Calliandra Haematocephala- A Natural Flower Colourant for Dyeing Khadisilk

ANTARA VINODH

(16PTF001)

A Thesis submitted to the

Avinashilingam Institute for Home Science and Higher Education for Women

Coimbatore-641 043

In partial fulfilment of the requirement for the degree of

MASTER OF SCIENCE IN TEXTILE AND FASHION APPAREL

APRIL, 2018

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Certified as Bonafide Research Work

Signature of the
Head of the Department

Signature of the
Supervisor

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INTRODUCTION

I. INTRODUCTION

A wide range of natural products are found in India and it is one of the 17 mega diverse countries. Almost all parts of the plants like root, bark, leaf, fruit, wood, seed, flowers, etc. produce these colourful dyes. India is well known as the country of colours as it delivers the wealth of beautiful floral resources. Floral dye sources are very important for dyeing textile materials as it provides both dye as well as fragrance. Dye pigments of flower provides colour to the textile while the fragrance of the floral dye will help in retaining the freshness of the textile material. These keep the dyes body odour away from the garment for a long period of time. Colour of the floral pigment is due to chromophores present in the flower to display the hue of the colour. Many natural dyestuff and stains are been obtained mainly from plants and dominated as sources of natural dyes, producing different colours like red, yellow, blue, black, brown and a combination of these (Singh and Srivastava, 2015).

The Textile industry is one of the oldest industries in India. The sector has made significant contributions in terms of forex earnings, employment and is one of the major contribution to the economy. Indian Textile Industry occupies a very important place in the economic life of India. The Indian textile industry is one of the largest in the world with a massive raw material and textiles manufacturing base. Our economy is largely dependent on the textile manufacturing and trade in addition to other major industries. About 27% of the foreign exchange earnings are on account of export of textiles and clothing alone. The textiles and clothing sector contributes about 14% to the industrial production and 3% to the gross domestic product of the country. Around 8% of the total excise revenue collection is contributed by the textile industry. Regarding all this, the textile industry accounts for as large as 21% of the total employment generated in the economy. Around 35 million people are directly employed in the textile manufacturing activities. Indirect employment including the manpower engaged in agricultural based raw-material production like cotton and related trade and handling could be stated to be around another 60 million (Divatia, 2003).

The use of synthetic dye exponentially has been increased in many important industries, such as textile, pharmaceutical and food processing. The synthetic dyes are easily available and they show superior fastness properties over natural dyes. But, though synthetic dyes exhibit superior fastness properties, it produces many side effects on human body causing allergic reaction. Synthetic dyes are not easily degradable and bio-accumulated in natural environment. It has been estimated that, nearly 10, 00,000 tons of synthetic dyes were used per annum. The synthetic dye may cause pollution, skin diseases, health hazards to human and other important organisms. Thus the use of ecofriendly and biodegradable dyes has the main concern in the whole world (Patil, Gaikwad *et al*., 2016).

Regarding all over the world, growing consciousness about organic value of eco-friendly products has generated renewed interest of consumers towards use of textiles (preferably natural fibre product) dyed with eco-friendly natural dyes. Natural dyes are known for their use in colouring of food substrate, leather as well as natural fibres like wool, silk and cotton as major areas of application since pre-historic times. Although this ancient art of dyeing textiles with natural dyes withstood the ravages of time, but due to the wide availability of synthetic dyes at an economical price, a rapid decline in natural dyeing is been continued. Whereas, even after a century, the uses of natural dyes never erode completely and they are being still used in different places of the world. Thus, natural dyeing of different textiles and leathers has been continued mainly in the decentralized sector for specialty products besides the use of synthetic dyes in the large scale sector for general textiles or apparels (Samanta and Konar, 2016).

Natural dyes which are obtained from different sources such as plants, animals, and minerals, are renewable and sustainable bio-resource products with minimum environmental impact and known since antiquity for their use, not only in colouration of textiles but also as food ingredients and cosmetics. With the discovery of synthetic dyes, the use of natural colourants declined sharply, whereas, the application of number of synthetic dyes has detrimental effects on environment and associated allergic, toxic, carcinogenic, harmful responses. In spite of growing environmental and health concerns eco-friendly nontoxic natural dyes re-emerged as a potential viable ‘Green chemistry’ option as an alternative/co-partner to some extent to synthetic dyes. Recent resurgence in research and development on natural dye production and application is observed due to increasing popularity of more natural lifestyle based on naturally sustainable goods. Natural flora or fauna is full of exquisite colors, fascinating and attracting human being towards a vast portfolio of possibilities. A large number of plant and animal / insect sources have been identified for extraction of color and their diversified use in textile dyeing and other disciplines (Kumar Jha, Kumar *et al*., 2015).

At present, ecological considerations are becoming an important factor in the selection of consumer goods all over the world. Natural dyes are known for their use in the selection of food, substrata, leather as well as natural protein fibres like wool, silk and cotton as the major areas of application since prehistoric time. Dyeing is an antiquated art as well as modern science complex. Now a days, there is an excessive use of synthetic dyes estimated at around 10,000,000 tons per annum, the production and application of which the release of vast amount of waste and unfixed colorants causing serious health hazards and disturbing eco-balance of nature. Natural dyes are the colours derived from plants and animals or insect matter without any chemical processing. They can offer not only a rich and varied source of dye stuff, but also the possibility of an income through sustainable sale of these dye plants (Ghurde, Padwad *et al*., 2014).

Environmentalists are always worried about the rampant use of synthetic dyes in textile industry as they cause water pollution and waste disposal problems. Natural dyes are biodegradable and do not cause any health hazards and hence they can be easily used without much environment concerns. Despite this, use of natural dyes for dyeing textiles has been restricted mainly to artisan or craftsman, small scale or cottage level dyers and printers as well as to small scale exporters and producers dealing with high-valued eco-friendly textile production and sales. Over the past few years, many commercial dyers have started using natural dyes to overcome the environmental damage caused by synthetic dyes. Also, synthetic dyes such as azo dyes are reported to be carcinogenic and can cause allergic reactions. Germany was the first to take initiative to put ban on production and use of numerous specific azo dyes. Netherlands, India and some other countries also followed the ban.

Silk has been known as the "Queen of fibers" since discovery. Clothes made from silk are luxurious and have many excellent qualities including the luster, light weight, superior mechanical performance, fine, smooth texture, excellent moisture transportation, and excellent draping quality. The production of silk fabric was a great invention in the history of human civilization. However, silk is one of the natural fibers most sensitive to environmental degradation factors that cause deterioration of its intrinsic properties, becoming fragile and therefore difficult to preserve (Nilson, 2014).

Natural dyes are well known for producing very uncommon, soothing and soft shades as compared to synthetic dyes. This shift in paradigm in favour of natural dyes is also attributed to the stringent environmental standards imposed by many countries in response to toxic and allergic reactions associated with synthetic dyes. Natural dyes exhibit several important properties that provide them a significant edge over synthetic dyes (Arora, Agarwal *et al*., 2017). As a result, natural dyes are among the promising options for developing a greener textile process and such interest is reflected to an increased number of recent publications.

Since a lot of people have realized not only in the textile industry but also the textile consumers of the need to develop and demand of dyeing textiles with natural dyeing methods offer an important alternative in this regard as these are safer in use with minimum health hazards, have easy and loss disposal problems of effluent. Bearing this concept, the investigator has taken up the study on "**Calliandra Haematocephala - A natural flower colourant for dyeing Khadi silk"** with the following objectives;

- To extract dye from the selected natural source *Calliandra Haematocephala* and parameters.
- To dye and evaluate the performance of the dyed khadi silk fabric.
- To reuse the dye solution and measure the colour strength.

REVIEW OF LITERATURE

II. REVIEW OF LITERATURE

The review of literature pertaining to the study on **"*Calliandra Haematocephala*- A Natural Flower Colourant for Dyeing Khadisilk"** is discussed under the following headings

2.1 Natural Dye

2.1.1 History of Natural Dyes

2.1.2 Advantages and Disadvantages of Natural Dyes

2.1.3 Classification of Natural Dyes

2.1.3.1 Plant Dyes

2.1.3.2 Blue Dyes

2.1.3.3 Morinda

2.1.3.4 Safflower

2.1.3.5 Yellow Dyes

2.1.4 Powder Puff Flower

2.1.4.1Botanical Name

2.1.4.2 Uses

2.1.4.3 Properties

2.2 Silk

2.2.1 History of Silk

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2.2.3.1 History of Khadi

2.2.3.2 Introduction

2.2.3.3 Meaning and Definition

2.2.4 Properties of silk

2.2.5 Natural Colourants for dyeing Silk Fabric

2.2.6 Dyeing of Silk

2.3 Colour Strength

2.1. NATURAL DYEING

Natural dyes are dyes or colourants derived from plants, invertebrates or minerals. The majority of natural dyes are vegetable dyes from plant sources - roots, berries, bark, leaves and wood and other biological sources such as fungi and lichens. Colour has always played an important role in the formation of different cultures of human being all over the world. Natural dyes are also used in clothing, as well as in cosmetic industry (Henna, Catechu), pharmaceutical industry (Saffron, Rhubarb) and in food industry (Annatto, Curcumin and Cochineal) during last few decades, ecological concerns related to the use of most of the synthetic dyes, motivated R&D scholars all over the globe to explore new eco-friendly substitutes for minimizing their negative environmental impacts, and various aspects of bio-colorant applications. Colours in the "ruddy" range of reds, browns, and oranges are the first tested colours in a number of ancient textile sites ranging from the Neolithic to the Bronze Age across Egypt, Mesopotamia and Europe, followed by evidence of blues and then yellows, with green appearing somewhat later (Bechtold, 2009).

Dyeing is an ancient art, which predates written records. It was practiced during the Bronze Age in Europe. Primitive dyeing techniques included sticking plants to fabric or rubbing crushed pigments into cloth. The methods became more sophisticated over time and techniques were developed using natural dyes from crushed fruits, berries and other plants, which were boiled into the fabric and gave light and water fastness (Jothi, 2008). A plant usually contains a mixture of natural dyes. This mixture is often extracted and used to dye textiles. However, a particular dye may be extracted from the mixture and used (Extracting and testing a natural plant dye, 2008).

2.1.1 HISTORY OF NATURAL DYES:

The earliest written record of the use of natural dyes was found in China dated 2600 BC. Chemical test of red fabrics found in the tomb of King Tutankhamen in Egypt showed the presence of alizarin, a pigment extracted from madder. Tyrianpurple, a well renowned natural dye, occupied a prominent position in Roman history (Schetkly, Ethel Jane Mc.D., 1986).

The ability of natural dyes to colour textiles has been known since ancient times. The earliest written record of the use of natural dyes was found in China dated 2600BC. Tyrian purple, a well renowned natural dye, occupied a prominent position in Roman history (Driessen, 2003).In China, dyeing with plants, barks and insects has been traced back more than 5,000 years. The essential process of dyeing changed little over time. Typically, the dye material is put in a pot of water and then the textiles to be dyed are added to the pot, which is heated and stirred until the colour is transferred (Kakhia, 2016).

Natural dyes were used only for colouring of textiles from ancient times till the nineteenth century. As the name suggests, natural dyes are derived from natural resources. Colouring materials obtained from natural resources of plant, animal, mineral, and microbial origins were used for colouration of various textile materials. Different regions of the world had their own natural dyeing traditions utilizing the natural resources available in that region. Use of natural dyes started to decline after the invention of synthetic dyes in the second half of the nineteenth century (Raja, 2014).

