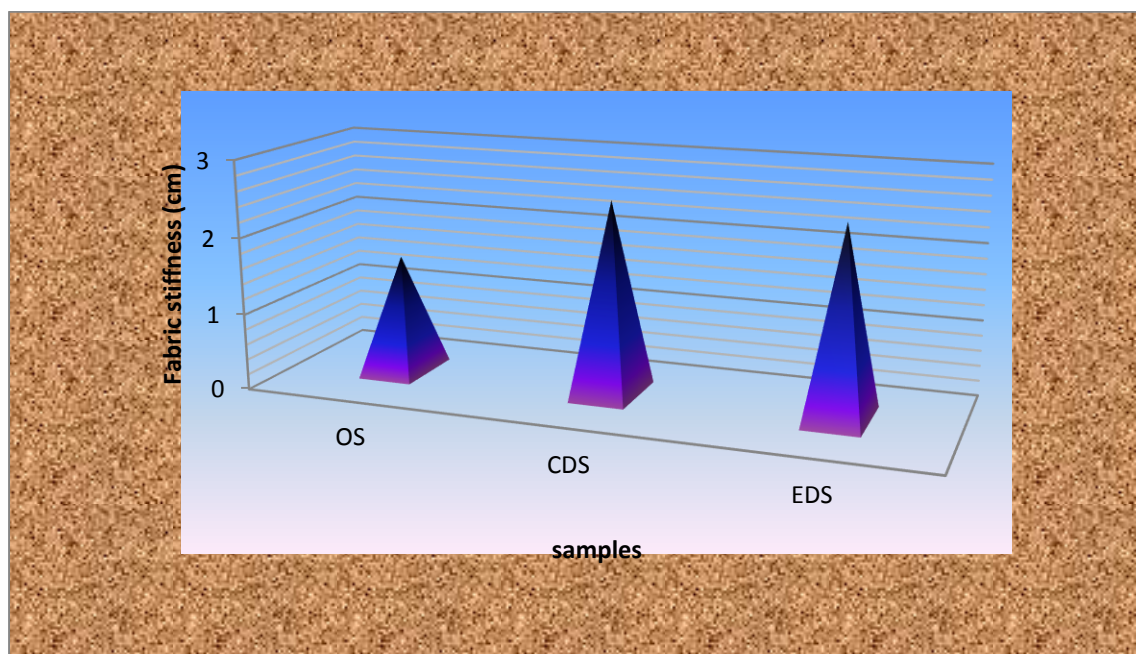
Fabric stiffness and analysis of variance of the samples OS, CDS and EDS in warp direction are shown in Table 16 and Figure 12.

Table 16
Fabric Stiffness (warp)

S.No	Samples	Mean value (cm)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	1.56	-	-	9.094**
2.	CDS	2.56	0.96	61.53	
3.	EDS	2.46	0.9	57.69	

** - Significant at 1% level

Figure 12
Fabric Stiffness (warp)



From Table 16 and Figure 12, it is clear that the stiffness of the fabrics increased when compared with original. The increase in fabric stiffness was found to be 61.53 per cent in CDS and 57.69 per cent in EDS. The increase in the fabric stiffness when compared with the original was found to be significant at one per cent level.

Fabric stiffness (weft)

Fabric stiffness and the analysis of variance of the samples OS, CDS and EDS in weft direction are presented in Table 17 and Figures 13.

