

**Efficacy of Ultrasonic Assisted Dye Extraction from  
*Hibiscus hispidissimus* for UV Protection**

By

**Jinsiya, N M  
(13PTF007)**

A thesis submitted to the  
**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

Signature of the  
Head of the department

Signature of the guide

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# 1. INTRODUCTION

Colour is one of the element of nature that made the human living more aesthetic and fascinating. The whole spectrum of colours can be obtained from a multitude of plants, animals and fungi to dye textiles, to colour our skin, hair and the food we eat (Mehta *et al.*, 2014). Highly coloured substances are known as colourants, can be used to impart colour to an infinite variety of materials described technically as substrates. Colourants can be subdivided in to dyes. Dyeing is a common application used for colouring fibrous substances and staining in the case of biological materials (Shivakumar *et al.*, 2011).

Natural dyes have been used extensively since long periods. It was practiced during the Bronze Age in Europe. The earliest written record of the use of natural dyes was found in China dated 2600 BC (Siva, 2007). Natural dyes can be sorted into three categories- natural dyes obtained from plants for example indigo, those obtained from animals for example cochineal and those obtained from minerals for example ocher (Bechtold *et al.*, 2007). Natural dyes/colourants derived from flora and fauna are delivered to be safe because of its non-toxic, non-carcinogenic and biodegradable nature (Cristea *et al.*, 2003).

The use of natural dyes for textile dyeing purposes, decreased to a large extent after the discovery of synthetic dyes in 1856. Synthetic dyes are produced from cheap petroleum sources and show superior fastness properties (Aminnodin and haji, 2010). With a distinct lowering in synthetic dyestuff costs, the natural dyes were virtually unused at the beginning of twentieth century (Faravani *et al.*, 2008).

Dyeing of textiles with synthetic dyestuff is characterized by high negative impact on the environment as well as end users. In the year 1996 Germany and other European countries imposed the ban on the use of carcinogenic dyes (Kamal *et al.*, 2005). Therefore interest towards natural dyes has increased considerably on account of their compatibility with environment, relatively low toxicity and allergic effects as well as availability of various natural colouring sources such as from plants, insects, minerals and fungi (Teli *et al.*, 2002). Natural

dyes produce very uncommon, soothing and soft shades as compared to synthetic dyes. Dyeing with natural dyes however, normally requires the use of mordants (Kulkarni, 2011).

There is a growing demand for developing suitable extraction technique for natural dyes from plant materials. Ultrasonic approach has been widely used mainly in food industry (Pai *et al.*, 2011) and also in textile industry (Sivakumar *et al.*, 2011). Application of ultrasonic probe can increase the extraction of dyes from different parts of various plant resources. The reason for the improvement could be due to better leaching of natural dye materials from plant cell wall membranes and mass transfer to solvent assisted by acoustic cavitation provided by ultrasonic (Kamel *et al.*, 2011).

For successful commercial use of natural dyes, the appropriate and standardized dyeing techniques need to be adopted without sacrificing required quality of dyed textile materials. Therefore, to obtain newer shades with acceptable colour fastness behaviour and reproducible colour yield, appropriate scientific techniques or procedures need to be derived from scientific studies on dyeing methods, dyeing process variables, dyeing kinetics and compatibility of selective natural dyes (Samanta and Agarwal, 2009).

Consumers have become increasingly aware of the need for sun protection, which is related to the incidence of sun induced skin damage and its relationship with increased exposure to UV-light. Ultra violet radiation can lead to acute and chronic reactions and damage such as acceleration of skin ageing (Sivaramakrishnan, 2008). Reducing the exposure time to sunlight using sunscreen and protective clothes are the ways of protection against the deleterious effect of ultra violet radiation. Apart from sunscreen lotion materials and accessories made of textile materials are largely used for UV protection.

Natural fibers especially wool and silk absorb more ultraviolet rays because of the presence of natural tannins and lignins. Przewozna and Zimniewska (2009) reported that many fabrics dyed with plants are characterized with good UV protection factors. The UV protection factor is the factor by which the period of exposure to sun can be extended without reddening of the skin. It can now be

measured using a spectrophotometer equipped with an integrating sphere (Bajaj *et al.*, 2000). It is reported that some natural dyes not only dye with unique and elegant colours, but they also provide UV protective functions to the fabric.

India is rich in natural wealth and there is ample scope to explore and revive the application of natural dyes on textiles. Hence the present study **“Efficacy of Ultrasonic Assisted Dye Extraction from Hibiscus *hispidissimus* for UV Protection”** aims with the following objectives.

- To extract dye by aqueous and ultrasonic method
- To optimize different parameters for dye extraction and dyeing
- To assess the UV protection property of the dyed fabric.

## 2. REVIEW OF LITERATURE

The review of literature pertaining to the study entitled "**Efficacy of Ultrasonic Assisted Dye Extraction from *Hibiscus hispidissimus* for UV Protection**" is discussed under the following headings:

### 2.1 Silk

#### 2.1.1 Introduction

#### 2.1.2 Discovery

#### 2.1.3 Source

#### 2.1.4 Composition

#### 2.1.5 Types

#### 2.1.6 Properties of silk

##### 2.1.6.1 Physical properties

##### 2.1.6.2 Chemical properties

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### 2.2 Mordants

#### 2.2.1 Introduction

#### 2.2.2 Mordanting technique

##### 2.2.2.1 Pre- Mordanting process

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### 2.4 Natural dye

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- 2.6 *Hibiscus hispidissimus*
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  - 2.6.2 Medicinal uses

## **2.1 SILK**

### **2.1.1 Introduction**

Silk is one of the oldest fiber known to man. Silk is an animal fiber produced by certain insects to build their cocoons and webs. Although many insects produce silk, only the filament produced by the mulberry silk moth, *Bombyx mori*, and a few others in the same genus is used by the commercial silk industry (Babu, 2012)). Silk is a protein fiber. It is made up of protein 'fibroin'. Silk is the only natural fiber that occurs in the form of a fine continuous filament (Arora, 2010).

Silk fibers have been historically referred to as the "queen of textiles" and used as a natural source of textile materials for thousands of years owing to its excellent performance such as luster, comfortableness, breathability, hygroscopicity and so on (Li *et al.*, 2012). Silks natural beauty and properties of comfort in warm weather and warmth during colder months have made it useful in high-fashion clothing. Silk fibers have outstanding natural properties which rival the most advanced synthetic polymers (Chen *et al.*, 2003).

### **2.1.2 Discovery**

The history of silk development spans through centuries and can be traced around the World's very ancient trade route called 'Silk Road'. Many fascinating legends are woven around the discovery of silk but none contains such a wide acceptance as the story of Chinese queen Hsi-Ling-Shih or Silingehi, wife of the Emperor W Hang who ruled over China about 2,500 BC (Datta and Nanavaty, 2007). She was drinking tea under a tree when a ball of silk fell in to her tea. She picked it out and started wrapping it around her finger. She saw a small worm. In an instant she realized that this worm was the source of silk fiber. The Chinese guarded their knowledge of silk (Arora ,2010).

In the third century AD., some Chinese maidens who were sericulture experts, were mysteriously kidnapped and carried through Korea to Japan, where they instructed the people of the court and later Japanese craftsmen who soon produced silk quickly and scientifically. About the same time a Chinese princess married a prince from Khotan and carried with her some eggs of silkworms and

some seeds of the mulberry tree by ingeniously concealing them in her head dress. Later the knowledge of sericulture spread to other parts of the world (Shenai, 1991)

### **2.1.3 Source**

Silk is produced by the larvae from a variety of insects and spiders of the phylum *Arthropoda*. The most useful silks are from the protein secretions of the *Bombycidae* family. They include the domesticated moth *Bombyx mori*, subfamily *Bombycinae*, the wild Chinese tussah moth *Antheraea pernyi* and the Indian tussah moth *Anthorea mylitta* from the subfamily saturniiae (Robson, 1995).

### **2.1.4 Composition**

A silk filament contains 72-81% fibroin, 19-28% sericin, 0.8-1.0% fat and wax and 1.0-1.4% colouring matter and ash of the total weight. The molecular weight of sericin ranges from 10 to 310 KDa. Silk produced by spiders is mechanically superior to any insect silk (Zhou *et al.*, 2000). In its chemical composition, silk fibroin is much simpler than wool (keratin) fiber. Silk fiber chemically a polypeptide, is built up from mainly four amino acids, viz glycine (38-41%), alanine (30-33%), serine (12-16%) and tyrosine (11-12%) (Ghosh, 2004).