2.1.2 ADVANTAGES AND DISADVANTAGES OF NATURAL DYES:

Natural dyes are not harmful to the environment, which makes it so appealing for consumers. Natural dyes are biodegradable and disposal of these dyes do not cause pollution. Renewable – Natural dyes are obtained from renewable sources that can be harnessed without imposing harm to the environment. Natural dyes produce a soothing shade and safer to use.(http://www.keycolour.net/blog/advantages-disadvantages-natural-dyes/).

Natural dyes are considered to be eco-friendly as these are obtained from renewable resources as compared to synthetic dyes which are derived from non-renewable petroleum resources. These are biodegradable and the residual vegetal matter left after extraction of dyes can be easily composted and used as fertilizer. They produce soft colors soothing to the eye which are in harmony with nature. Natural dyes are considered to be an eco-friendly alternative for dyeing of textile materials, especially natural fiber textiles. (Saxena, 2014).

Larger amount of natural dyes may be needed in order to dye a specific amount of fabric as opposed to synthetic dyes. For instance, one pound of cotton may be dyed with just five grams of synthetic dye, whereas 230 grams of natural dye are needed to dye the same amount of material. Therefore, using natural dyes is more expensive than synthetic dyes. More so, quality may not be as consistent as what synthetic dyes can deliver. Availability is another issue with natural dyes is their availability. It can be difficult to produce because the availability of raw materials can vary from season to season, place, and species, whereas synthetic dyes can be produced in laboratories all year round. Natural dyes can also be harmful to some extent. Logwood has ingredients, hematein and hematoxylin, that can be have harmful effects when inhaled, ingested, or absorbed through the skin. Bloodroot, another natural dye source, can cause irritation and inflammation when inhaled.(http://www.keycolour.net/blog/advantages-disadvantages-natural-dyes/).

2.1.3 Classification of Natural dyes:

2.1.3.1 Plant dye:

Historically, plants have been used for the extraction of a majority of natural dyes. Various plant parts including roots, leaves, twigs, stems, heartwood, bark, wood shavings, flowers, fruits, rinds, hulls, husks, and the like serve as natural dye sources. The famous natural blue dye, indigo is obtained from the leaves of the plant indigofera tinctoria. Some plant-derived dyes have other applications also, for example, as food ingredients and medicines in traditional medicine systems supply chain of these dyes is in place. Some of the natural dyes were well known in the past for their dyeing properties and have remained in use even now. A renewed interest in natural dyes has increased their commercial availability.

2.1.3.2 Blue dyes:

Indigo is the only important natural blue dye. Leaves of the plant indigofera tinctoria are the best source of this dye. This very important dye popularly known as the ‘‘king of natural dyes’’ has been used from ancient times till now for producing blue color and is today most popular for denim fabrics. The colouring matter is present in indigo plant leaves as a light yellow substance called indicant Natural Dyes: Sources, Chemistry, Application and Sustainability Issues. The leaf production from one acre of cultivated indigo plants is approximately 5,000 kg which can yield about 50 kg of pure natural indigo powder after processing. It is produced by fermenting the fresh plant leaves, and cakes thus prepared are used for dyeing purposes.

2.1.3.3 Morinda:

The root and bark of the tree Morinda growing in India and Sri Lanka is used for getting red shades. Maximum colouring matter can be obtained from the 3 to 4-year-old tree. Mature trees have very little dye. Dye is extracted from the chipped material with water after a preliminary wash to remove free acids. Various shades including purple and chocolate can be produced with the use of mordants.

2.1.3.4 Safflower:

Safflower is an annual herb known to have originated in Afghanistan. It is mainly cultivated for oil from its seeds which are rich in polyunsaturated fatty acids. The safflower florets were traditionally used for extracting dye which was valued for its bright cherry-red colour. It contains two colouring matters, a water-soluble yellow present in abundance (26–36 %) which was not used as a dye and the scarlet red water-insoluble carthamin present only to the extent of 0.3–0.6 %. The yellow amorphous colouring matter has to be completely removed from carthamin before the latter is used for dyeing as its presence even in small quantities affects the pure pinkish shade imparted by the red dye. Safflower has been employed to give cherry-red direct dyeings on silk and cotton. The dye is extracted from dried safflower florets by continuously washing it with acidulated water to remove all the water-soluble yellow colouring matter.

2.1.3.5 Yellow dyes:

Turmeric:

Turmeric is a well-known natural dye. The dye is extracted from the fresh or dried rhizomes of turmeric. The dye present is chemically curcumin belonging to the Diarylmethane class. It is a substantive dye capable of directly dyeing silk, wool, and cotton. The shade produced is fast to washing but its fastness to light is poor. The natural mordants such as tannin obtained from myrobolan can be used to improve the fastness properties. Turmeric dyeings can be over dyed with indigo for production of fast greens.

Saffron:

Saffron is an ancient yellow dye belonging to the family Iridaceae and is obtained from the dried stigmas of the plant Crocus sativus. It is grown in the Mediterranean, Iran, and India, and used for cooking as well as medicinal purposes. The dye is extracted from the stigmas of flowers by boiling them in water. It imparts a bright yellow colour to the materials. It can directly dye wool, silk, and cotton. Alum mordant produces an orange yellow known as saffron yellow.

Pomegranate:

Rinds of pomegranate (Punica granatum) fruits are rich in tannins and are used for mordanting purposes. A yellow dye is also present which can be used to dye wool, silk, and cotton with good fastness properties. It is also used along with turmeric for improving the light fastness of the dyed materials (Kumar Samantha, 2016).

Myrobalan:

Dried myrobolan (Terminalia chebula) fruits have high tannin content and also contain a natural dye that is used for producing bright yellow shades for all textile materials. Myrobolan is also used as a natural mordant to fix different natural dyes on textile materials. Myrobolan is a part of the famous Ayurvedic preparation ‘‘triphala’’ and dyed materials are also imparted with medicinal properties such as antimicrobial, antifungal, and so on. (Saxena, 2014).

2.1.4 POWDER PUFF FLOWER:

2.1.4.1 Botanical Name: Calliandra Haematocephala

2.1.4.2 Uses:

This plant is traditionally used as anti-oxidant and as blood purifier (Moharran, 2006). This small to medium sized naturally evergreen tropical shrub makes a wonderful container plant and may also be used for a continuous flowering Bonsai specimen. The flowers are produced in cylindrical or globose inflorescences and have numerous long slender stamens which give rise to the common names powder-puff, powder puff plant, and fairy duster. These plants flower all year round, but the best blooming is in spring and summer. They can be easily pruned. Calliandra is often fed on by caterpillars, such as the larvae of statira sulphur (Aphrissa statira).(https://en.wikipedia.org/wiki/Calliandra).

2.1.4.3 Properties:

The red powder puff flowers are attractive to butterflies and hummingbirds but only appear during November-April (Kiritikar and Basu, 1999). The presence of antibacterial and insecticidal properties (Parekh and Chanda, 2007) in leaves and seeds from former researches has drawn attention in investigating anthelmintic activities in flowers as no earlier researches are reported on its flowers part (Tiwari, 2016).

2.2 SILK

2.2.1 HISTORY OF SILK:

The Chinese kept the secret of the silk-thread production for ages selling the products at a very high price. The silkworm (bombix mori) is an useful insect that has learned not only to leave the man in the course of ages, but also to adapt itself to the man' changeable demands. Probably it is the unique example of insect completely domesticated. It's difficult to establish the right age of the beginning of the silkworm-breeding: it is almost sure it began in China from 2500 to 3000 B.C., but some people attribute its origin to India in 600 B.C. Only towards 1000 or 1100 A.C. silk spread over the Mediterranean, consequently in Sicily by the Arabs and the Norman Prince Ruggero II, reaching splendours times. As regards Italy, silk manufacture extended from Sicily to other provinces; but from the 1800 the breeding interested more the

Center-North ones. From the agricultural breeding activity and cocoon production, other connected activities developed: the silkworm's egg production, the silk reeling, throwing, weaving dyeing and lastly the fabric's printing and finishing. Today, the silk worm breeding and the silk reeling have given up in Italy. Silk as raw material is imported from China and South America. Instead the weaving, dyeing and printing works are still active and represent a fundamental landmark all over the word (Masciadri, 2002).

The art of dyeing is as old as our civilization. Dyed textile remnants found during archaeological excavations at different places all over the world provide evidence to the practice of dyeing in ancient civilizations. Natural dyes were used only for coloring of textiles from ancient times till the nineteenth century. As the name suggests, natural dyes are derived from natural resources. Colouring materials obtained from natural resources of plant, animal, mineral, and microbial origins were used for coloration of various textile materials. Different regions of the world had their own natural dyeing traditions utilizing the natural resources available in that region (Raja, 2014).

India harbours a wealth of useful germ plasm resources and there is no doubt that the plant kingdom is a treasure-house of diverse natural products. One such product from nature is the dyes. Dyes are one of the most important uses of the plants, as they are related with cultural practices, rituals, arts and crafts, fabrics and to satisfy personal embodiment (Patni, 2011).

2.2.2 TYPES OF SILK:

Raw silk is of two types namely,

1. Mulberry
2. Non Mulberry

The destruction arises from the rearing of silk worms either upon mulberry leaves or on other plants. Mulberry silk is produced in Karnataka, West Bengal, Jammu and Kashmir Tamil Nadu, and Andhra Pradesh. Non Mulberry silk is produced in Assam, Bihar, Madhya Pradesh, Orissa, Manipur and West Bengal.

2.2.2.1 Tsar Silk

It is the silk reeled from cocoons of silk worms belonging to saturnidae family which are feeding on leaves of oak, asan and arjan trees.

2.2.2.2 Eri Silk:

It is spun from cocoons of silk worms belonging to saturnidae family, which are feeding on castor leaves.

2.2.2.3 MugaSilk:

It is silk produced only in Assam from cocoons of silk worms belonging to saturnidae family which are feeding on san and soalu leaves. It has a rich golden colour. (http://www.iiem.com/em/silks/silk%20exports.pdf).

2.2.2.4 Fagara silk:

Fagara silk is obtained from the giant silk moth Attacus atlas L. and a few other related species or races inhabiting the Indo-Australian bio-geographic region, China and Sudan. They spin light-brown cocoons nearly 6 cm long with peduncles of varying lengths (2-10 cm).

2.2.2.5 Coan silk:

The larvae of *Pachypasa atus* D., from the Mediterranean bio-geographic region (southern Italy, Greece, Romania, Turkey, etc.), feed primarily on trees such as pine, ash cypress, juniper and oak.

2.2.2.6 Spider silk:

Spider silk – another non-insect variety – is soft and fine, but also strong and elastic. The commercial production of this silk comes from certain Madagascan species, including *Nephila madagascarensis, Miranda aurentia* and *Epeira* (http://inserco.org/en/types _ of_silk).

2.2.3 Khadi silk:

2.2.3.1 History of khadi:

Khadi was conceived in 1920’s as a symbol of Swadeshi movement and self-reliance of the villages. The Gandhian ideas of simplicity, Swadeshi spirit and decentralization are embodied in Khadi. It was a practical attempt to relieve the poverty and uplift the standards of

Indian village people. In 1919, when there was a movement to boycott foreign goods, Gandhiji appealed to his countrymen to adopt Khadi. At the same time of Nagpur session (1920) the Indian National Congress decided to encourage Khadi (Sekhri, 1992).

The All India Spinner's Association, also known as Akhil Bhartiya Charkha Sangh, was formed in 1925. Though this body was closely associated with the Indian National Congress, it was completely autonomous in its working. Till 1935 the Association concentrated its activities on propagation, production and sale of Khadi. The main objective of this association was that Khadi could have "a permanent effect only when carried out as a part and parcel of the wider program of non-violent village uplift or village reconstruction" (Nanda, 1935).