Table 17

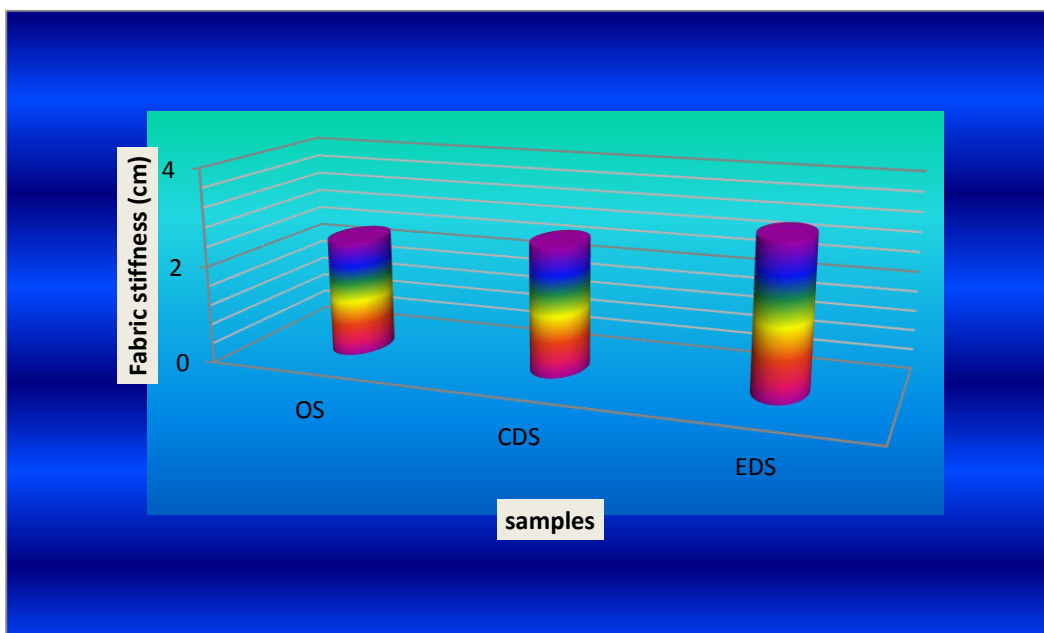
Fabric Stiffness (weft)

S.No	samples	Mean value(cm)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	2.38	-	-	5.183 ^{NS}
2.	CDS	2.62	0.24	10.08	
3.	EDS	3.1	0.72	30.25	

NS- Not Significant

Figure 13

Fabric Stiffness (weft)



From Table 17 and Figure 13, it is clear that the fabric stiffness of EDS sample increased by 30.25 per cent and ,CDS sample by 10.08 per cent over original. The increase in stiffness was found to be maximum in EDS followed by CDS. There was no significant difference in fabric stiffness when compared with original.

4.3.2.6 Crease recovery (warp)

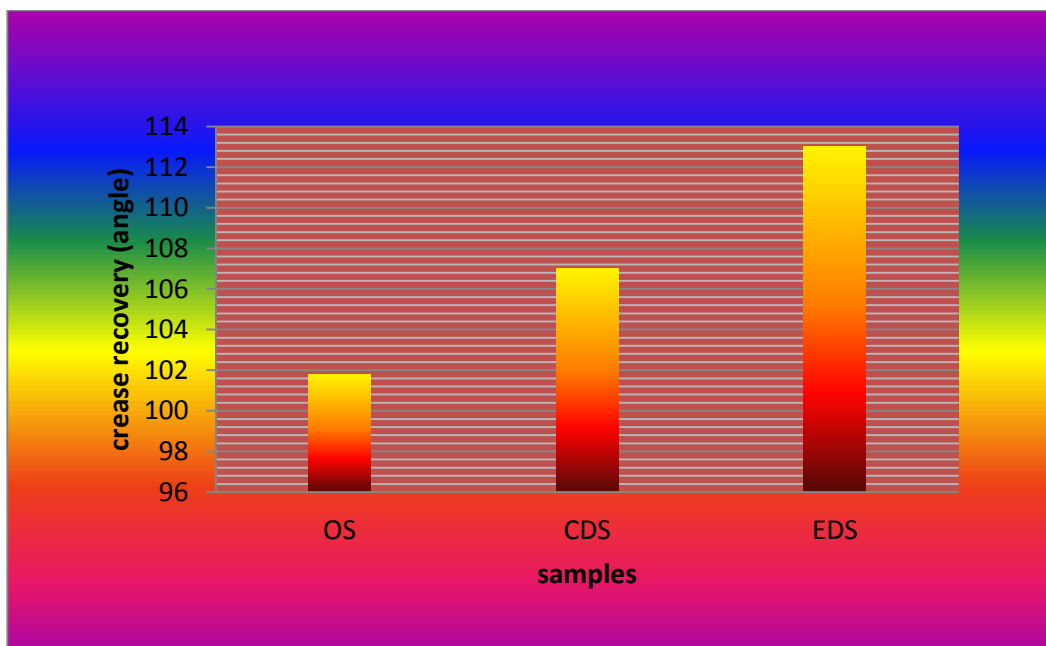
Crease recovery and analysis of variance of the sample OS, CDS and EDS in warp directions are shown in Tables 18 and Figure 14.