### **2.1.5 Types**

Natural silks are broadly classified as mulberry and vanya. Mulberry silks alone accounts for more than 95% of the total raw silk production. Vanya sericulture is universally known as forest or wild sericulture. Tropical and temperate tasar and muga are the commercially exploited vanya silks, in addition to eri silk which is reared indoor (Ojha and Panday, 2004).

### **2.1.6 Properties of Silk**

#### **2.1.6.1 Physical properties**

**Absorbency:** Silk absorb a considerable amount of moisture from the air and under favorable conditions will absorb as much as 30% its weight.

**Density:** Silk is a compact material and possess a density of 1.3 g/cc in the raw state and 1.25 g/cc in the degummed state.

**Effect of heat:** Silk can withstand high temperatures and unaffected by heating to a temperature as high as 140°C, but rapid disintegrations set in at 170°C (Shenai, 1991).

**Tensile Strength:** Studies conducted on some mulberry and non-mulberry varieties by Iizuka *et al.* (1993) reveal that the tenacity, elongation and modulus are all dependent on the linear density of the filament and the linear density or the mean size in turn depends on the silkworm race. The tenacity ranges between 2.5 and 4.82 gram-force per denier (g/d) for Japanese and Chinese mulberry varieties, 2.4 and 4.32 g/d for Indian mulberry varieties (Iizuka., 1996). In a study on chemical structure and physical properties of *Antheraea assama* (muga) silk, it has been reported that the tenacity of muga varies between 3.2 and 4.5 g/d (Freddi *et al.*, 1994).

**Luster and Handle:** The raw state silk filament is harsh and non-lustrous. On removing the sericin the silk receives its well-known soft, elegant luster. The handle of degummed silk is scroopy.

**Abrasion resistance:** The surface of silk is very easily chafed by abrasion. So maximum care is needed, when the material is in wet swollen state.

**Influence of light:** Silk fibers may also be damaged by prolonged exposure to direct sunlight (Gulrajani, 1993).

#### **2.1.6.2 Chemical Properties**

**Action of acid:** Silk readily absorbs dilute acids from solutions and in so doing increases in luster and acquires 'scroop' which is a characteristic crackling sound emitted when the fiber is squeezed or pressed. Silk protein like wool, can be decomposed by strong mineral acids. Concentrated sulphuric and hydrochloric acids dissolve silk and nitric acid colours silk yellow (Sonwalkar, 1993).

**Action of alkalis:** Silk is not damaged by weak alkalis but gets dissolved in hot concentrated alkali solutions.

**Affinity for dyes:** It has good affinity for dyes. It readily absorbs acid, basic and direct dyes.

**Effect of perspiration:** Silk fabrics are damaged by perspiration.

**Effect of bleaches:** Hydrogen peroxide and sodium perborate give good results for bleaching silk. But chlorine bleaches are very harmful (Arora, 2010).

### **2.1.7 Silk Fabric**

The silk fabric comprise the fabric woven with raw silk and degummed after weaving and the fabric woven by using the degummed silk yarn. The former one is called as raw silk fabric and the latter is called the glossed silk fabric. All the raw silk fabric are woven in white colour. After weaving they are degummed and then they are dyed in plain colour or printed as per desire (Panda, 2010).

## **2.2 MORDANTS**

### **2.2.1 Introduction**

Mordants are the substance capable of binding natural dyes to textile substrate (Prabhu *et al.*, 2011). Most natural dyestuffs are unable to form strong bonds with fibers without the aid of mordant material. Mordant is required to increase affinity and binding of dye to the fiber or to create stronger bonds in comparison to using only dye. Thus mordant also improve fastness properties. However, using mordant material can change colour properties and the resultant hue. Mordant material can be divided into two main groups: acidic and basic mordants. Acidic mordants are usually tannin derivatives whereas metal salts of aluminium, chromium, iron etc. used as basic mordants (Bechtold *et al.*, 2007).

Traditionally the use of natural dyes for textile dyeing is connected with the Mordanting process, pre- or post-mordanting. The process of Mordanting has been known for many centuries and it is used to improve dye adsorption on the fiber and its wash fastness because the mordant has a substantivity for both colorant and the fiber and, in bonding with the dye on the fiber forms an insoluble precipitate. The same dye stuff gives different hues or even different colour by using different mordants (Zarkogianni *et al.*, 2010)

## **2.2.2 Mordanting Technique**

There are three methods for application of mordants based on the time of their usage. They are:

1. Premordanting
2. Post mordanting
3. Simultaneous mordanting

### **2.2.2.1 Pre- mordanting process**

As suggested by the name, in premordanting, the mordants are applied to the fabric prior to dyeing. In this process the material is first impregnated into a mordanting bath followed by fixing with sodium carbonate solution. The dyeing is subsequently done in a separate bath. It is a two bath process (Muthu, 2014).

### **2.2.2.2 Simultaneous mordanting process**

In this process the dye and mordant which does not react with each other instantly are simultaneously applied to the fiber from the same bath. First the dye solution is added and the temperature of the bath is raised to 50°C. Dissolved and diluted mordant is then added, the amount of which depends on the depth of the shade. The material is entered in the bath and the temperature is raised to the optimum and dyeing is carried out depending on the fiber used (Casselmann, 1993).

### **2.2.2.3 Post mordanting process**

As the name suggests, the material is first dyed with the natural dye at optimum conditions. The dye bath is cooled to room temperature and the dyed fabric is taken out. The fabric is then transferred directly to a separate bath containing mordant solution. After fixing, the sample is taken out, washed with water and dried in the shade (Karmakar, 2010).

## **2.3 UV PROTECTION PROPERTIES OF NATURAL DYE**

Ultraviolet radiation can cause erythema, sun allergies, increased skin ageing and cancers especially melanoma, which are caused by the damage to

chromosomes in the skin cells. Efficient protection against harmful UV radiation is required when human skin is exposed during sunny conditions. Recognition of the important role that textiles play in improving protection for people significantly increased at the beginning of the twenty-first century. There are many reports related to the investigation of clothing with a high sun protection factor (Rieker *et al.*, 2001).

The UV blocking properties of textiles are influenced by many factors. The structure of the fabric, its density, porosity, thickness, surface mass and type of weave, as well as the type of dyes applied to the fabric play a very important role in ensuring good UV protection (Gambichler *et al.*, 2012). UV radiation amounts to about 6% of solar radiation and consist of UV-A (330-400 nm), UV-B (290-320 nm), and UV-C radiation (220-280 nm). UV-A can penetrate the top layer of skin, there by damaging the inner layers. UV-B radiation can cause sunburn and is thought to be the major reason for skin cancer as it inhibits the synthesis of DNA, RNA and proteins. UV-C is absorbed by the ozone layer and thus causes less damage (Gupta, 2005).

Many dyes absorb UV radiation. Studies show that for a given colour, the darker the shade the higher the protection. As the spectral region of all dyes extend to the UV region (290-400 nm), all dyes act as UV absorbers (Gupta, 2005). A study on the ability of the natural dyestuffs to block UV radiation shows that the application of natural dyes to linen and hemp fabrics results in better UV protection, in comparison with synthetic dyes in the same colour ranges used on the same fabric (Schimidt *et al.*, 2002).

## **2.4 NATURAL DYE**

### **2.4.1 Introduction**

Dyeing is a method which imparts beauty to the textile by applying various colours and shades on to the fabric. Dyeing can be done at any stage of the manufacturing of textile –fiber, yarn , fabric or a finished textile products including garments and apparels. The property of colour fastness depends upon selection of proper dye and selection of the method for dyeing. Dyeing with natural dye is traditional craft of India. Natural dye has been used throughout history and first

written record of natural dyeing is thought to date back to around 4,500 years ago (Jayalakshmi and Soudanyaa, 2014). Natural colours are defined as those colorants that are obtained from animal or vegetable matter without chemical processing. Natural dyes are considered as mordanting dyes. They are mainly applied on cotton, silk, and wool fibers (Karmakar, 2007).

Considering the energy saving and environmental protection, dyeing of textiles with natural dyes has been receiving increasing attention from researchers and manufacturers. It has been reported that natural dyes can be successfully extracted from many plants such as henna leaves, *Calypha wilkesiana* leaves, rhubarb, beetroot, etc. (Mahale *et al.*, 2003). The worldwide demand for natural raw materials is nowadays of great interest as a result of increased consumer awareness and a popular demand for natural products. Natural dyes are particularly relevant today because of their attractiveness in terms of minimal environmental and health impact versus synthetic dyes, which has been suggested as a source of human diseases as skin cancer or allergic contact dermatitis (Moreau and Goossens, 2005).