Khadi, the Hand-spun and Hand-Woven cloth, whether of cotton, silk or wool, with which we have become familiar, has seen many ups and downs. Since time immemorial, it has been the prime and universal cottage industry of this ancient land. During its long march over the centuries, it has experienced many vicissitudes, which have only strengthened it and made it a vital fulcrum around which the village community life has been built up. From simple beginnings, it reached the highest water mark of achievement and renown, then for a couple of centuries, went down, and has come up again with a new vigour and a new vitality claiming an honoured place in the economic life of the nation (http://shodhganga.inflibnet.ac.in/bitstream/10603/43272/8/08_chapter%201.pdf).

2.2.3.2 Introduction:

Gandhiji started his movement for khadi in 1918. His emphasis at first was on khadi as providing relief to our poverty- stricken masses. But one finds a change in his emphasis from 1934, more especially from 1935, when he began on insisting on khadi for the villager's own use, rather than merely for sale to others. His imprisonment in 1942 and 1943 gave him time to ponder further over his khadi movement, and when he came out of jail he came with a determination to give a new turn to khadi work in order to make khadi serve the needs of villagers themselves first and foremost. He poured out his soul to his fellow-workers in 1944, and urged them to effect the change (Joshi, 2002).

Khadi first caught the imagination of the nation during the freedom movement under Mahatma Gandhi, who propagated it as not just a fabric, but a way of life. One that is centred in the village, where the practice of khadi would be able to generate employment, income and, hence, self-reliance. Khadi was meant to become a supplementary industry to agriculture, a crucial element in a self-sustaining economy (Banerjee, 2011).The idea of reviving of traditional industries generates from the significant role they are playing in promoting employment, particularly to the poor (Prabin Baishya., 1989).

2.2.3.3 Meaning and Definition:

"Khadi can and should have only one meaning, i.e hand- woven cloth made from hand-spun thread. Silk-thread, just fiber and wool woven in this manner may be called, if we like, silk, jute and woolen Khadi, respectively. Khadi is a hand spun and hand woven cloth made in India from cotton, silk and woolen yarn. Started with the spinning in Takli, 2 spindle new model charkha, 4 spindle, 6 spindle, 8 spindle, 10 spindle and 12 spindle new model charkhas have been introduced in spinning of Khadi yarn. Likewise a number of handlooms including Wardha loom, Gram Laxmi loom, and Pit loom are in operation in weaving Khadi cloth (Desai, 2003).

Khadi is the central core of the constructive activities as recommended by him. According to him there could be no swaraj without universal and voluntary acceptance of khadi. In his words, "I am a salesman of swaraj. I am a devotee of khadi. It is my duty to induce people, by every honest means, to wear khadi (Joshi, 2002).

2.2.4 Properties of silk:

- **Composition:** The silk fibre is chiefly composed of 80% of fibroin, which is protein in nature and 20% of sericin, which is otherwise called as silk gum.
- **Strength:** Silk as a fibre, has good tensile strength, which allows it to withstand great pulling pressure. Silk is the strongest natural fibre and has moderate abrasion resistance. The strength of the thrown yarns is mainly due to the continuous length of the fibre. Spun silk yarn though strong is weaker than thrown silk filament yarns.
- **Elasticity:** Silk fibre is an elastic fibre and may be stretched from 1/7 to 1/5 of its original length before breaking. It tends to return to its original size but gradually loses little of its elasticity. This would mean that the fabric would be less sagging and less binding resulting in the wearers comfort.

- **Drapability:** Silk has a liability and suppleness that, aided by its elasticity and resilience, gives it excellent drapability.
- **Heat Conductivity:** Silk is a protein fibre and is a non-conductor of heat similar to that of wool. This makes silk suitable for winter apparel.
- **Absorbency:** Silk fabrics being protein in nature have good absorbency. The absorptive capacity of the silk fabric makes comfortable apparel even for warmer atmosphere. Fabrics made from silk are comfortable in the summer and warm in the winter.
- **Cleanliness and Washability:** Silk fabric does not attract dirt because of its smooth surface. The dirt, which gathers can be easily removed by washing or dry cleaning. It is often recommended for the silk garments to be dry-cleaned. Silk fabrics should always be washed with a mild soap and strong agitation in washing machine should be avoided.
- **Shrinkage:** Silk fabrics are subjected only to normal shrinkage which can be restored by ironing. Crepe effect fabrics shrink considerably in washing, but careful ironing with a moderately hot iron will restore the fabric to its original size.
- **Effect of Heat:** Silk is sensitive to heat and begins to decompose at 330° F (165° C). The silk fabrics thus have to be ironed when damp.
- **Effect of Light:** Silk fabric weakens on exposure to sun light. Raw silks are more resistant to light than degummed silk.
- **Resistance to Insects:** Silk may be attacked by the larvae or clothe moths or carpet beetles (Islam Kiron, Textile learner one stop solution for textiles).
- **Physical structure:** Physical structure of the A. atlas fibers was determined in terms of % crystallinity and peak positions using a Rigaku D-max/BΘ/2Θ X-Ray diffractometer (Rigaku Americas, Woodlands, TX) with Bragg–Brentano parafocusing geometry, a diffracted beam monochromator, and a copper target X-ray tube set to 40 kV and 30 mA. Fibers were powdered and compressed to form pellets for the X-ray measurements.
- **Tensile properties:** Samples were conditioned for at least 24 hours under the standard testing conditions of 21 °C and 65 % relative humidity before testing. Fineness of the fibres was calculated in terms of denier (weight in grams per 9,000 m of the fibres) by weighing a known length of the fibres (Reddy, 2013).

2.2.5 Natural colourants for dyeing silk fabric:

Natural dyes are found from natural sources such as from plants, animals, insects or minerals. Although the ancient art of dyeing with natural dyes withstood the ravages of time, a rapid decline in natural dyeing continued due to the wide availability of synthetic dyes at an economical price, but now-a-days the importance of natural dyes is more relevant worldwide in the context of increasing environment consciousness. The use of natural organic dyes obtained from renewable resources such as plants and trees has the potential for not only preserving the precious petrochemicals but also the all-endangered environment for our coming generations (Jahan, 2015).

Local plants are one source of natural colourant which is grouped under plant sources. They are easily available in the country and can be considered as zero cost dyes as they are planted for other purposes. Plants are the major sources of natural colourants and almost all their parts such as stem, leave, fruits, seeds and pills are used for extracting natural colour and they have antimicrobial, antifungal, insect repellent, deodorant, disinfectant and other medicinal values (Jihad, 2014).

The natural dyes present in plants and animals are pigmentry molecules, which impart colour to the materials. These molecules containing aromatic ring structure coupled with a side chain are usually required for resonance and thus to impart colour. There is a correlation of chemical structure with colour, chromogen-chromophore with auxochrome (Purrohit, 2011).

2.2.6 Dyeing of silk:

The dyeing of silk with natural dye was carried out in three stages; Extraction of dyes from plant parts, Mordanting (fixing dye with fabric) and Dyeing. Different plant parts (used as source of dye) were crushed and dissolved in distilled water and allowed to boil in a beaker kept overwater bath for quick extraction for 2 hours. All the colour was extracted from flowers, roots and leaf by the end of 2 hours. The solution was filtered for immediate use (Jihad, 2014). The silk samples were dyed with dye extract keeping M : L ratio as 1:30. Dyeing was carried out at 100°C and continued for 1 hour (Chandramohan, 2011).

The obtained colour of silk samples found deeper than cotton samples and mordanted silk fabrics with alum show greater colour fastness to wash than cotton fabrics(alum and copper sulphate mordanted) show. Other tested dyeing properties of silk fabrics observed better than those in cotton fabrics. Finally the test concluded with the result that silk fabrics can be made more sophisticated with strong versatile shades of Powder puff flower (Datta, 2015).

Silk has always been the symbol of royalty due to its lustrous appearance and peach like softness. The coloration of this royal fiber is also an art form. The process varies largely in the form of hanks and woven pieces. There are numerous ranges of dyestuff available for the use of silk dyeing. Almost every class of dyestuff used for cotton or wool can be used for dyeing silk. In general the dyestuffs are applied by techniques similar to those of wool or cotton (Uddin, 2010).

2.3 Colour strength:

Colour characteristics of all the dyed samples (without mordanting, premordanting, postmordanting and simultaneous mordanting) were measured by using a spectrophotometer Datacolor 600 (Jahan, 2015). Colour fastness to washing of the dyed fabric samples was determined as per ISO: 105-A02 – 1995 method using a launder-O-meter following ISO-3 wash fastness method. The wash fastness rating was assessed using grey scale as per ISO-105-A02 (loss of shade depth) and ISO-105-AO3 extent of staining. Colour fastness to rubbing (dry and wet) was assessed manually by hand rubbing one sample ten times and grey scale as per ISO-105-AO3 extent of staining. All the treated samples subjected to washing fastness and all the treated samples shows no colour staining to washing fastness. The colour change to dry and wet rubbing for all the treated samples was excellent and there was slight no colour staining (Jihad, 2014).The spectral reflectances of the dyed samples were measured using a Text Flash Spectrophotometer (Data colour corp.) (Saravanan, 2011).

EXPERIMENTAL PROCEDURE

III. METHODOLOGY

The methodology pertaining to the study on **"*Calliandra Haematocephala*- A Natural Flower Colourant for Dyeing Khadisilk"** is discussed under the following headings

3.1 Selection of fabric

3.1.1 Degumming of silk fabric

3.2 Selection of natural dye source

3.2.1 Collection and Processing of Natural Source

3.3 Selection of mordant

3.3.1 Mordanting Techniques

3.4 Pilot study

3.4.1 Optimization of Dye extraction

3.4.1.1 Aqueous Method

3.4.1.2 Alkaline Method

3.4.2 Optimization of dyeing parameters

3.4.2.1 Dyeing Concentration

3.4.2.2 Concentration of Mordant

3.4.2.3 Dyeing Time

3.4.2.4 Mordanting Time

3.4.2.5 Dyeing Temperature

3.4.2.6 Mordanting Temperature

3.5 Optimized parameters for final study

3.5.1 Preparation of Dye and Mordant

3.5.2 Dyeing Methods- Dip and Dry Method

3.6 Final study

3.6.1 Nomenclature of sample

3.7 Evaluation

3.8 Subjective evaluation

3.8.1 Objective Evaluation

3.8.2.1 Physical evaluation

3.8.2.1.1 Fabric thickness

3.8.2.1.2 Fabric weight

3.8.2.2 Mechanical evaluation

3.8.2.2.1 Fabric tensile strength and elongation

3.8.2.3 Comfort

3.8.2.3.1 Fabric stiffness

3.8.2.3.2 Fabric Drape

3.8.2.4 Colour fastness test

3.8.2.4.1 Fastness to sunlight

3.8.2.4.2 Fastness to crocking

3.8.2.4.3 Fastness to wet and dry pressing

3.8.2.4.4 Fastness to washing

3.8.2.5 Wettability & absorbency

3.8.2.5.1 Drop test

3.8.2.5.2 Sinking test

3.8.2.5.3 Capillary rise test

3.9 Reuse of dye bath

3.10 Statistical Analysis

3.1 Selection of fabric:

Khadi is a versatile fabric. It has the unique property of keeping the wearer warm in winter as well as cool in summer season. In khadi silk, the ratio of khadi and silk fabric is 50:50. This fabric requires dry cleaning. It shrinks about 3% after the first wash. It is quite an expensive fabric. Khadi silk provides a royal and rich look. The various types of apparels made from khadi silk are salwarkameez, kurta pajama, saris, dupattas, shirts, vest and jackets. Apparels like kurta, jacket, sari blouses requires lining to be given to ensure its longetivity. (www.fibre2fashion.com). Therefore khadi silk fabric was selected for the present study and procured from from Khadi Bhavan, Saibaba colony.