Table 18
Crease Recovery (warp)

S.No	samples	Mean value (angle)	Loss or gain original	% loss or gain over original	'F' value
1.	OS	101.8	-	-	1.953 ^{NS}
2.	CDS	107.0	5.2	5.10	
3.	EDS	113.0	11.2	11.2	

NS- Not significant

Figure 14
Crease Recovery (warp)



From Table 18 and Figure 14, it is clear that the crease recovery of sample EDS and CDS increased when compared to control. The increase in fabric crease recovery was maximum in sample EDS. Statistical analysis proved no significant difference in crease recovery when compared with the original in warp direction.

Crease recovery (weft)

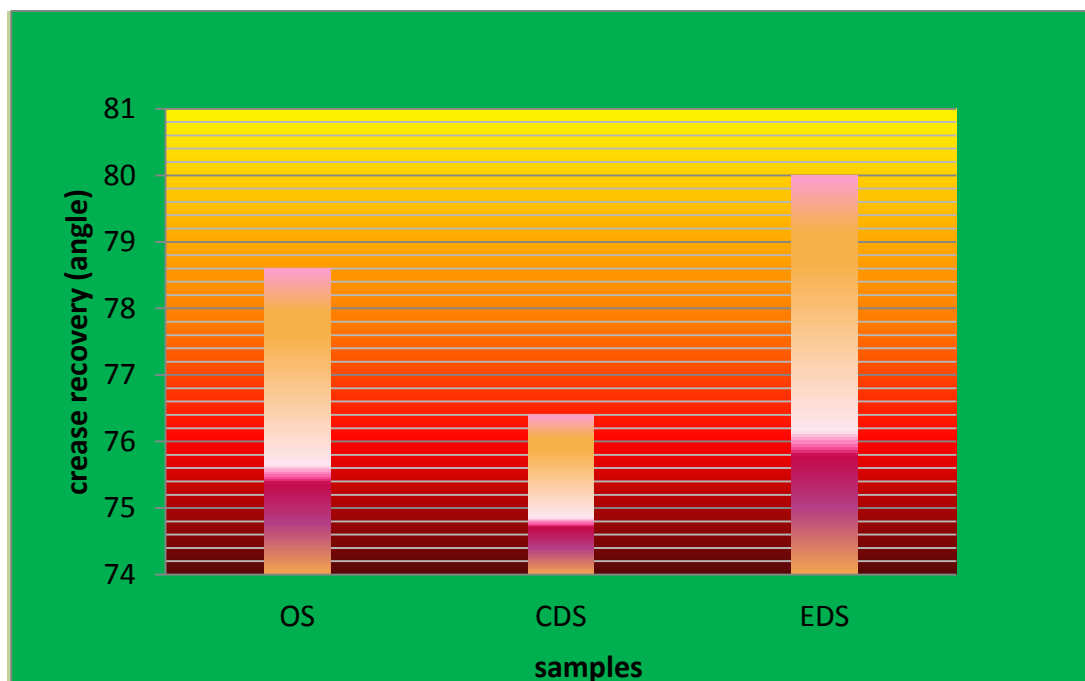
Crease recovery and the analysis of the variance of the sample OS, CDS and EDS in weft direction are shown in Table 19 and Figure 15.

Table 19
Crease recovery (weft)

S.No	samples	Mean value (angle)	Loss or gain over original	%loss or gain over original	'F' value
1.	OS	78.60	-	-	5.393 ^{NS}
2.	CDS	76.40	2.2	2.798	
3.	EDS	80.00	1.4	1.781	

NS - Not significant

Figure 15
Crease Recovery (weft)



From Table 19 and Figure 15, it is clear that crease recovery of sample EDS increased when compared to control. The increase in fabric crease recovery was maximum in sample dyed with enzyme assisted extract in both the directions. Statistical analysis proved no significant difference in weft direction.