#### **2.4.2 Advantages**

Natural dyes are considered to be ecofriendly as these are obtained from renewable source as compared to synthetic dyes which are derived from non-renewable petroleum resources. These are biodegradable and the residual vegetable matter left after extraction of dyes can be easily composted and used as fertilizer. In addition to environmental benefits natural dyes also offer functional benefits to the wearer and users of such textile. Many of the natural dyes absorb in the ultra violet region and therefore fabrics dyed with such dyes offer good protection from ultraviolet light (Muthu, 2014). Natural dyes can exhibit better biodegradability and generally have a higher compatibility with the environment (Ibrahim *et al.*, 2010).

#### **2.4.3 Disadvantages**

Natural dyestuff has some drawbacks leading to a rise in use of synthetic dyestuffs. For instance dye plants of the same species may contain different amount of same dyestuff ingredient depending on its origin. Moreover the natural

dyestuff source might be found for a particular season only in a year, which limits the production (Zhang *et al.*, 2011).

Nevertheless, the use of natural dye has been always limited by the absence of a standard method of dyeing with natural dye and as a consequence results in the non-reproducibility of the shades (Guesmi *et al.*, 2012). The extractions are unsuitable for large scale preparations as they are laborious and time consuming. Only a small amount of dye can be obtained by a large amount of the animal or vegetable matter. Hence they are invariably expensive. The natural dyes are not pure and lack uniformity of quality. Further their fastness properties were not good and they faded in light (Singh, 2002).

#### **2.4.4 Classification**

Natural dyes can be classified in various ways. Earlier method of classification was based on the alphabetical arrangement of dyes. Later on, numerous other methods of classification were adopted.

##### **2.4.4.1 Classification based on structure**

Natural organic dyes and pigment belong to a wide range of chemical classes, such as indigoid, anthroquinoids, naphthoquinones, polymethines, ketones, imines, flavones, and chlorophyll.

#### **Indigo**

This is one of the most ancient natural dyes used in textiles by humans. It is also considered to be the most important dye of plant origin. The whole plant is used for the extraction of dye. The extracted dye is supplied in powder form. The dye gives a deep blue colour on silk, wool, cotton, etc. (Agarwal and Patel, 2000).

#### **Anthroquinone**

Some of the most important red dyes are based on the anthroquinone structure. These are obtained from both plants and insects. They form complexes with metal salts and the resultant metal-complex dyes have good fastness to light.

## **Naphthoquinones**

Lawsone otherwise known as hennotannic acid is the most prominent member of this class which is obtained from *L. inermis*.

## **Flavones**

Many pale-yellow flowers, such as carnations and *Cyclamen persicum*, contain 2', 4,4', 6'- tetrahydroxy chalcone (TCH), 2'-glucoside, *Carthamus tinctorius*, commonly known as safflower, produces a rare yellow glucoside, safflomin A, which is used as a colourant. Flavonoles and flavones are very pale yellow and are mostly invisible to the human eye (Tanaka *et al.*,2008).

## **Anthocyanins**

A total of 19 types of anthocyanidins, aglycones or chromophores of anthocyanins are known, but there are only six major ones: pelargonidin, cyaniding, peonidin, delphinidin, petunidin and malvidin. Their colour depends greatly on the number of hydroxyl groups on the B-ring: the larger number of groups, the bluer the colour.

## **Carotenoids**

Carotenoids are isoprenoid compounds with polyene chains that may contain upto 16 conjugated double bonds. More than 700 naturally occurring carotenoids are known. Carotenoids differ from anthocyanins and betalains in that they play essential roles in plant life (Auldridge *et al.*, 2006).

### **2.4.4.2 Classification based on application**

Natural dyes are of two types: substantive or non- mordant dyes and adjective or mordant dyes. Substantive colours produce fast shades at boil and include walnuts and lichens; mordant dyes require an additional chemical to make the colour permanent (Gulrajani, 1993).

### **2.4.4.3 Classification based on colour**

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

### **Natural yellow colours**

Yellow natural dyes include turmeric, kamela, tesu, marigold, larkspur, harshingar, annatto, berbr's and dolu. The number of plants that yield yellow dyes is much higher than the number that yields other colours.

### **Natural red dyes**

Red dyes include mainly safflower, manjit, patang and lac. The sources of these dyes are limited. Throughout history red dyes have been of significant and in some cases legendary importance. Almost all of natural dye has a quinone structure. They are mainly comprised of anthroquinones or naphthoquinones. Some of the prominent natural red dyes are obtained from plant, animal, and mineral sources (Chakraborty *et al.*, 2005).

### **Natural blue dyes**

Indigo is the only natural blue vat dye, which is synthesized too. The pigment is present in the form of a soluble colourless glucoside indicant (a combination of glucose and indoxyl) which oxidizes to form insoluble pigment indigotin. It is fast to washing, light, milling, perspiration and peroxide bleach.

### **Natural black dye**

One important black natural dye is Logwood which is also known as Cam peachy wood, because it was discovered by the Spaniards on the bay of Campeche in Mexico. It is still used today for dyeing silk in deep shades on an iron tannate mordant.

### **Natural brown dyes**

Brown dyes are rich in tannins, moderately fast colours obtained like henna, onion skins etc. The majority of natural brown dyes are obtained from quinone based dyes, naphthoquinones and anthroquinones (Erica *et al.*, 1995).

## **2.5 EXTRACTION OF NATURAL DYE**

Extraction of colour from natural sources is a very important step in isolation, identification, characterization and dyeing of various natural and manmade fibers. The ancient methods used by traditional dyers and artisans were

based on aqueous extraction. The extraction of colour from raw materials was carried out in boiling water for a prolonged period

### **2.5.1 Conventional Aqueous Extraction**

Aqueous extraction was traditionally used to extract dyes from plants and other materials. In this method, the dye containing material is first broken in to small pieces or powdered and sieved to improve extraction efficiency. It is then soaked with water in earthen, wooden, or metal vessels for a long time usually overnight to loosen the cell structure and then boiled to get the dye solution which is filtered to remove non dye plant remnants (Hardman and Pinhey, 2009).

### **2.5.2 Acid and Alkali Extraction process**

As many dyes are in the form of glycosides, these can be extracted under dilute acidic or alkaline conditions. The addition of the acid or alkali facilitates the hydrolysis of glycosides resulting in better extraction and higher yield of colouring materials. An acid hydrolysis process is used for the extraction of dye from tesu flowers. Alkaline extraction is suitable for dyes having phenolic groups as they are soluble in alkali, which improve the dye yield (Bechtold and Mussak, 2009).

### **2.5.3 Solvent Extraction**

Solvent extraction of any substance is basically the extraction of colouring matter or any other compound in the substance, using a non-aqueous liquid. This non aqueous liquid is generally an organic solvent which dissolves the water insoluble compounds present in the substance. This extraction process require a soxhlet extraction equipment where a suitable solvent is used at an ambient or low temperature of 40-60°C which facilitates dissolution of water insoluble compounds in the raw material (Bhattacharyya, 2010).

### **2.5.4 Ultrasound and Microwave Extraction**

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

cavitations are formed in the liquid. These increase in size but upon reaching a certain size, they cannot retain their shape. When this happens, the cavity collapses or the bubbles burst creating high temperature and pressure. The creation of very high temperature and high pressure increases the extraction efficiency within a short time.

In microwave extraction, the natural sources are treated with a minimum amount of solvent in the presence of microwave energy sources. Microwave increases the rate of the processes so the extraction can be completed in a shorter time with better yield (Muthu, 2014).

### **2.5.5 Enzyme Assisted Extraction**

This method of extraction uses the enzymes produced by the microorganisms present in the atmosphere or those present in the natural resources for assisting the extraction process. Indigo extraction is the most common example for this type of extraction. Indigo occurs in the *Indigoferae* species of plants as a glucoside called indican. This is extracted with water when it gets hydrolyzed by the enzyme into indoxyl and dextrose. In alkaline medium indoxyl gets oxidized to indigo, which is precipitated (Singh, 2002).

## **2.6 *Hibiscus hispidissimus***

### **2.6.1 Introduction**

*Hibiscus hispidissimus* belonging to the family Malvaceae is known as “Comfort root” in English and “Uppanacham” in Malayalam. It is distributed throughout India. Wild Hibiscus is a large climber, with sometimes reddish stems covered with hooked prickles. Alternately arranged leaves, 6-8 cm, are palmately 3-5-lobed, hairy, heart-shaped at base, prickly on the nerves. Leaf margins are toothed, lobes long-pointed, and the leaf-stalks are 5-10 cm long, prickly. Stipules are lance shaped. Yellow flowers arise singly in leaf axils, carried on 3-5 cm long prickly stalks. Bracts below the flowers are 8-12, with leafy appendages. Sepals are hairy. Seed capsules are 1 cm long, ovoid, pointed, enclosed in enlarged sepal cup. Wild Hibiscus is commonly found in the evergreen forests of Western Ghats (Krishnakumar *et al.*, 2008).