3.1.1. Degumming of silk fabric

Silk consists of a natural gum called sericin. The sericin present in the silk varies from 20%-30% depending on the variety of silk and type of cocoon that are obtained. It is very essential to degum the raw silk properly. Otherwise a film of sericin will prevent the penetration of dye stuff which may result in improper dyeing. Degumming has to be carried out effectively and hence degumming is called heart of processing (Phulkan, 2003). Therefore selected silk fabric was degummed prior to dyeing.

Degumming was carried out using the recipe as given below:

Silk Material	: 1 meter
Detergent powder	: 50 grams
Water	: 10 litres
Temperature	: 100^0 C
Material and liquor ratio	: 1:20
Time	: 1 hour

Silk fabric of 1 meter is immersed in the bath containing 50 grams of soap powder which dissolved in ten litres of water, was heated up to 100^o C for half an hour with material liquor ratio of 1:20. The fabric is then taken out from the bath and washed thoroughly with cold water later the silk fabric was dried under the shade.

3.2 SELECTIONOF NATURAL DYE SOURCE

Natural dyes are dyes or colourants which are derived from plants, invertebrates or minerals. Most of the natural dyes are vegetable dyes from plant source (NIIR Board 2012). Natural dyes are considered as eco-friendly dyes or mordant dyes as they require the inclusion of one or more metallic salts. Calliandra Haematocephala is selected as the natural dye source. It is available only in red colour. Calliandra Haematocephala (Hassk.), family Fabaceae (Pea or Legume) is an evergreen shrub or small trees widely distributed in the tropics native to tropical America, Bolivia, cultivated in different regions as Malaysia, South Florida, Australia and introduced to Egypt (Williamson, 1990).

Considering the above facts and also based on the availability, easy application, natural dye source namely Calliandra Haematocephala flower was selected for the study (Plate I)

3.2.1 Collection and Processing of Natural Source:

Calliandra Haematocephala is a large, multiple trunked, low-branching, evergreen shrub having silky leaflets which are glossy copper when new, which turns to a dark metallic green. The profuse, fragrant bloom is the main reason for its popularity, with big puffs, two to three inches across, of watermelon pink, deep red, or white silky stamens, produced during warm months (Edward, Gilman et al., 1993). Calliandra Haematocephala is collected by hand picking from the plant in the home garden in and around Coimbatore. Flowers were dried under the shade in room temperature for 3 days. Then the dried flowers were powdered and kept in an air tight container (Plate II)

Plate I

Powder Puff flower

Plate II

Dried Flowers

3.3 SELECTION OF MORDANT

Natural dyes are substantive and require a mordant fix to the fabric and prevent the colour from either fading with exposure to light or washing out. These compounds bind the natural dyes to the fabrics. Mordants are needed to set the colour when using natural dyes. Different mordants will give different hue colour with the same dye. A mordant is thus a chemical agent which allows a reaction to occur between the dye and the fabric (Padama, 2007). If the dyeing process is taking place through the aqueous method, a mordant is required to improve the fastness of the dyed fabric.

The natural mordant Alum (aluminium potassium sulphate) and myrobalan is selected for this study. Alum is most commonly used natural mordant. It does not affect the fabric. The use of too much mordant will make the fabric feel more sticky and harsh. The use of alum is recommended as it softens fabric and helps with even dyeing (www.chemistryexplained.com).

3.3.1 Mordanting Techniques

Mordanting is the first step of the actual dyeing process. It can be carried out in three ways. They are pre-mordanting, post-mordanting and simultaneous mordanting. In the most of the cases simultaneous mordanting is the best for penetration of dye into the fabric, this method increased the depth of the colour. In simultaneous technique mordanting is the second step after which dyeing is done. Post mordanting technique technique was carried out after dyeing (Burrows, 2004). For this study all the three mordanting techniques were tried.

3.4 PILOT STUDY

3.4.1 Optimization of Dye extraction

The method used for extraction was the aqueous extract from fresh flowers. In this method, dye from flowers were extracted an aqueous solution (3 g in 100ml distilled water) and the extraction process was carried out at a temperature range of 90-100°C for half an hour. Colouring materials from the flowers were extracted for dying of the fabric. After the extraction procedure is complete, the flowers were taken out from the liquor and they were taken for extraction of dye for the second time (Grover and Patni, 2011).

The extraction methods of natural dyes basically depends on the medium in which the dye is extracted. There are mainly two methods used in extraction of natural dye.

Plate III

Optimization

3.4.1.1 Aqueous Method

The dye from the selected natural source is extracted in the aqueous medium. Three grams of powdered flowers is dissolved in 100 ml of water and boiled for 30 minutes at 100°C. Then the dye extract was filtered and the extract solution was kept aside for dyeing (Plate III).

3.4.1.2 Alkaline Method

The dye from the selected natural source is extracted in the alkaline medium. Three grams of ground flower powder is dissolved in the prepared solution of alkaline 1% with the addition of 1 g of sodium bicarbonate in 100ml of water. Then the prepared solution is filtered and the extract solution was kept aside for dyeing.

3.4.2 OPTIMIZATION OF DYEING PARAMETERS

The optimization was carried out to determine the optimum values of various parameters such as dye concentration, dyeing time, mordant concentration, mordanting time. In each optimization the value of parameter being optimized were varied, when keeping all parameters constant. In every parameter, the optimum value from the previous experiment was used. The parameters which were constant in all experiment was used. The parameters which were constant in all experiment were:

Material Liquor ratio	: 1:20
Dye Extraction temperature	: 100°C
Mordanting temperature	: 100°C
Dye extraction medium	: Aqueous and alkaline

(I) AQUEOUS METHOD:

Water	: 100ml
Dye extraction temperature	: 100°C
Mordanting temperature	: 100°C
Time	: 30 minutes

(II) ALKALINE METHOD:

Water	: 100ml
Dye extraction temperature	: 100° C
Time	: 30 minutes
Alkali used	: 1 gram of Sodium bicarbonate

3.4.2.1 Dyeing Concentration

The concentration of dye source was found by varying dye concentration such as 1%, 2%, and 3% in 100 ml of water. Dye is prepared in three cups respectively. A known weight of silk fabric was immersed into respective concentration. Optical density of dye absorption is noted and the concentration of dye source was calculated.

3.4.2.2 Concentration of Mordant

The optimum concentration of mordant was determined on the basis of highest percentage of dye absorption. The concentration of alum was kept as 3 percent respectively. Based on absorption, mordant concentration was calculated.

3.4.2.3 Dyeing Time

To optimize the dyeing of silk fabric was dyed individually with optimized percentage of dye for 15min, 30min and 45min in which the liquor ratio was taken as 1:20. The maximum dye absorption is noted and then taken as the optimum dyeing time.

3.4.2.4 Mordanting Time

To optimize the mordanting time silk fabric was mordanted with optimized percentage of mordant and were boiled in 100°C. The optical density was recorded after 15, 30 and 45minutes. Optimum mordanting time for silk material was selected on the basis of highest optical density.

3.4.2.5 Dyeing Temperature

To optimize the dyeing temperature the silk fabric was dyed individually with optimized percentage of dye for 90° C, 100° C and 110° C with the liquor ratio of 1:10. The maximum dye absorption is noted and then taken as the optimum dyeing temperature.

3.4.2.6 Mordanting Temperature

To optimize the mordanting temperature of silk fabric was dyed individually with the optimized percentage of the dye for 90° C, 100° C, and 110° C with the liquor ratio of 1:20. Optimum mordanting temperature for silk material was selected on the basis of highest optical density (Appendix ii)

3.5 OPTIMIZED PARAMETERS FOR FINAL STUDY

TABLE I

Dyeing Parameters, Trial and Optimized Concentration

S.NO	DYEING PARAMETERS	TRIAL CONCENTRATION	OPTIMIZED CONCENTRATION
1	Dye extraction time (minute)	15,30,45	30
2	Dyeing concentration(gram/100ml)	1,2,3	3
3	Concentration of mordant alum (%)	1,2,3	3
4	Mordanting time (minute)	15,30,45	30
5	Dyeing time (minutes)	15,30,45	30
6	Dyeing temperature (degree)	90°,100°,110°	100°C

From the above Table I, the dyeing concentration was obtained maximum at the percentage of 3, the dyeing time was obtained as 30 minutes. The mordanting concentration was obtained maximum at the percentage of 3, and mordanting time was kept as 30 minutes. The overalls dye extraction time was obtained at 30 minutes. Dyeing temperature was optimized as 100°C.

3.5.1 Preparation of Dye and Mordant

The powdered form of the natural source was taken according to the weight of fabric and is added with required amount of water for the dye extraction in two separate beakers, the mordants (Alum and Myrobalan) were added along with the solution in the two separate beakers. After the solution was prepared the beakers were kept in a water bath for 30 minutes at the temperature of 100°C. Then the beakers are taken out from the water bath for 30 minutes, the solutions are obtained, and then the solutions are used for dyeing process.

3.5.2 Dyeing Methods- Dip and Dry Method

The pre-treated silk fabric was dyed with dye extracts along with the mordants Alum and Myrobalan. Keeping M: L ratio as 1: 20. Dyeing was done by the conventional dyeing method. In this method silk fabric was directly immersed on the dye bath slowly and kept in rest position without any disturbance for 30 minutes. After dyeing, the dyed material was taken out and washed with cold water. Dyed silk fabric was dried at room temperature.

3.6. FINAL STUDY

3.6.1 Nomenclature of sample

TABLE II

Nomenclature of sample

S.NO	Fabric details	Nomenclature of the sample
1	Os	Original Silk
2	Ds	Degummed silk
3	Dds	Dyed silk

3.7. EVALUATION

3.8. SUBJECTIVE EVALUATION

The fully dyed samples were evaluated by objective evaluation.

3.8.1 Objective Evaluation

Textile is the process of inspecting, measuring, evaluating characteristics and properties of textile materials. The original samples are dyed with the Calliandra Haematocephala flower along with the natural mordants and the dyed samples were tested using the sample pieces from the same relative portions of the material for their respective laboratory tests.

3.8.2.1 Physical evaluation

3.8.2.1.1. Fabric thickness

3.8.2.1.2. Fabric weight

3.8.2.2 Mechanical evaluation

3.8.2.2.1. Fabric tensile strength and elongation

3.8.2.3. Comfort

3.8.2.3.1. Fabric stiffness

3.8.2.3.2. Fabric Drape

3.8.2.4. Colour fastness test

3.8.2.4.1. Fastness to sunlight

3.8.2.4.2. Fastness to crocking

3.8.2.4.3. Fastness to wet and dry pressing

3.8.2.4.4. Fastness to washing

3.8.2.5. Wettability & absorbency

3.8.2.5.1. Drop test

3.8.2.5.2. Sinking test

3.8.2.5.3. Capillary rise test

3.9. Reuse of dye bath

3.8.2.1. Physical evaluation

3.8.2.1.1 Fabric weight

Fabric weight is the mass per unit area of a length of material that also plays a role in determining the density of a material. The fiber content, yarn size, and fabric count impact the weight of the material (Bubonia, 2014).

A sample of 10 ×25 cm was cut using a GSM cutter. It has sharp blades which cuts through the fabric when pressure is imparted with a slight twist. The weight of the cut sample was found using Electronic Weighting Balance (Plate IV). The inference found was calculated using the following formula

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

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

3.8.2.1.2. Fabric thickness

Fabric thickness is defined as perpendicular distance through the fabric, which determines the dimension between the upper and the lower side of the fabric. It is dependent on the fabric weave as well as the thread's position in the binding repeat (Sirkova, 2012).