4.3.2.7 SINKING TEST

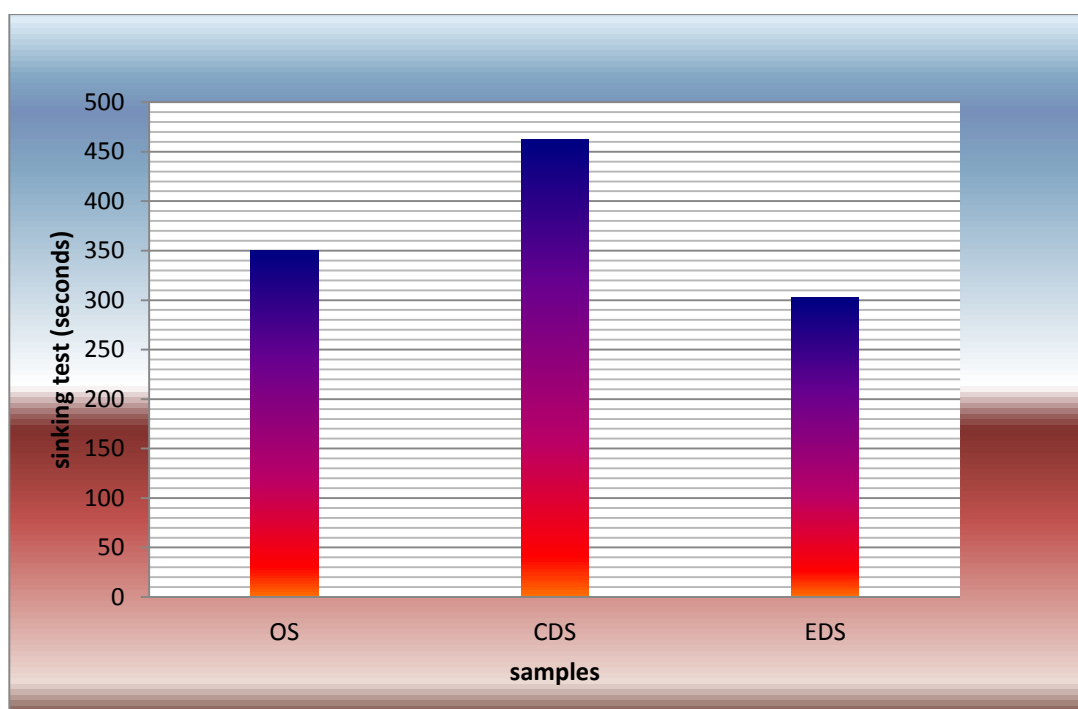
Sinking test and the analysis of the variance of the sample OS, CDS and EDS are shown in Table 20 and Figure 16.

Table 20
Sinking Test

S.No	Samples	Mean value(seconds)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	350.6	-	-	40.707**
2.	CDS	462.0	111.4	31.77	
3.	EDS	302.6	48	13.69	

** - significant at 1% level

Figure 16
Sinking Test



From Table 20 and Figure 16 it is clear that the time taken by sample EDS to is decreased when compared with the control. where as the time taken by CDS sample has increased. There was a significant difference in absorption at 1% level when compared between the dyed samples and the original.

4.3.2.8 DROP TEST

Drop test and the analysis of the variance of the samples OS, CDS and EDS are shown in Table 21 and Figure 17.