### **2.6.2 Medicinal Uses**

*Hibiscus hispidissimus* Griffith is used in tribal medicine of Kerala, the southernmost state of India, to treat liver diseases. Leaves are reported to improve digestion and are antihelmintic. The juice of leaves mixed with honey is used in the treatment of eye diseases while the roots are used in the treatment of kidney disorders, burning sensation and swellings (Marg, 2010).

### 3. EXPERIMENTAL PROCEDURE

Experimental procedures pertaining to the study on "**efficacy of Ultrasonic Assisted Dye Extraction from *Hibiscus hispidissimus* for UV Protection**" are discussed under the following headings.

#### 3.1 Extraction of Natural Dye

##### 3.1.1. Selection of Source

##### 3.1.2 Aqueous Extraction of Dye from *Hibiscus hispidissimus*

##### 3.1.3 Optimization of various parameters for Aqueous Extraction of Dye

###### 3.1.3.1 Solvent

###### 3.1.3.2 Dye Concentration

###### 3.1.3.3 Time

###### 3.1.3.4 pH

###### 3.1.3.5 Temperature

##### 3.1.4 Aqueous extraction of dye under optimized conditions

##### 3.1.5 Ultrasonic Extraction of Dye from *Hibiscus hispidissimus*

##### 3.1.6 Optimization of various parameters for Ultrasonic Extraction of Dye

###### 3.1.6.1 Dye Concentration

###### 3.1.6.2 Time

###### 3.1.6.3 pH

###### 3.1.6.4 Temperature

##### 3.1.7 Ultrasonic Extraction of dye under optimized conditions

#### 3.2 Selection of fabric

##### 3.2.1 Pre-treatment of fabric

###### 3.2.1.1 Degumming

#### 3.3 Mordanting

##### 3.3.1 Selection of mordants

###### 3.3.2 Selection of mordanting techniques

- 3.4 Dyeing
  - 3.4.1 Optimization of Various Parameters for Dyeing
  - 3.4.2 Dyeing by conventional method
  - 3.4.3 Dyeing by ultrasonic method
  - 3.4.4 Nomenclature
- 3.5 Evaluation of Dyed Fabrics
  - 3.5.1 Subjective Evaluation – Visual Inspection
  - 3.5.2 Objective Evaluation
    - 3.5.2.1 Fabric Weight
    - 3.5.2.2 Fabric Thickness
    - 3.5.2.3 Fabric Strength and Elongation
    - 3.5.2.4 Fabric Stiffness
    - 3.5.2.5 Drape Test
    - 3.5.2.6 Crease Recovery
    - 3.5.2.7 Drop Test
    - 3.5.2.8 Sinking Test
    - 3.5.2.9 Capillary Rise Test
- 3.6 Colour Fastness Test
  - 3.6.1. Fastness to Sunlight
  - 3.6.2. Fastness to Crocking
  - 3.6.3. Fastness to Wet and Dry Pressing
  - 3.6.4. Fastness to Washing
- 3.7 Evaluation of UV Protection Factor

### **3.1. EXTRACTION OF NATURAL DYE**

#### **3.1.1. Selection of Source**

Natural dyes are non-toxic, non-polluting, non-carcinogenic, non-poisonous and environmental friendly. They produce harmonizing colours, gentle, soft, lustrous, and subtle creating restful. Natural dyes can offer not only a rich and varied source of dyestuff, but also the possibility of an income through sustainable harvest and shade of these plants (Jothi, 2008). *Hibiscus hispidissimus* (Kasthuri bhendi) (Plate 1) was selected for the present study for extracting dye. Kasthuri bhendi flowers were collected in and around Calicut district, Kerala. The pedal and receptacles were removed and then dried under shade (Plate 1). The dried flowers were powdered using a grinder to get a coarse powder (Plate 1).

#### **3.1.2 Aqueous Extraction of Dye from *Hibiscus hispidissimus***

Three grams of kasthuri bhendi powder were taken and soaked in 100 ml of distilled water and heated in a beaker kept over a water bath for 2 hours at 90°C. The resulting liquid was filtered and used as dye solution. The optical density of the dye solution was measured using UV-visible spectrophotometer at 400 nm (Maximal Absorbance Wavelength).

#### **3.1.3 Optimization of various parameters for Aqueous Extraction of Dye**

##### **3.1.3.1 Solvent**

To determine the suitable solvent for extraction, 3 g of *Hibiscus hispidissimus* were crushed with different solvents such as water, ethanol, and NaOH individually and placed in water bath for two hours at 90°C. The resulting dye extract was filtered and the color intensity was measured at 400 nm using spectrophotometer. The solvent which produce maximum colour yield was selected as the suitable solvent and used for further study.

### **3.1.3.2 Dye Concentration**

To determine the optimum concentration of the dye source, dried kasthuri bhendi powder at various concentration such as 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5% were taken individually and soaked in 100 ml of distilled water and heated in a beaker kept over a water bath for 2 hours at 90°C. The resulting liquid was filtered and used as dye solution. The concentration at which the colour intensity was higher was fixed as optimum dye source concentration and used for the subsequent study.

### **3.1.3.3 Time**

To determine the optimum extraction time, the dye extraction was carried out at different time intervals such as 30-180 min. The dye solution was analyzed using spectrophotometer at 400 nm. The time at which the absorbance was maximum was selected as optimum time.

### **3.1.3.4 pH**

To determine the optimum pH for extraction, the pH of the solvent was adjusted to 1, 3, 5, 7, 8 and 10 using 1N citric acid or 1 N Na<sub>2</sub>CO<sub>3</sub>. Optical density of the dye solution was determined (Plate 2).

### **3.1.3.5 Temperature**

To determine the optimum temperature, the dye extraction was carried out at different temperatures such as 30- 90°C at 10°C interval. The optical density of the dye solution was analyzed spectrophotometrically at 400 nm. The temperature at which the absorbance was maximum was selected optimum temperature for dye extraction.

### **3.1.4 Aqueous 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 (Plate 1).

### **3.1.5 Ultrasonic Extraction of Dye from *Hibiscus hispidissimus***

The flowers of *Hibiscus hispidissimus* was dried in the shade. Then the shade dried flowers were powdered to get a coarse powder. Three grams of flower powder were taken and soaked in 100 ml of distilled water. The beaker was then placed in the ultrasonic cleaner for 1 hour at 80°C. The resulting liquid was filtered and used as dye solution. The optical density of the dye solution was measured using a spectrophotometer at 400 nm (Rahman *et al.*, 2013).

### **3.1.6 Optimization of various parameters for Ultrasonic Extraction of Dye**

#### **3.1.6.1 Dye Concentration**

To determine the optimum concentration of the dye source, dried kasthuri bhendi powder at various concentration such as 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5% were taken individually and soaked in 100 ml of distilled water. The beaker was then placed in the ultrasonic cleaner for 90 min at 80°C. The resulting liquid was filtered and used as dye solution. The concentration at which the color intensity was maximum was fixed as optimum dye concentration and used for the subsequent study.

#### **3.1.6.2 Time**

To determine the optimum extraction time, the dye extraction was carried out at different time intervals such as 30-180 min. The dye solution was analyzed using spectrophotometer at 400 nm. The time at which the absorbance was maximum was selected as optimum time.

#### **3.1.6.3 pH**

To determine the optimum pH for extraction, the pH of the solvent was adjusted to 1, 3, 5, 7, 8, and 10 using 1N citric acid or 1 N Na<sub>2</sub>CO<sub>3</sub>. Optical density of the dye solution was determined (Plate 2).

#### **3.1.6.4 Temperature**

To determine the optimum temperature, the dye extraction was carried out at different temperatures such as 40-80°C. The optical density of the dye solution was analysed spectrophotometrically at 400 nm. The temperature at which the

absorbance was maximum was selected as optimum temperature for dye extraction.

### **3.1.7 Ultrasonic Extraction of dye under optimized conditions**

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

## **3.2. SELECTION OF FABRIC**

Silk is a protein fibre produced as a continuous filament. Silk is the most elegant textile in the world with unparalleled grandeur, natural sheen, and inherent affinity for dyes, high absorbance, light weight, soft touch and high durability and known as the “Queen of Textiles” the world over. On the other hand, it stands for livelihood opportunity for millions owing to high employment oriented, low capital intensive and remunerative nature of its production. Silk fabric made out of plain weave having the specification was selected for the present study:

Warp : 61 EPI (Ends per Inch)

Weft : 59 PPI (Picks per Inch)

### **3.2.1 Pre-treatment of fabric**

#### **3.2.1.1 Degumming**

Prior to the dyeing of raw, a process known as ‘boiling off’ or ‘degumming’ removes the sericin from raw silk. Sericin is easily not soluble in cold water. The degumming in a solution of soap in the presence of mild alkali like soda ash is commonly practiced. The silk fabric was treated with soap (5gpl) and soda ash (1gpl) at a temperature of 90°C for 45 minutes. Then the fabric selected was washed and dried under shade.