Thickness tester (Plate VI) has a broad anvil, upon which a pressure foot is pressed by spring. The detail indicated the thickness of the material in thousands of an inch between the anvil and the pressure foot. Each division of the dial gives a reading of 0.01mm. The sample was then placed on the anvil without tension or creases and the pressure foot was lowered onto the sample for two seconds at 2 kg pressure.

The dial reading was recorded. The readings were calculated similarly and mean value of the readings from the original and dyed silk material was tabulated (Plate V).

3.8.1.2 Mechanical Evaluation

3.8.1.2.1. Fabric strength & Elongation

This study was designed to explore the simplest forms of relationships that exist between fabric behavior under tensile and tear types of stresses and the properties of the component yam elements in the fabric. Tensile and tear stresses were chosen because these are the strength characteristics most commonly used in routine quality assessment. The usefulness of the test for cloth strength is one of assessment of quality and not one of assessment of serviceability. Nevertheless, when a demand for a minimum strength is added to the cloth specification, the manufacturer is restricted to a minimum quality fiber and yarn and to a range of twist factors. Therefore, a fall in strength may yield a valuable warning of a change in the quality of raw material or yarn or of deterioration in the control of finishing processes (Vasanth and Devarakonda, 2002).

Eureka Brand Pendulum Tensile Strength Tester was used to determine the breaking the strength and elongation of the increased samples processed. The capacity of the machine and the rate of transverse curve 90 kg and 40 cm per minute respectively. The sample was cut to a width of 2 inches and 12 inches length. The yarns raveled from both the edges until the the width measured to one inch.

The sample was mounted centrally, gripped along the full width to prevent slippage of the sample. The pendulum of the tester was set vertically and the pointer at zero on the scale. When the load was applied on the sample it ruptured the sample, whereas the mechanism was stopped and the dial reading was recorded in kilogram for breaking strength and elongation in centimeters were been noted. Three readings of the original and dyed samples both in warp and weft direction were recorded.

Plate IV

Electronic Weighing Balance

Plate V

Fabric Thickness Tester

Plate VI **Plate VII**

Fabric Stiffness Tester **Drape meter**

Plate VIII

GSM Cutter

Comfort

3.8.2.3.1. Fabric stiffness

Shirley Stiffness tester was used to test the stiffness of the fabric. Sample A was cut to the size of 15 cm ×2.5 cm using the template. The sample was placed on the platform with the template at the center of fit, so that the leading edges coincide. Both were slowly pushed together forward until the leading edges of the samples and template project beyond the edges 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 slide platform. For each side reading were taken (Plate VI).

3.8.2.3.2. Drapability Test

Drape is the ability of a fabric to hang in soft folds and to fit around a figure, particularly in movement, without creating distorted creases (Aldrich, 2012).

Drapability of the fabric was determined using Eureka Drape Tester. A circular piece of fabric was cut by placing a circular disc and the same was cut in a pattern sheet. A hole was made to the specimen through which it was fitted and the fabric was allowed to hang on its own weight. A small disc was placed on the top of iy and the instrument was closed. A light was switched on. A pattern sheet was placed and the drape of the fabric was drawn by following the shadow of the draped fabric (Plate VII).

Drape co-efficient was determined by considering the following,

The drape co-efficient is given by,

$$F = \frac{A - Ad}{AD - As} \times 100$$

AD- the area of the specimen

Ad- the area of the supporting disc

As-the actual projected area of the specimen

Five readings were taken from the original sample and from the dyed samples, and their mean values were calculated and tabulated (Plate V).

3.8.2.4. Colour fastness test

In natural dyeing colour fastness of the natural dyes requires considerable attention and careful selection of material and processes. The colour fastness quantifies the colour change on a dyed material under specific conditions and also the transfer of dyestuff to uncoloured adjacent material which is also known as bleeding of the dyestuff (Bechtold and Mussak, 2009). Colour fastness test such as colour fastness to sunlight, crocking, pressing and fastness to washing were determined.

3.8.2.4.1. Fastness to sunlight

The test measures the resistance to fading to dyed textiles when exposed to daylight and is very important (Savile, 1999). The dyed samples of 6 cm length and 1 cm width size were taken from each of the dyed materials. The entire samples were divided into eight by making distance of two inches in the larger side marked up to eight. Each sample was covered to prevent the samples from direct sunlight. For the successive seven days the other portion were exposed accordingly along with the first portion. The first portion got seven days exposure to sunlight. The changes in colour of dyed samples were compared with the original and specimens were rated using grey scale.

3.8.2.4.2. Fastness to wet and dry crocking

Sasmira Crock Meter was used for ascertaining the fastness of dyed textiles to wet and dry crocking. It consist of two metal block, while the upper block had an arrangement to move to and from the base by the means of a rotating handle. These were a finger knob attached to the upper movable block to hold the original material with rig. The sample were cut into pieces with the size of 20 cm × 10 cm. the sample was fixed on the case block with longer size in the direction of rubbing. The white material (5 cm×5cm) was fixed on the finger knob of upper movable block with a ring.

The number of rubs was standardized and fixed as ten rubs. The white material was rubbed against the dyed sample along with track of 10 cm with pressure of 900 grams on the finger. The colour transfers from the dyed samples to while material was assessed with gray scale for staining. The colour fastness of each dyed materials to dry and wet crocking was carefully observed and recorded separately.

3.8.2.4.3. Fastness to wet and dry pressing

Test specimens of 5cmx10cm were cut. They were of same size, for dry pressing a hot iron was placed on each of the composite specimen for 5 seconds at a temperature of 350 Fahrenheit. And for wet pressing, the test specimen was wetted and then sandwiched between the white materials and pressed. The colour transfer from the dyed samples was determined using grey scale. The same procedure was repeated for all the samples of dyed materials. Thus the colour fastness of each dyed materials to wet and dry pressing was carefully observed and recorded individually.

3.8.2.4.4. Fastness to washing

Major loss of colour from the fabric is due to washing and results in staining over the adjacent fabric. Test samples of the dyed fabric measuring 5 x 10 cm size were cut. Each of them was sandwiched between the undyed white cloths which was desized well. Specimen was completely soaked in the soap solution about 5g/ 1 for 30 minutes at 400C. After that the sample was removed rinsed in cold water, squeezed well and dried. Evaluation of staining on the white adjacent fabric was found using gray scale. The same procedure was carried out for other dyed sample.

3.8.2.5. Wettability and absorbency test

The wettability and absorbency tests include drop test, sinking test and capillary rise test. Absorbency is one of the several factors that influence textile processing such as fabric preparation, dyeing, and the application of finishes views AATCC (2007). Water and moisture transportation of the fabric is an important feature of any fabric. This factor decided the comfort of fabric and really an important feature of fabric as it transport water from the body surface and makes the fed comfortable, (Sirkova, 2012).

3.8.2.5.1. Drop test

The drop test is a count of number of drops required to penetrate through to the underside of the fabric when all the drops fall on the same spot. In this experiment fabric is clipped on to a glass plate with a piece of filter paper sandwiched between the fabric and the glass. A frame holds the assembly at an angle of 45 degree-directly under the drop-forming device. The latter is prepared from a fine-bore-glass tube to produce a certain number of drops of a given size in a spot; a drought shield of large diameter is used. (Plate VIII). With the specimen in portion, the water supply is started and the drops begin to fall on the fabric. The endpoint is reached when the filter paper shows the sign of water (Jewel,2009).

3.8.2.5.2. Sinking test

A 1000ml beaker was filled with distilled water. Small samples of fabric were cut and were dropped on the surface of the water from a standard height. The stop watch was started when the fabric struck the surface of the water and stopped when the last corner sank below the water surface and the time required for the samples. The mean values were calculated.

3.8.2.5.3. Capillary rise test

The samples were cut into sizes of 15cm length and 10cm width from the original and dyed samples. One end of the sample strip was placed on a glass rod and at the other end 2 gram weight was attached to immerse inside beaker filled with distilled water. The rate of water level in the sample was measured after 60 seconds (Plate X).

3.9. Reuse of dyed bath

An attempt has been made to reuse the dye solution after first dyeing. Therefore the selected sample was dipped in Ixoracoccinea dye solution Instead of disposing the dyed solution it could be used for second dip to get lighter shade for further study. Usually the prepared dye solution has more fixation property. Hence the dyed solution was reused and again used to dye the degummed silk fabric which gives the lighter shade compared to original sample.

3.10 Statistical Analysis

The result of tests was analyzed statistically and ‘F’ values were calculated. The loss/gain percentage of the dyed sample was compared between the degummed sample and the dyed silk fabrics respectively. Significant difference at one percent level was noted.

RESULTS AND DISCUSSION

III. RESULTS AND DISCUSSION

The result pertaining to the study on, **"*Calliandra Haematocephala*- A Natural Flower Colourant for Dyeing Khadisilk"** is presented under the following heading:

4.1 Objective Evaluation

4.1.1. Fabric Weight

The fabric weight of the original, degummed and dyed samples and the statistical analysis is presented in the Table IV and Figure 1.

Table III

Fabric Weight

SI.NO.	SAMPLE	Mean weight (gm/sq.mt)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	0.308			34.28**
2	Ds	1.319	1.011	328.24	
3	Dds	1.389	1.081	350.97	

Values are mean of 5 readings

** - Significant at 1% level

Os-original Silk, DS- Degummed Silk, Dds-Dyed silk.

From Table III and figure 1, it is noted that the fabric weight of original silk is 0.308 (gm/sq.mt) whereas the weight of Ds and Dds has increased to 1.319 and 1.389 gm/sq.mt respectively.

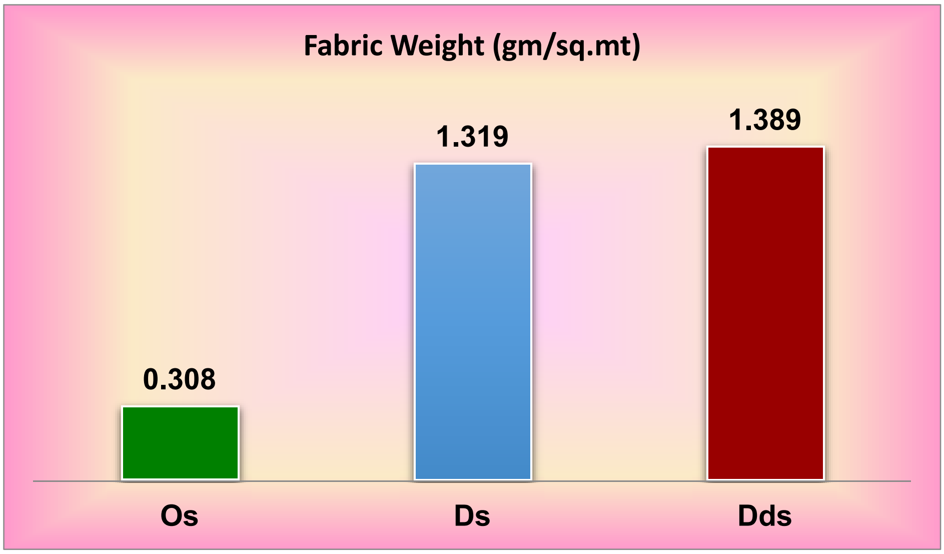

Figure 1

Fabric Weight

4.1.2 Fabric Thickness

The fabric thickness of the original, degummed and dyed samples and the statistical analysis is presented in the Table V and Figure 2.