Table 21
Drop Test

S.No	Sample	Mean value(sec)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	2.00	-	-	10.300**
2.	CDS	3.00	1.00	35	
3.	EDS	4.10	2.10	100	

** - significant at 1% level

Figure 17
Drop Test

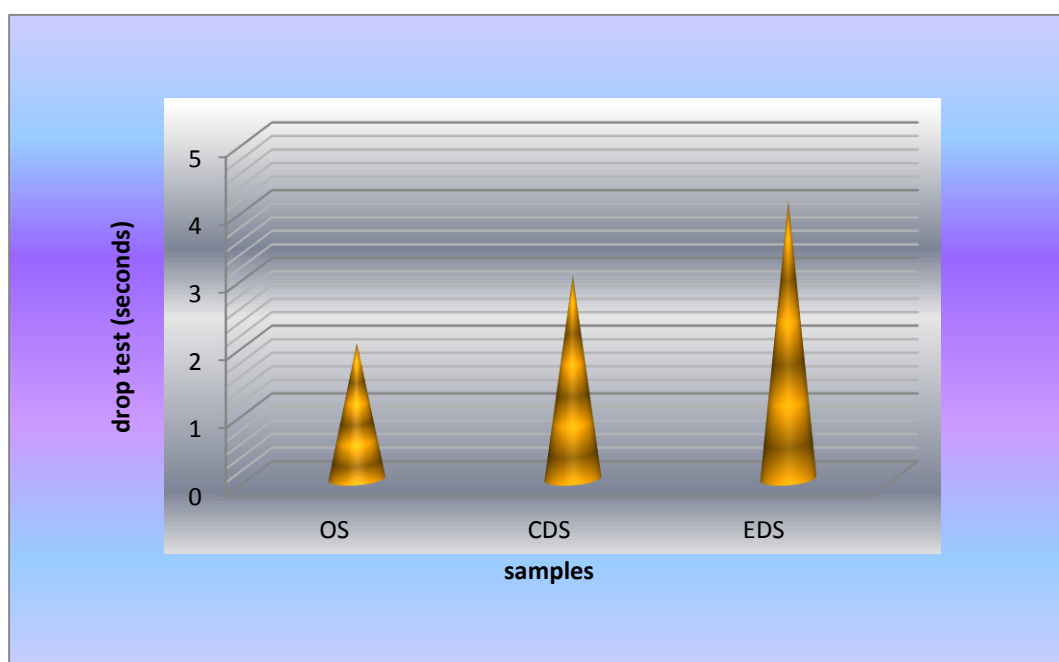


Table 21 and Figure 17 indicate that the time taken by the dyed samples to absorb a drop of water decreased when compared over original. Maximum decrease in absorbency was noted in EDS sample. Statistical analysis proved that there is a significant difference in absorption at 1% level when compared between the dyed samples and the original.