## **3.3 MORDANTING**

### **3.3.1 Selection of mordants**

A mordant is a substance used to set dyes on fabric or tissue sections by forming a co-ordinating complex with the dye which then attaches to the fabric or tissue.

Natural mordants such as orange peel, pomegranate rind, myrobolan and alum was selected. Pilot study was carried out to select the suitable mordant. Among the natural mordants, orange peel gave better colour when compared with other mordants. Hence alum was selected for this study (Plate 3).

### **3.3.2 Selection of mordanting techniques**

Mordanting is a process of impregnating textiles with a mordant 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. In Simultaneous mordanting and dyeing are carried out at the same time (Katyayini and Jacob, 2006) All the three types of mordanting were carried out and among these three mordanting techniques, the pre-mordanting technique was found to give better shade. Hence pre-mordanting technique was followed for the present study (Plate 4).

## **3.4 DYEING**

### **3.4.1 Optimization of Various Parameters for Dyeing**

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, 40, 60 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 intervals (30, 60, 90, 120 min). Dyeing temperature was determined by dyeing the selected fabric at different temperature (50, 60, 70, 80, 90,100°C).The optimized parameters for dyeing the selected silk fabric is as follows.

**Table 1****Optimization of Various Parameters for Dyeing**

<b>Parameters</b>	<b>Conventional Method</b>	<b>Ultrasonic Method</b>
Material : Liquor Ratio	1:20	1:20
Solvent	Water	Water
Dyeing time	2 hours	1 hour
pH	7	7
Dyeing temperature	90° C	70° C
Dye extract concentration	80% OWM	60% OWM
Mordant concentration	3%	3%

**3.4.2 Dyeing by conventional method**

Silk fabric was soaked in dye extract. The sample was kept over water bath for 2 hour. Dyeing was carried out at 90°C for 2 hour using 1: 20 material: liquor ratio at neutral pH. After dyeing, the samples were soaped at 60°C for 15 min and rinsed in water.

**3.4.3 Dyeing by ultrasonic method**

The pre-treated silk fabric was introduced into the dye bath containing material: liquor ratio of 1: 20 at a pH of 7. The beaker was then placed into the ultrasonic cleaner for extraction process at optimized conditions. The beaker was positioned in the centre of the tank. The bottom plane of beaker is aligned parallel to the cleaner bottom plane and distance between them was also maintained as same in all experiments. The ultrasonic cleaner generated a maximum ultrasonic power of 500W and a frequency of 40 KHz. The dyed sample were rinsed with hot water, soaped in a bath of liquor ratio 1: 50 using 3g/L non-ionic detergent at 70°C for 5 min, then rinsed and air dried (Mongkholrattanasit *et al.*, 2011).

### 3.4.4 Nomenclature

Table 2

Nomenclature of the Dyed Sample

Samples	Code
Original Sample	OS
Conventional Dyed Sample	CDS
Ultrasonic Dyed Sample	UDS

### 3.5 EVALUATION OF DYED FABRICS

#### 3.5.1 Subjective Evaluation – Visual Inspection

The fabric samples dyed with red sandal wood powder was evaluated visually by the members comprising of 21 PG students specializing in the field of Textiles and Clothing and Bio-textiles, evaluated the fabric for general appearance, brilliancy of shade and evenness of dyeing.

#### 3.5.2. Objective Evaluation

Objective evaluation includes laboratory tests. Testing refers to anything used to distinguish or defect substances in any matter. Textile testing as a whole refers to the vigorous testing done on textile material which may be inside the laboratory as well as in its natural setting using various testing aids and technical instruments (Jewel, 2009).

##### 3.5.2.1. Fabric Weight

A sample of 10×25cm was cut using a GSM cutter. It has sharp blades which penetrate onto the fabric when pressure is imparted with a slight twist. The weight of the cut samples was found using electronic weighting balance. The inference found was calculated using the following formula:

$$\text{Grams per square metre (GSM)} = \frac{\text{weight of the fabric * square meter}}{\text{area of square}}$$

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.

### **3.5.2.2. Fabric Thickness**

Thickness tester 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 read 0.01mm. The sample was placed on the anvil without tension or creases and the pressure foot was lowered onto the sample for two seconds at 2kg pressure. The dial reading was recorded. The readings were calculated similarly and mean value of the readings from the original and dyed cotton material was tabulated.

### **3.5.2.3. Fabric Strength and Elongation**

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

The original and dyed fabrics were tested for tensile strength using Eureka Cloth Strength Tester. 12 inches×2 inches specimen from each samples were cut in both warp 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 to zero. Before starting the machine the pendulum lock was released and the machine was started to run. At certain point the fabric starts to break, and the machine was switched off and the dial reading was taken.

Elongation reading was noted from the 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 (Adanur, 2001).

### **3.5.2.4. 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 A was cut to the size of 15 cm x 2.5 cm using the template. The sample was

placed on the platform with the 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 the 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 readings were taken.

#### **3.5.2.5. Drape Test**

Drapeability 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 it 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.

Drape co-efficient was 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

$$F = \frac{A_s - A_d}{A_D - A_d * 100}$$

Ten readings were taken from the original sample and from the dyed samples, their mean value was calculated and tabulated

#### **3.5.2.6. Crease Recovery**

Crease recovery is the ability of a fabric to recover from unwanted creases. Creasing of textile material is a complex effect involving tensile, flexing, compressive and other stresses. Creasing of a fabric during wear is not a change in appearance that is generally desired. The ability of a fabric to resist creasing is in the first instance dependent on the type of fibre used in its construction (Saville, 2004).

### **3.5.2.7 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 holding the assembly at an angle of 45 degrees-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.

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).

### **3.5.2.8 Sinking Test**

A 1000 ml 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 sample to sink was noted. The same procedure was repeated for all samples. The mean value was calculated.

### **3.5.2.9 Capillary Rise Test**

The samples were cut into sizes of 15 cm length and 10 cm 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 grams weight was attached to immerse inside beaker filled with distilled water. The rate of water level in the sample was measured after 60 seconds.

## **3.6. COLOUR FASTNESS TEST**

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

Colour fastness properties, of the dyed textile material depend upon the nature of dye, dyeing procedure, washing methods and after treatment methods. The grey

scale employed for colour fastness test is 1-5 grade In this scale one means poor and 5 represents excellent colour fastness (Smith, 2006).

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

### **3.6.1. Fastness to Sunlight**

The colour fastness of textile material to day light is very important property. Colour fastness determines the resistance of a material to change its colour characteristics as a result of exposure to light source (AATCC, 2007). 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 parts by marking 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 portions were exposed accordingly along with the first portion. The first portion got seven days exposure to sunlight. The changes in color of dyed samples were compared with the original and the specimens were rated using grey scale.

### **3.6.2. Fastness to Crocking**

Sasmira crock meter was used to determine the fastness of the dyed textile to wet and dry crocking. It has 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 cotton material with ring. The samples were cut into pieces with the size of 20\*10cm, the sample was fixed on the base block with longer side in the direction of rubbing the white desized original material (5\*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. Each sample was given ten strokes and the colour change and staining on the white cloth were graded.

### **3.6.3. Fastness to Wet and Dry Pressing**

Test specimens of 5cm\*10cm 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.

#### **3.6.4. Fastness to Washing**

Major loss of colour 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 undyed white cloth which was desized well. Specimen was completely soaked in the soap solution about 5 g / l for 30 minutes at 40°C, 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 grey scale. The same procedure was carried out for other dyed sample.

### **3.7 EVALUATION OF UV PROTECTION FACTOR**

The transmittance and UPF values of the original silk conventional and ultrasonic silk fabric dyed were measured using a Labsphere Inc USA and UV-2000F at 250- 400 nm range. The UPF value of the fabric is determined from the total spectral transmittance based on AATCC 183: 2010.

Fabrics with a UPF value in the range of 15–24 are defined as providing “good UV protection”, 25–39 as “very good UV protection”, and 40 or greater as “excellent UV protection”. There is no rating assigned if the UPF value is greater than 50 (Rungruangkitkrai *et al.*, 2012).