Table IV

Fabric Thickness

SI.NO.	SAMPLE	Mean thickness (mm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	0.193			4.731**
2	Ds	0.243	0.05	25.9	
3	Dds	0.237	0.044	22.7	

Values are mean of 5 readings

*-significant at 1% level

Os- original Silk, Ds- Degummed silk, Dds- Dyed silk.

From Table IV and figure 2, it is noted that the fabric weight of original silk is 0.193 mm whereas the weight of Ds and Dds has increased to 0.243 and 0.237 mm respectively.

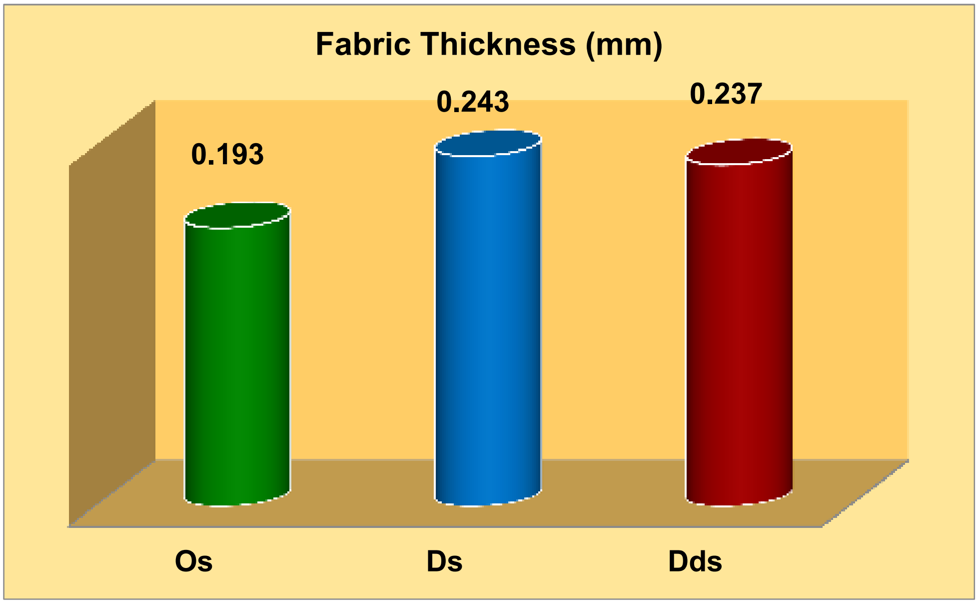

Figure 2

Fabric Thickness

4.1.3. Fabric Strength- warp

The fabric Tensile strength (warp) of the original, degummed and dyed samples and the statistical analysis is presented in the Table VI and Figure 3.

Table V

Tensile strength- warp

SI.NO.	SAMPLE	Mean strength (Kg/cm^3)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	110.0			12.562**
2	Ds	117.0	7.0	6.36	
3	Dds	143.0	33	30	

Values are mean of 5 readings
**-significant at 1% level
Os- original Silk, Ds- Degummed silk, Dds- Dyed Silk

From Table V and Figure 3, it is noted that the fabric weight of original silk is 110.0 (Kg/cm^3) whereas the weight of Ds and Dds has increased to 117.0 and 143.0 Kg/cm^3 respectively.

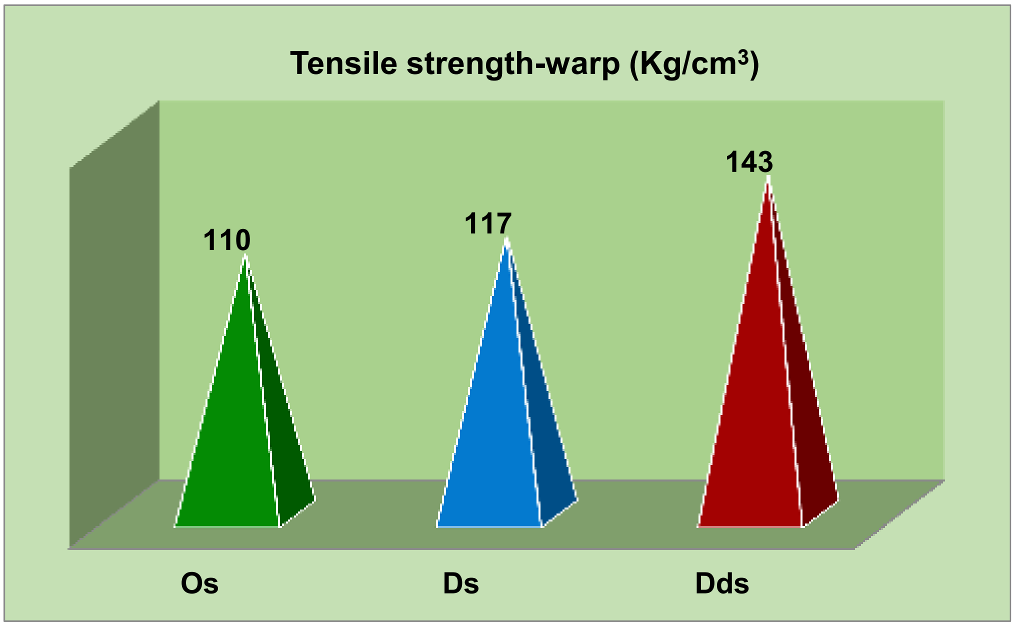

Figure 3

Fabric Strength- Warp

4.1.4 Fabric strength- Weft

The fabric Tensile strength (weft) of the original, degummed and dyed samples and the statistical analysis is presented in the Table VII and Figure 4.

Table VI

Fabric tensile strength- weft

SI.NO.	SAMPLE	Mean strength (kg/cm^3)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	111.2			119.264**
2	Ds	122.4	11.2	10.07	
3	Dds	163.4	52.2	46.94	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table VI and Figure 4, it is noted that the fabric weight of original silk is 111.2 (Kg/cm^3) whereas the weight of Ds and Dds has increased to 122.4 and 163.4 Kg/cm^3 respectively.

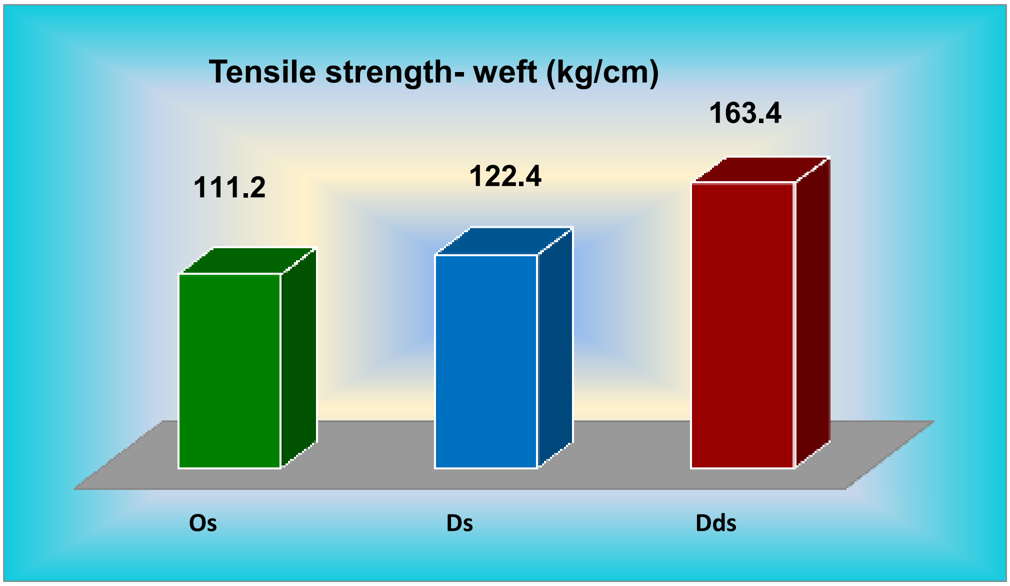

Figure 4

Fabric strength- weft

4.1.5 Fabric Elongation- Warp

The fabric Elongation (warp) of the original, degummed and dyed samples and the statistical analysis is presented in the Table VIII and Figure 5.

Table VII

Fabric Elongation- Warp

SI.NO.	SAMPLE	Mean Elongation (cm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	1.240			4.301**
2	Ds	1.302	0.062	5	
3	Dds	1.324	0.084	77	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table VII and Figure 5, it is noted that the fabric weight of original silk is 1.240 cm whereas the weight of Ds and Dds has increased to 1.302 and 1.324 cm respectively.

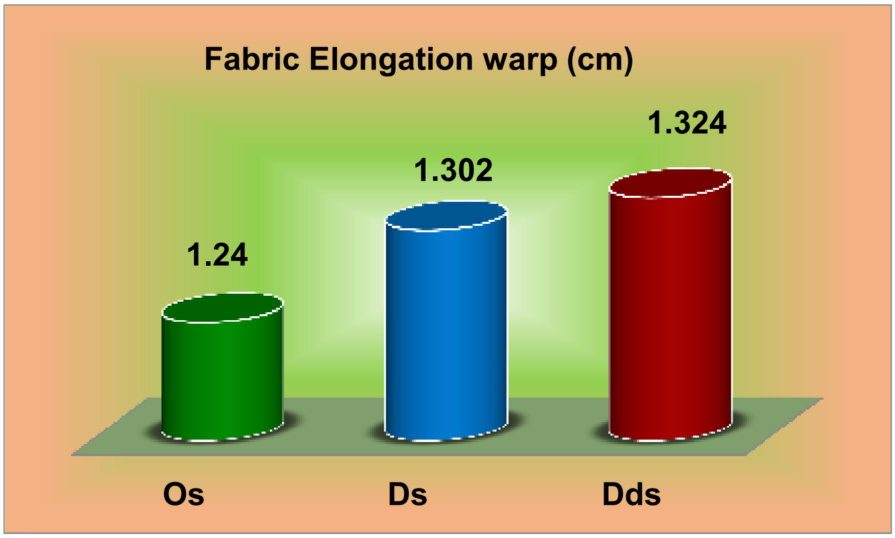

Figure 5

Fabric elongation- warp

4.1.6 Fabric Elongation- Weft

The fabric elongation (weft) of the original, degummed and dyed samples and the statistical analysis is presented in the Table IX and Figure 6.

Table VIII

Fabric Elongation- Weft

SI.NO.	SAMPLE	Mean Elongation (cm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	1.720			1.639^{NS}
2	Ds	1.834	0.114	6.627	
3	Dds	1.860	0.14	8.139	

Values are mean of 5 readings
**- Significant at 1% level
NS – Not significant
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table VIII and Figure 6, it is noted that the fabric weight of original silk is 1.720 cm whereas the weight of Ds and Dds has increased to 1.834 and 1.860 cm respectively.

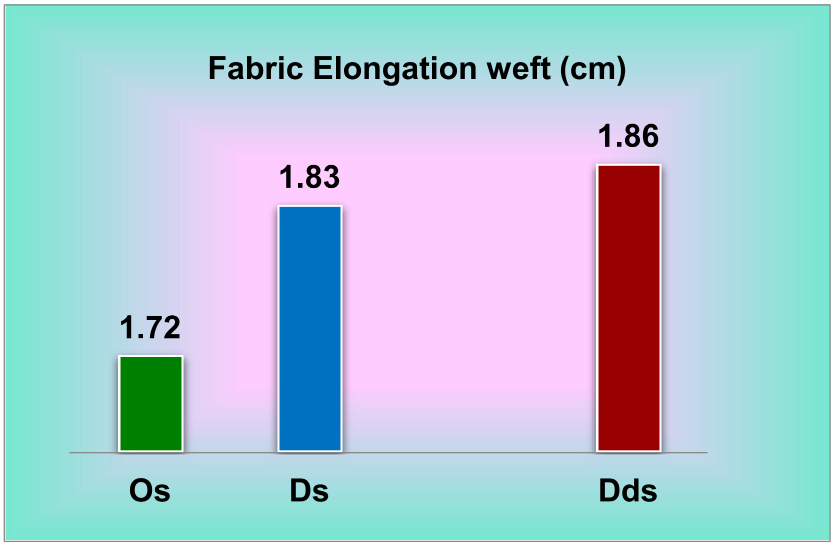

Figure 6

Fabric Elongation- Weft

4.1.7 Fabric Stiffness- Warp

The fabric stiffness (warp) of the original, degummed and dyed samples and the statistical analysis is presented in the Table X and Figure 7.