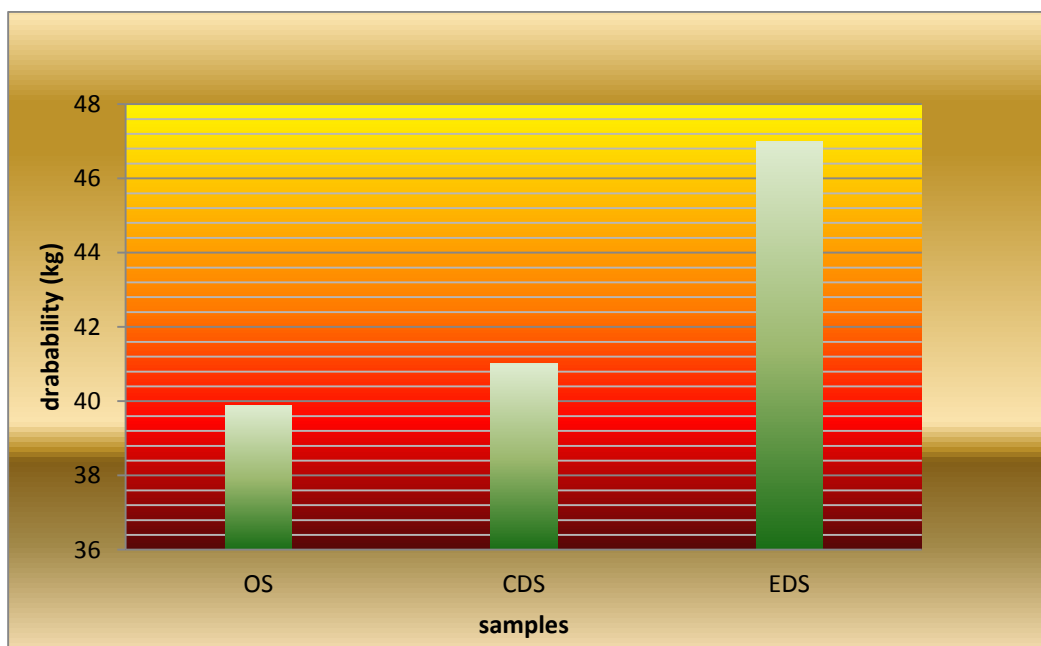
4.3.2.9 DRAPABILITY TEST

Drapability test and analysis of variance of the sample OS, CDS and EDS are shown in Table 22 and Figure 18.

Table 22
Drapability Test

S.No	samples	Mean value(kg)	Loss or gain over original	% loss or gain over original	'F' value
1.	OS	39.88	-	-	8.489**
2.	CDS	41.00	4.12	10.330	
3.	EDS	47.00	7.12	17.853	

Figure 18
Drapability Test



From Table 22 and Figure 18, it is clear that the drapability of samples CDS and EDS increased when compared to control. The increase in drapability was maximum in sample dyed with enzyme assisted dye extract and there was significant difference at 1% level when compared between the samples.

4.3.2.10 Colour Fastness Test

Colour fastness of the dyed samples to sunlight, crocking and washing were determined and the results are presented in Table 23.

Table 23
Colour Fastness to Sunlight, Crocking and Washing

S.No	sample	Sunlight		Washing		Crocking			
		Color change	Staining	Color change	Staining	Dry		Wet	
						Color change	staining	Color change	Staining
1.	CDS	4	4	4	5	5	4	5	4
2.	EDS	5	4	5	5	4	5	5	5

5- Excellent 4- very good

From Table 23, it is evident that all the dyed samples showed good fastness to sunlight. With regard to washing and crocking all the samples showed good fastness. When compared between CDS and EDS samples, EDS showed excellent color fastness to all the color fastness tests.

5. SUMMARY AND CONCLUSION

The globalization of markets and increasing demand for product has created a deep interest in the use of raw materials from natural resources. Organic awareness and environmental consciousness of consumers have revived the interest on natural dyed textiles. The major advantages of natural dyes are that they are biodegradable, non-carcinogenic, and non-mutagenic and color soothing to human eyes. Dyeing with natural dyes can be a way of value addition to the textile products.

In the present study, colorants extracted from the Asoka wood have been chosen for its dyeability. The colored constituents of Asoka wood possess properties which find good application in textile industry. The properties of Asoka wood colorants include their stability to oxidation, non-toxicity and flexibility. Traditionally, Asoka woods have been used in medicines to cure menstrual disorders associated with excessive bleeding, congestion, and pain. The silk fabric dyed with Asoka wood imparts beautiful shades and fastness properties to silk fabric.

There is growing demand for developing suitable extraction techniques for more efficient extraction of available active matters from the plant materials. The main objective of the present study is to determine the suitable method for the extraction of dye from Asoka wood.

Cellulases are one of the upcoming enzymes of textile industries. The main sources of cellulase are bacteria, fungi, plant and animal. Cellulases are used for degumming, oil extraction and also for natural dyeing.

Hence the present study **“Enzyme assisted natural dye extraction from Saraca Asoka wood and its application on silk fabric”** aims with the following objectives.

- To extract dye from Saraca Asoka wood using cellulase enzyme.
- To optimize different parameters for dye extraction.
- To evaluate the dyed fabric for its physical and mechanical properties.

METHODOLOGY ADOPTED

- Dyes from Asoka wood were extracted by two different methods conventional and enzyme assisted extraction
- **Optimization of dye extraction**
Various parameters such as solvent, dye concentration, pH and temperature were optimized for the extraction of dye
- **Dyeing**
Dyeing parameters such as time, dye concentration, mordants, mordanting Techniques were optimized for effective dyeing of the fabric
The dyed fabrics were evaluated both subjectively and objectively for Fabric Weight, Fabric Thickness, Fabric Strength, Fabric Elongation, Fabric Stiffness, Crease Recovery, Sinking Test, Drop Test, Drapability Test, Color Fastness to Sunlight, Crocking and Washing.