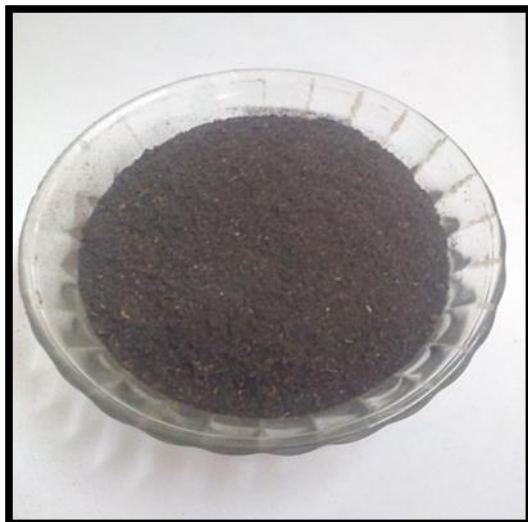
**Plate 1**  
**Dye Source**



***Hibiscus hispidissimus***  
**(kasthuri Bhendi)**



**Dried Kasthuri Bhendi**



**Kasthuri bhendi powder**



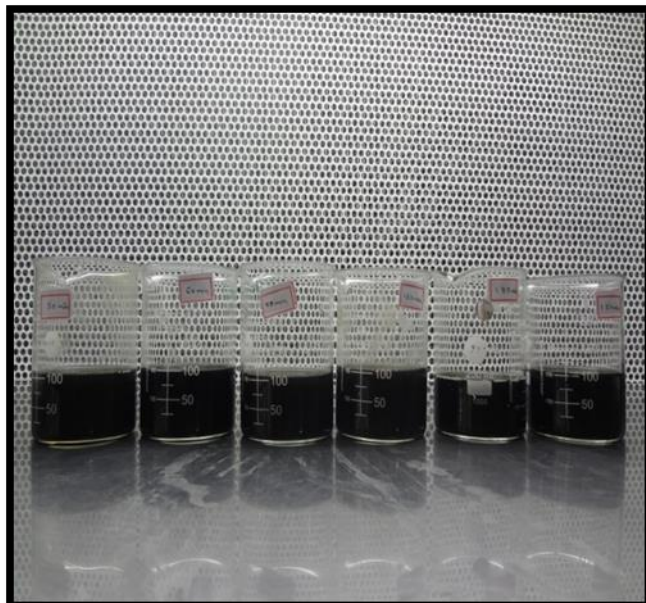
**Extract of Natural Dye**

## Plate 2

### Optimization of Dye Extraction - pH



**Ultrasonic Extraction**



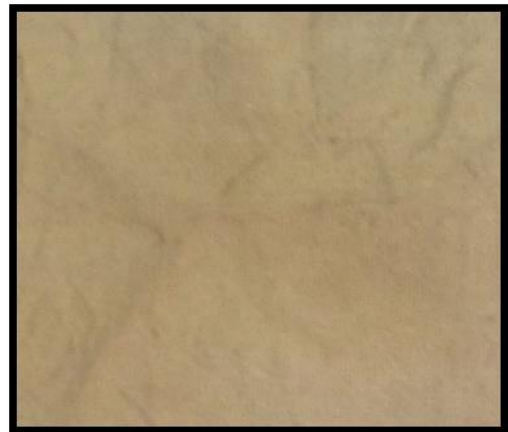
**Conventional Extraction**

**Plate 3**

**Selection of Mordants**



**Alum**



**Pomegranate**



**Myrobolan**



**Orange peel**

**Plate 4**

**Selection of Mordanting Technique**



**Pre- mordanting**



**Simultaneous mordanting**



**Post mordanting**

## 4. RESULTS AND DISCUSSION

The results of the present study are discussed under the following headings :

### 4.1 Optimization of various parameters for extraction of natural dye

#### 4.1.1 Solvent

#### 4.1.2 Effect of dye source concentration on extraction

#### 4.1.3 Effect of contact time on dye extraction

#### 4.1.4 Effect of pH on dye extraction

#### 4.1.5 Effect of temperature on dye extraction

### 4.2 Optimized conditions for dye extraction

### 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)

##### 4.3.2.4 Fabric strength (Weft)

##### 4.3.2.5 Fabric elongation (Warp)

##### 4.3.2.6 Fabric elongation (Weft)

##### 4.3.2.7 Fabric stiffness (Warp)

##### 4.3.2.8 Fabric stiffness (Weft)

##### 4.3.2.9 Crease recovery

##### 4.3.2.10 Drapability test

##### 4.3.2.11 Capillary rise test

##### 4.3.2.12 Sinking

##### 4.3.2.13 Drop test

### 4.4 Colour fastness test

### 4.5 Evaluation of UV protection property

## 4.1 OPTIMIZATION OF VARIOUS PARAMETERS FOR EXTRACTION OF NATURAL DYE

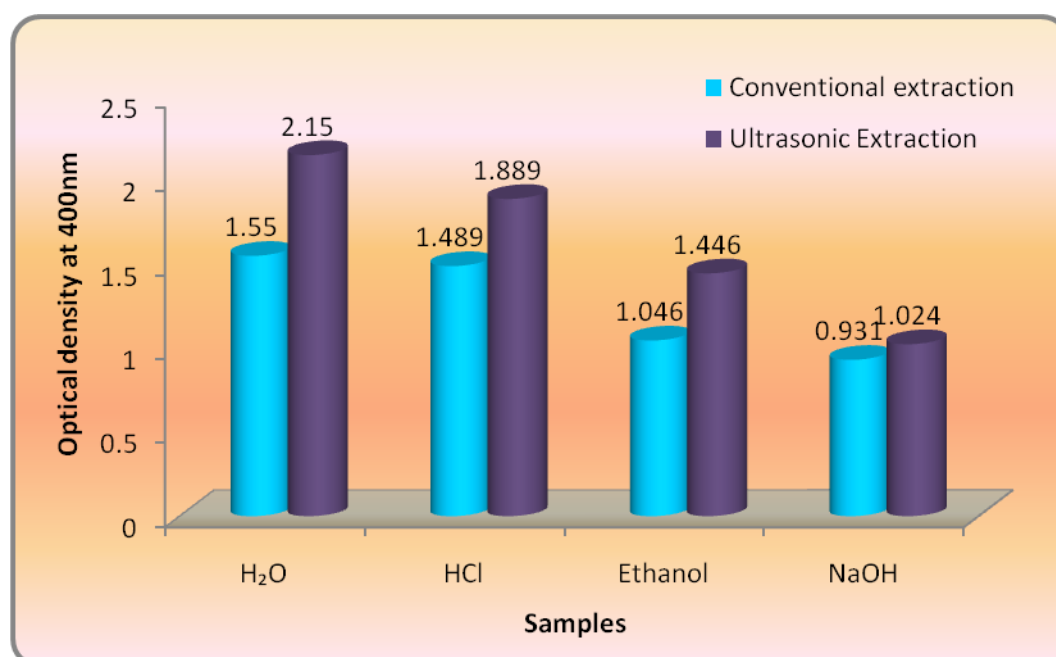
### 4.1.1 Solvent

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**

Solvent	Optical Density at 400nm	
	Conventional extraction	Ultrasonic Extraction
H <sub>2</sub> O	1.550	2.150
HCl	1.489	1.889
Ethanol	1.046	1.446
NaOH	0.931	1.024

**Figure 1**  
**Selection of Solvent**



Various solvent such as water, NaOH, ethanol and HCl were used separately as extraction medium and the colour intensity was measured. From Table 3 and Figure 1, it is clear that maximum colour yield was obtained with water as extraction medium, followed by HCl, ethanol and NaOH. Hence water was selected as extraction medium for the extraction of dye from *Hibiscuss hispidissimus*.

#### 4.1.2 Effect of dye source concentration on dye extraction

The effect of various concentration of dried *Hibiscuss hispidissimus* powder was studied and the results are presented in Table 4 and Figure 2.