Table IX

Fabric Stiffness- Warp

SI.NO.	SAMPLE	Mean Stiffness(cm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	2.80			21.539**
2	Ds	2.06	0.74	26.43	
3	Dds	1.80	1	35.71	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table IX and Figure 7, it is noted that the fabric weight of original silk is 2.80 cm whereas the weight of Ds and Dds has increased to 2.06 and 1.80 cm respectively.

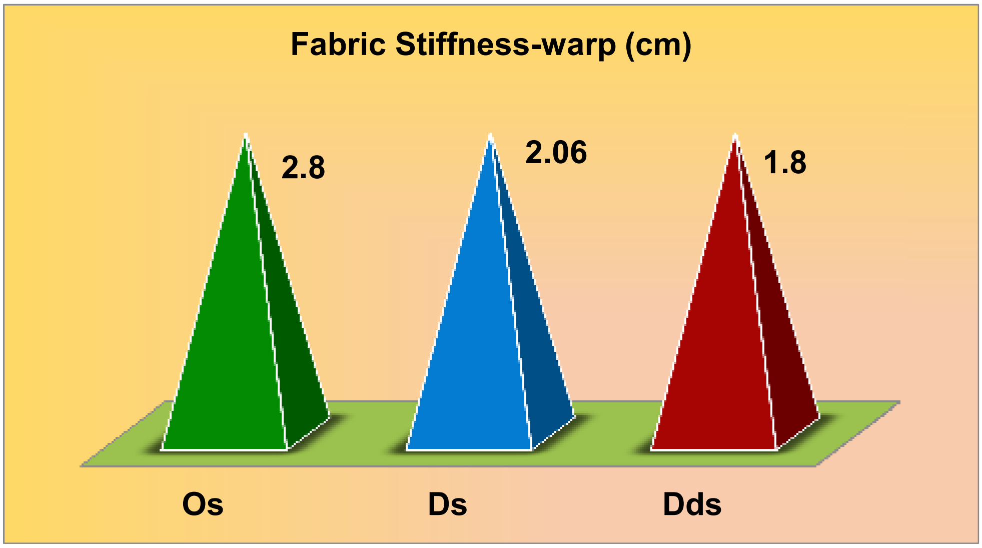

Figure 7

Fabric Stiffness- Warp

4.1.8 Fabric Stiffness- Weft

The fabric stiffness (weft)of the original, degummed and dyed samples and the statistical analysis is presented in the Table XI and Figure 8.

Table X

Fabric Stiffness- Weft

SI.NO.	SAMPLE	Mean Stiffness (cm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	2.50			17.735**
2	Ds	1.820	0.68	27.2	
3	Dds	1.620	0.88	35.2	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk,   Ds- Degummed silk, Dds- Dyed silk

From Table X and Figure 8, it is noted that the fabric weight of original silk is 2.50 cm whereas the weight of Ds and Dds has increased to 1.820 and 1.620 cm respectively.

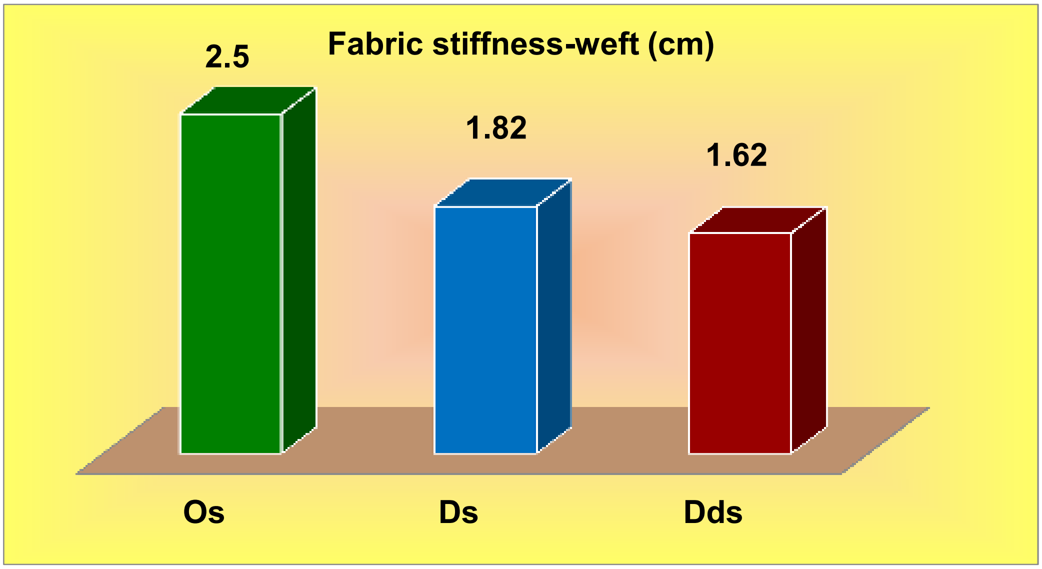

Figure 8

Fabric Stiffness- Weft

4.1.9 Drapability Test

The fabric drapabilityof the original, degummed and dyed samples and the statistical analysis is presented in the Table XII and Figure 9.

Table XI

Drapability Test

SI.NO.	SAMPLE	Mean Drape coefficient (Percent)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	54.06			105.386**
2	Ds	57.08	3.02	5.586	
3	Dds	60.12	5.06	9.359	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table XI and Figure 9, it is noted that the fabric weight of original silk is 54% whereas the weight of Ds and Dds has increased to 57% and 60% respectively.

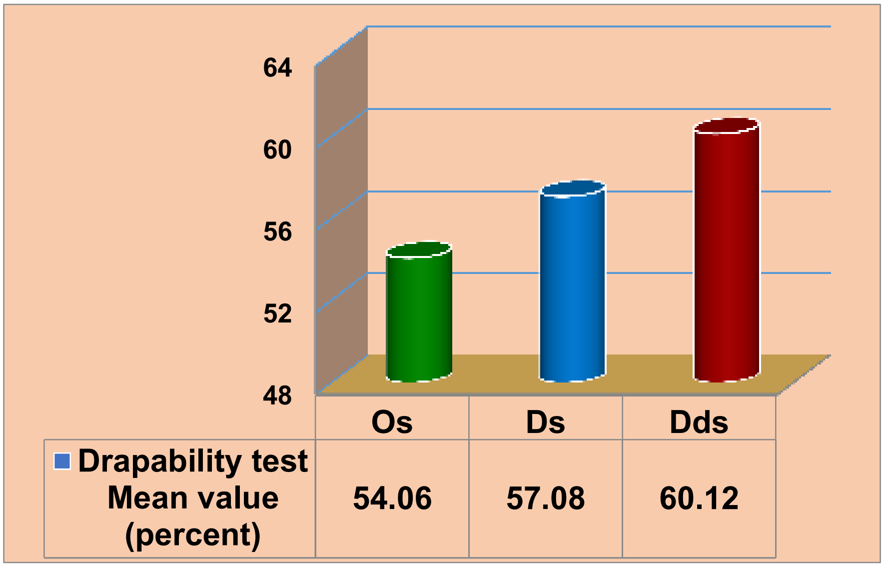

Figure 9

Drapability

4.2 Wettability and Absorbency Test

4.2.1 Drop Test

The drop testof the original, degummed and dyed samples and the statistical analysis is presented in the Table XIII and Figure 10.

Table XII

Drop Test

SI.NO.	SAMPLE	Mean absorbency (seconds)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	31			85.946**
2	Ds	24	7	22.58	
3	Dds	27	4	12.90	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table XII and Figure 10, it is noted that the fabric weight of original silk is 31seconds whereas the weight of Ds and Dds has increased to 24 and 27 seconds respectively.

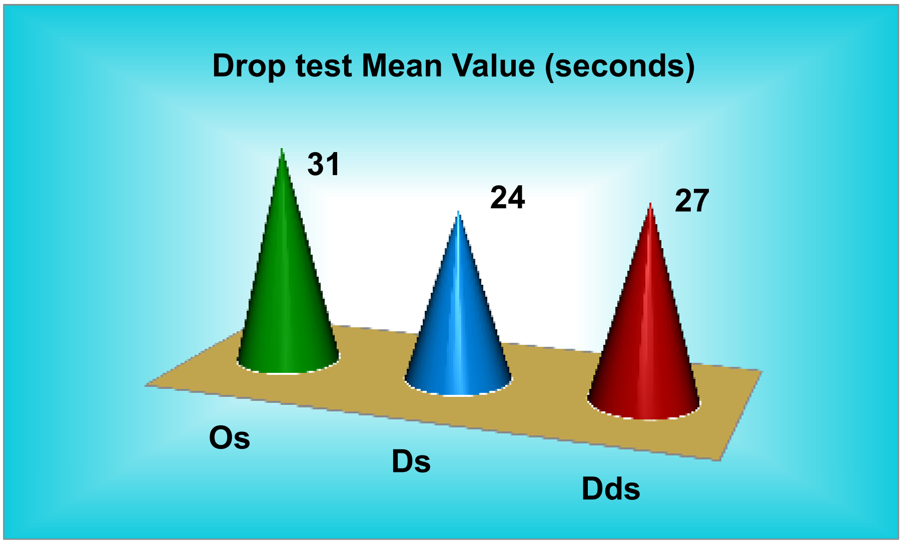

Figure 10

Drop Test

4.2.2 Sinking Test

The sinking test fororiginal, degummed and dyed samples and the statistical analysis is presented in the Table XIV and Figure 11.

Table XIII

Sinking Test

SI.NO.	SAMPLE	Mean Sinking time (seconds)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	42			167.290**
2	Ds	36	6	14.285	
3	Dds	38	4	9.523	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table XIII and Figure 11, it is noted that the fabric weight of original silk is 42 seconds whereas the weight of Ds and Dds has increased to 36 and 38 seconds respectively.

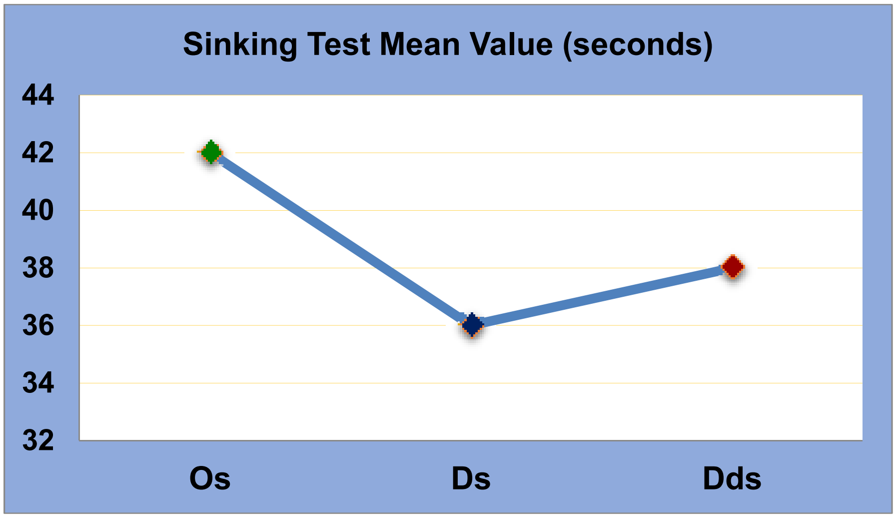

Figure 11

Sinking Test

4.2.3. Capillary Rise Test

The capillary rise test fororiginal, degummed and dyed samples and the statistical analysis is presented in the Table XV and Figure 12.