FINDINGS

- Water was found to be the suitable solvent for dye extraction
- Asoka wood powder at a concentration of 7% and 9% per cent was found to be the optimum concentration for dye extraction by conventional and enzyme assisted extraction method respectively.
- A contact time of 90 mins for conventional and 150 mins for enzyme assisted extraction resulted in maximum color yield.
- The color intensity of the dye extract was found to be maximum at 70°C and 100°C for enzyme assisted extraction and conventional extraction respectively.
- The optimum pH for dye extraction was found to be 9 and 10 for enzyme assisted extraction and conventional extraction respectively.
- For extraction the mass liquor ratio selected was 1:20
- Mordant selected was pomegranate rind. Premordanting, technique was selected for the present study.

- Dyeing time of 150 minutes at 90°C resulted in maximum dye absorption by the fabric
- The results of visual evaluation of dyed fabrics revealed that all the dyed fabrics were rated excellent in appearance, brighter in shade and evenly dyed irrespective of the methods
- Fabric thickness was found to be maximum for the fabric dyed with enzyme assisted dye extract
- The increase in fabric stiffness was maximum in EDS sample in both warp and weft directions
- Fabric strength was found to be maximum in EDS sample followed by CDS fabric in both warp and weft directions
- Decrease in fabric elongation was noticed in EDS sample in both warp and weft directions
- Increase in fabric weight was recorded in EDS sample when compared with CDS sample
- EDS sample exhibited excellent color fastness to sunlight, washing and crocking followed by CDS sample
- The silk fabric dyed by enzyme assisted extract was found to exhibit lower absorbency than CDS samples.

CONCLUSION

Natural dye extracts from medicinally rich herbs, plants, flowers, fruits, stems, barks and minerals make the whole production cycle devoid of any chemical use with almost no carbon footprint. Natural dyes are not only biodegradable, but also have medicinal properties like anti – bacterial, anti –inflammatory and anti-allergic which are beneficial for the skin of the person using it. It ensures that the discharge of the dyeing unit is devoid of any toxic chemicals and heavy metals protecting the precious water sources.

In this study, it was observed that silk fabrics dyed with dried *Saraca Asoka* have good properties. The degree of dye uptake was a function of the concentration of the dye in the fabric. The ratings for washing and rubbing fastness of the samples dyed by both conventional and enzymatic assisted method were good and very good. Therefore, it can be concluded that enzyme assisted dye extraction is a very

interesting alternative to conventional extraction. Enzyme are biodegradable, can be reused and effective at lower temperature. These factors make them extremely interesting from the point of view of an industry that strives to reduce its environmental impact.

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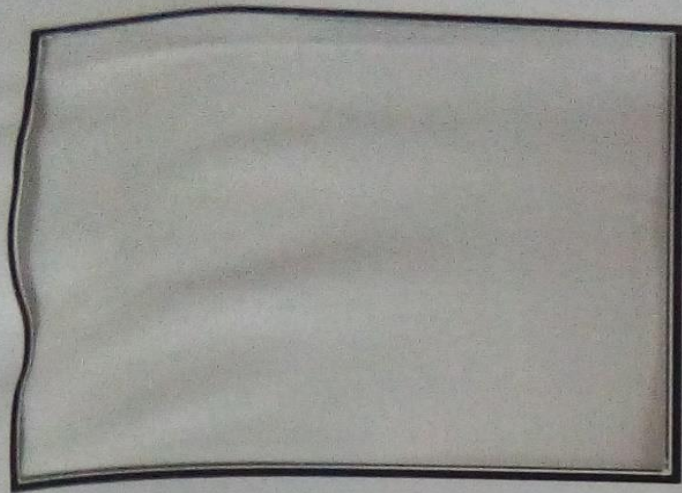
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APPENDIX - 1
Fabric Samples



Original Silk Fabric



**Silk Fabric Dyed with
Conventional Dye Extract**



**Silk Fabric Dyed with Enzyme
Assisted Dye Extract**