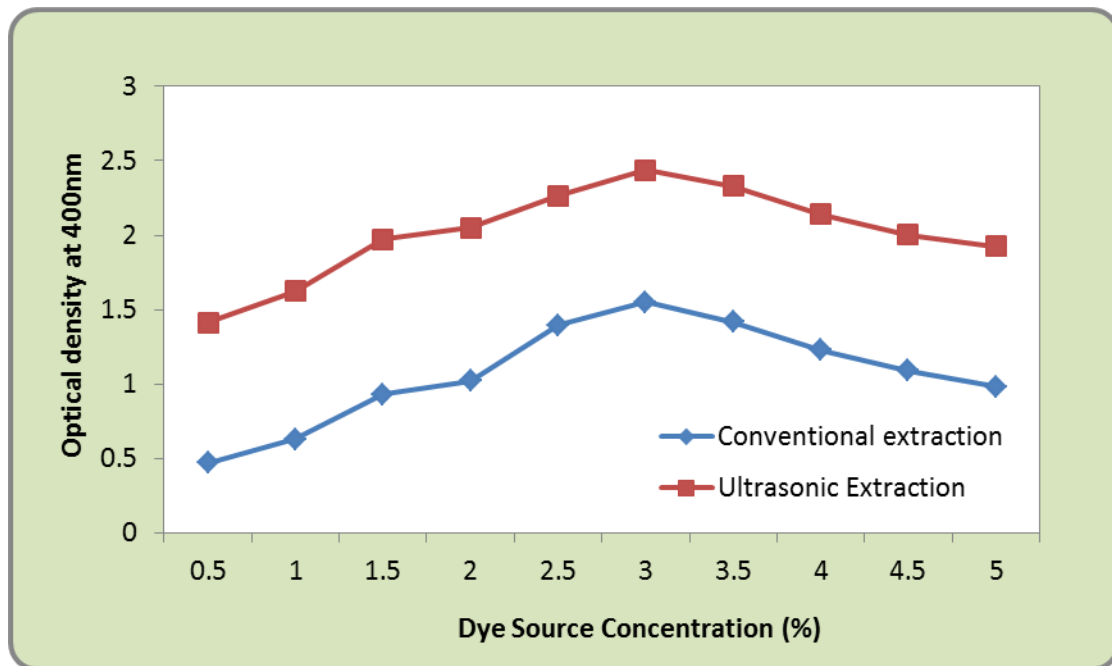
**Table 4**

**Effect of dye source concentration on dye extraction**

Dye Source Concentration (%)	Optical Density at 400nm	
	Conventional extraction	Ultrasonic Extraction
0.5	0.472	1.415
1	0.631	1.623
1.5	0.934	1.971
2	1.024	2.052
2.5	1.395	2.264
3	<b>1.550</b>	<b>2.437</b>
3.5	1.525	2.429
4	1.504	2.330
4.5	1.489	2.301
5	1.491	2.300

**Figure 2**

**Effect of dye source concentration on dye extraction**



From Table 4 and Figure 2, it is clear that the colour yield in conventional and ultrasonic method increases with increasing concentration of the natural colouring matter up to 3%. Above 3% there was no significant increase in colour yield in both the methods. Hence the optimum dye source concentration was fixed as 3% for both the methods. The colour achieved from the ultrasonic extraction method was higher than the colour yield achieved from the conventional method. The extra vibration and agitation conferred on the dye extraction system by use of the ultrasonic energy may account for this (Kamel *et al.*, 2009).

**4.1.3 Effect of contact time on dye extraction**

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

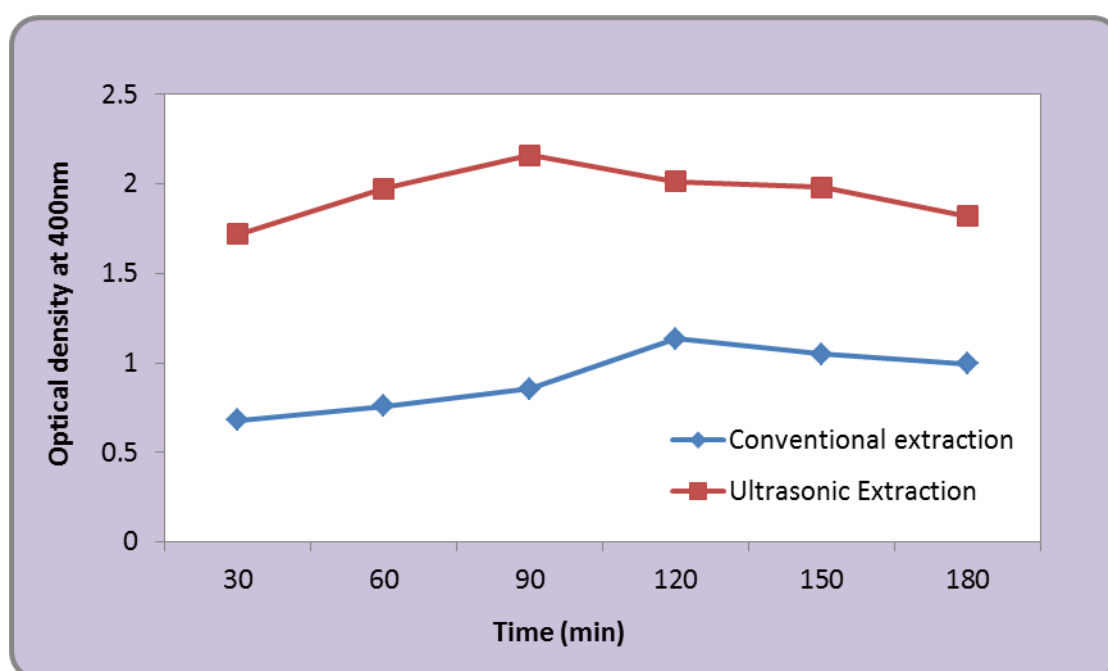
**Table 5**

**Effect of contact time on dye extraction**

Time (min)	Optical Density at 400nm	
	Conventional extraction	Ultrasonic Extraction
30	0.680	1.720
60	0.759	1.974
90	0.856	<b>2.162</b>
120	<b>1.134</b>	2.014
150	1.049	1.980
180	0.994	1.821

**Figure 3**

**Effect of contact time on dye extraction**



In extraction process time is very important parameter. From Table 5 and Fig.3 it is evident that the colour yield increases with increasing time in both the methods and reached maximum at 90 min for ultrasonic method and 120 min for conventional method. Hence 90 min for ultrasonic and 120 min for conventional method was selected as optimum time for dye extraction. When compared with the extraction methods, the colour yield is high in ultrasonic method as shown by an optical density of 2.162 than conventional method (1.134). The extraction yield decreased below or above the optimum time. This might be due to the constant heat for a long time results in the decomposition of the dye material while short time causes incomplete extraction (Prabhu *et al.*, 2002).

#### 4.1.4 Effect of pH on dye extraction

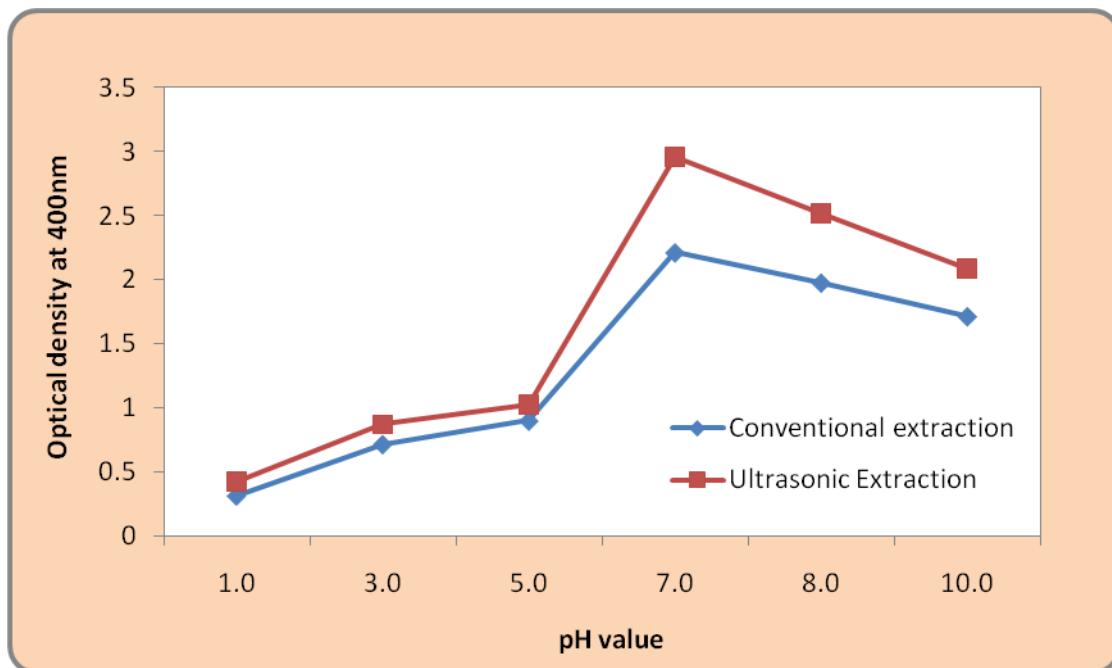
To determine the optimum pH for the effective extraction of natural dye, the extraction was carried out at different pH (1-10 ) and results are shown in Table 6 and Figure 4.