Table XIV

Capillary Rise Test

SI.NO.	SAMPLE	Mean capillary rise (mm)	Gain of Loss Over Original	% of gain or loss over original	"F" Value
1	Os	58			19.741**
2	Ds	62	4	6.896	
3	Dds	66	8	13.793	

Values are mean of 5 readings
**- Significant at 1% level
OS- Original silk, Ds- Degummed silk, Dds- Dyed silk

From Table XIV and Figure 12, it is noted that the fabric weight of original silk is 58 mm whereas the weight of Ds and Dds has increased to 62 and 66 mm respectively.

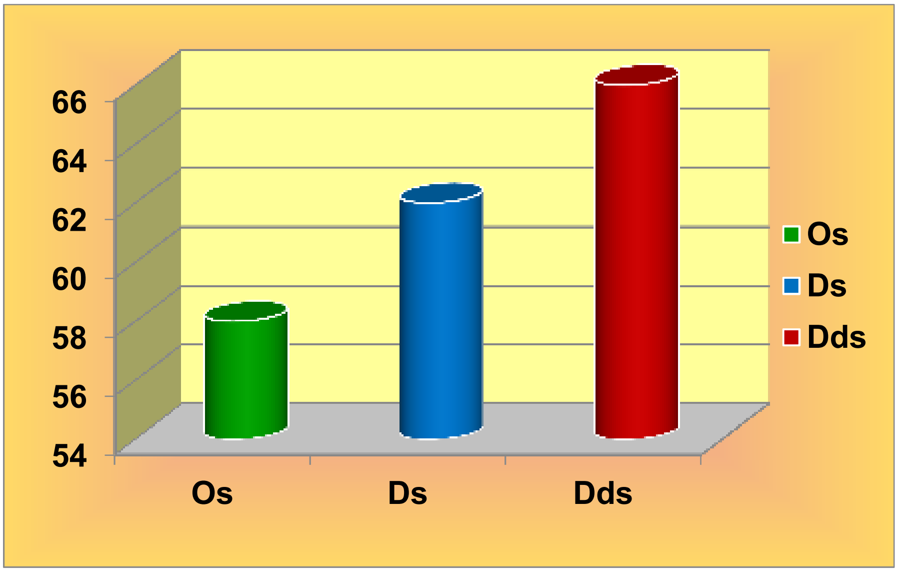

Figure 12

Capillary Rise Test

4.3 Colour Fastness Test

Colour fastness of the original, degummed and dyed samples and the statistical analysis is presented in the Table XVI.

Table XV

Colour Fastness to Sunlight, Crocking, Pressing and Washing.

SAMPLE	Colour fastness to sunlight	Colour fastness to washing	Colour fastness to pressing		Colour fastness to crocking	
			Dry		Wet	
Dds	4	4	4	4	4	3

Dds- Dyed Silk : 1- Very poor, 2- Poor, 3- Moderate, 4- Good, 5- Excellent

The dyed sample has good fastness to sunlight. As regards pressing it has got good fastness to pressing in both dry and wet conditions. The sample has got good fastness to dry crocking and moderate in wet crocking. The dyed sample has got good washing fastness

SUMMARY AND CONCLUSION

V. SUMMARY AND CONCLUSION

Textile industry is one of the nation's oldest and dynamic segment of entire manufacturing industries. Textile industries in India are the backbone of the national economy. It is the largest industry in India occupying a vital role in the Indian economy and occupies a unique place in our country. One of the earliest to come into existence in India, it accounts for about 14 percent of the total industrial production, contributes to nearly 30 percent of the total exports and is the second largest employment generator after agriculture.

The natural dyes have become a considerable interest in dyeing of textile dye to their ecofriendly nature. Dyeing of textile material is the process of applying dye, so that the material not only changes colour but it quickly retains the dye. Fastness property of dye depends on the chemical structure of the colourant, dye concentration, presence of foreign substance and state of dye inside the fiber.

The demand for natural dyes in the modern world is increasing day by day. Global awareness is also set for the use of the natural resources for saving the environment and the earth from pollution and ecological imbalances. Therefore it is an urge to utilize the vast diversity of natural resources of colour pigments, natural dyes like other natural products are becoming more popular. At present, international consumption of natural dyes is very low compared to synthetic dyes. It is obviously due to the existing limitations and technical problems in natural dyeing process. The decreased use of chemicals for dyeing will increase the speed of dyeing cycles, leading to less energy consumption and reduced cost of textile dyes.

Silk fabric remains a dream in every Indian women hood, with the increase in demand for silk products worldwide and the call for eco-friendly natural product. Considering these in mind the present research has been carried out on**"Calliandra Haematocephala- A natural flower colourant for dyeing Khadisilk",** with the following objectives;

- To extract dye from the selected natural source *Calliandra Haematocephala* and stand optimize parameters.
- To dye and evaluate the performance of the dyed khadisilk fabric.
- To reuse the dye solution and measure the colour strength.

METHODOLOGY

The methodology pertaining to the on **"Calliandra Haematocephala- A natural flower colourant for dyeing Khadisilk",** is presented in the following steps.

Khadi silk fabric was selected for the study due to its excellent properties to absorb natural dyes. This was degummed with detergent. Calliandra Haematocephala flower were collected and was made to powder form and used for dyeing. The mordant selected was Alum and Myrobalan .The dye and the mordants were extracted for conducting the pilot study. The optimization for various parameters namely dyes concentration, mordant concentration, temperature and time was done. The three methods namely pre-mordanting, simultaneous-mordanting, post-mordanting was tried and finally was selected. The samples were dyed after the pilot study. Based on the dyeing parameters the silk fabric was dyed with optimized parameters.

OPTIMIZED PARAMETER FOR FINAL STUDY

S.NO	Dyeing parameters	Trial Concentration	Optimized Concentration
1	Dye extraction time (minute)	15,30,45	30
2	Dyeing concentration(gram/100ml)	1,2,3	3
3	Concentration of mordant alum (%)	1,2,3	3
4	Mordanting time (minute)	15,30,45	30
5	Dyeing time (minutes)	15,30,45	30
6	Dyeing temperature (degree)	90°,100°,110°C	100°C

An attempt was made in reuse the dye solution. Dyed fabrics were evaluated for the following aspects;

- Fabric Weight
- Fabric Thickness
- Tensile strength- warp
- Fabric tensile strength- weft
- Fabric Elongation- Warp

- Fabric Elongation- Weft
- Fabric Stiffness- Warp
- Fabric Stiffness- Weft
- Drapability Test
- Drop Test
- Sinking Test
- Capillary Rise Test
- Colour Fastness to Sunlight, Crocking, Pressing and Washing.

FINDING OF THE STUDY

- Optimization was done using 1gm, 2gm and 3gm of the dye powder with 100 ml of water. It is then heated for half an hour and fabric was inserted in the dye solution for half an hour and was removed and calculated.
- The degummed and dyed samples were evaluated objectively.
- The fabric weight is increased 1.389 per cent after dyeing because of more dye intake
- The dyed sample recorded the highest increase in fabric weight by uptake is more. The dyed sample recorded the highest increase in fabric weight by 350.97 per cent.
- The thickness of the fabric is increased by 22.7 per cent
- The tensile strength of the samples along with warp is increased after dyeing by 30 per cent.
- The tensile strength in weft side of the samples is increased after degumming and dyeing process. The gain is shown by 46.94 per cent.
- The elongation along warp of dyed sample has exhibited that 8.139 per cent.
- The dyed silk fabric has lose its stiffness in warp after dyeing; and the decrease in stiffness is 35.71 per cent and in weft direction a decrease in stiffness is 35.2 per cent.
- The fabric drapability is increased after dyeing because of dye uptake is more. The dye sample recorded the highest in fabric drapability by 9.359.
- The dyed silk sample could absorb a drop of water in 27 seconds against 31 seconds as regarded for original.

- The dyed sample sank completely into the water within 38 seconds whereas the original has taken 42 seconds. The degummed sample 36 seconds and it could be concluded that the dyeing has not hindered the absorbency.
- The dyed fabric could wick water upto 66 mm within 60 seconds as 58 mm. The degummed silk fabric has wicked water upto 62 mm in hour seconds.
- With regard to colour fastness tests, good fastness is seen towards sunlight, washing, pressing and dry crocking. Moderate fastness is observed when subjected to wet crocking.
- The dyed solution was reused and the degummed silk fabric was dyed.
- After the fabric was dyed subjective evaluation was done.
- In subjective evaluation the fabric thickness, Fabric tensile strength and elongation, Fabric stiffness, Drapability, colour fastness test, Wettability and absorbency test.

CONCLUSION

The fabric dyed with the Calliandra Haematocephala flower using alum as mordant gave the best result in strength, elongation and absorbency properties. The performance of this study obviously proves that the silk fabric has good absorbency of dye. Colour fastness was also excellent with the mordant. Hence it could be concluded that degummed silk fabric could be dyed with the selected natural source effectively. Hence the natural source and the mordant used for the dyeing propose are safe and eco-friendly.

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APPENDICES

APPENDIX- i

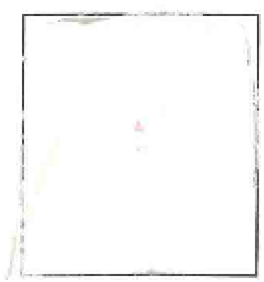
Original and Degummed Samples

Fabric Details

Fabric: Khadi silk

Price per Meter: Rs. 280/-

Original Silk fabric



Degummed Silk fabric

APPENDIX-ii

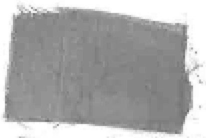
Fabric Dyed in aqueous and Alkaline Solution

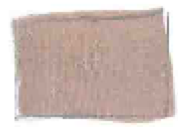
Silk fabric dyed in Aqueous medium

Silk fabric dyed in Alkaline Medium

APPENDIX- iii

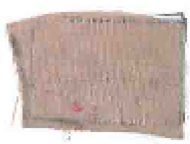
Fabric dyed in Alum and Myrobalan Mordant

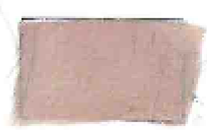


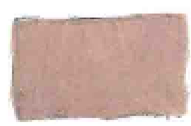


Pre mordantingSimultaneous Post mordanting

AlumAlumAlum

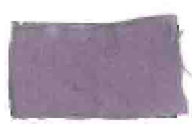


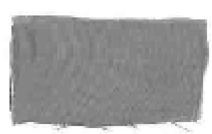




Pre mordantingSimultaneous Post mordanting

MyrobalanMyrobalanMyrobalan



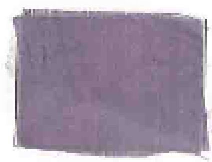


Premordanting Simultaneous Postmordanting

Alum + Myrobalan Alum + MyrobalanAlum + Myrobalan

APPENDIX- iv

Final Study Samples Silk Fabric Dyed with Alum and Myrobalan Mordant



Simultaneous

Myrobalan + Alum