**Table 6**  
**Effect of pH on dye extraction**

pH	Optical Density at 400nm	
	Conventional extraction	Ultrasonic Extraction
1	0.312	0.427
3	0.714	0.875
5	0.898	1.029
7	<b>2.214</b>	<b>2.961</b>
8	1.978	2.521
10	1.715	2.091

**Figure 4**

**Effect of pH on dye extraction**



The pH of extraction solution is an important extraction process parameter because it can exert great impact on the stability of the natural dyes (Zhou *et al.*, 2014). From Table 6 it is clear that the maximum dye yield was achieved at pH 7 for both conventional and ultrasonic dye extraction methods. The dye extraction increases with increase in pH and reaches maximum at pH 7 and then decreases with an increase in pH above 7 in both the methods. Hence the initial pH of the extraction medium was maintained at 7 for both methods.

**4.1.5 Effect of temperature on dye extraction**

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**

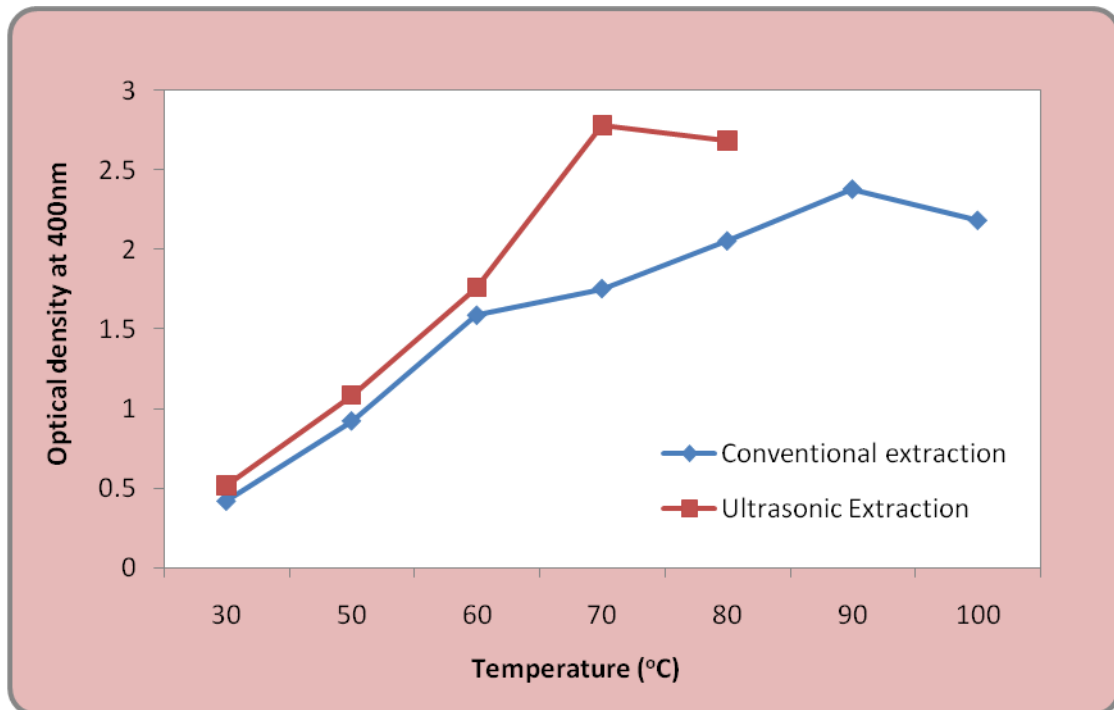
**Effect of temperature on dye extraction**

Temperature (°C)	Optical Density at 400nm	
	Conventional extraction	Ultrasonic Extraction
40	0.421	0.521
50	0.925	1.089
60	1.589	1.766
70	1.756	<b>2.786</b>
80	2.056	2.690
90	<b>2.381</b>	NA
100	2.185	NA

NA – Not Applicable

**Figure 5**

**Effect of temperature on dye extraction**



Temperature has greater influence on pigment extraction as pigments are temperature sensitive. Thus, the temperature was considered as a factor for the extraction of pigment. Temperature influences the extraction yields in both

positive and negative (Yang and Zhai, 2010). From Table 7 and Figure 5, it is clear that the colour yield increases with increase in extraction temperature in both ultrasonic and conventional methods with pronounced increase at 60°C in ultrasonic method and 90°C for conventional method. The present study result is in concurrent with Guesmiet *al*(2013) who have reported that the ultrasonic method produced a higher depth shades at a lower extraction temperature. The improvement is due to the acoustic cavitation phenomenon. Maran *et al.* (2015) have reported an optimum temperature of 55°C for the extraction of natural pigments from *Bougainvillea glabra* flowers.

#### 4.2 OPTIMIZED CONDITIONS FOR DYE EXTRACTION

Optimized conditions for the extraction of dye from *Hibiscus hispidissimus* are presented in Table 8.

**Table 8**

**Optimized Conditions for Dye Extraction**

Parameters	Optimized conditions	
	Conventional extraction	Ultrasonic extraction
Solvent	Water	Water
Time	120 min	90 min
Temperature	90°C	70°C
Concentration	3%	3%
pH	7	7

The dye was extracted under optimized conditions. The natural dye was extracted from dried *Hibiscus hispidissimus* powder using water as solvent at 90°C for 120 min for conventional extraction and at 70°C for 90 min for ultrasonic extraction. The extract was filtered and used as dye source.

### 4.3 EVALUATION OF DYED FABRIC

#### 4.3.1 Subjective Evaluation – Visual Inspection

Subjective evaluation of conventionally dyed and ultrasonic dyed samples was carried out 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	7	3	-	8	2	0	7	3
2.	UDS	9	1	-	8	2	0	9	1

From Table 9, it is clear that the conventionally dyed sample was rated as excellent in general appearance by 70 per cent of the judges whereas UDS 90% of the judges rated UDS as excellent in general appearance. With regard to brightness of shade both UDS and CDS was rated as bright by 80 per cent of judges. With respect to evenness of dyeing for CDS 70% of the judges and for UDS 90% of the judges rated as evenly dyed.

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

#### 4.3.2 Objective Evaluation

##### 4.3.2.1 Fabric Weight

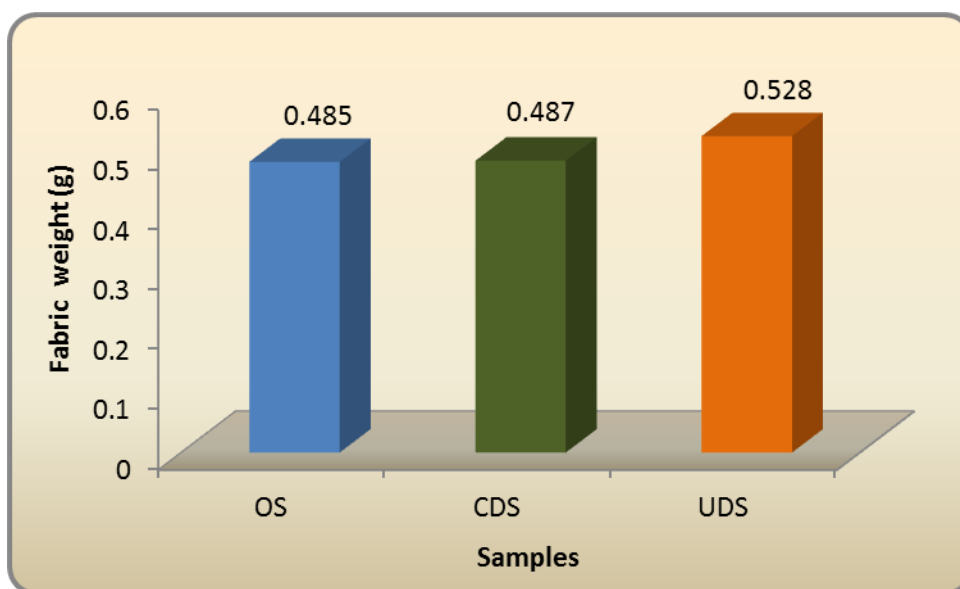
The fabric weight and analysis of variance of the samples OS, CDS, and UDS are given in Table 10 and Figure 6.

**Table 10**  
**Fabric weight**

S.No	Sample	Mean value (g)	Loss or gain over original	% loss or gain over original	'F' Test
1	OS	0.485			4930.11**
2	CDS	0.487	0.002	0.41	
3	UDS	0.528	0.043	8.86	

\*\* - Significant at 1% ( $p < 0.01$ )

**Figure 6**  
**Fabric weight**



From 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 ultrasonic dyed sample. The result indicates that the dye absorption is maximum in fabric dyed with dye solution extracted by ultrasonic method. There is a significant different at 1% level when compared between the samples.

#### **4.3.2.2 Fabric Thickness**

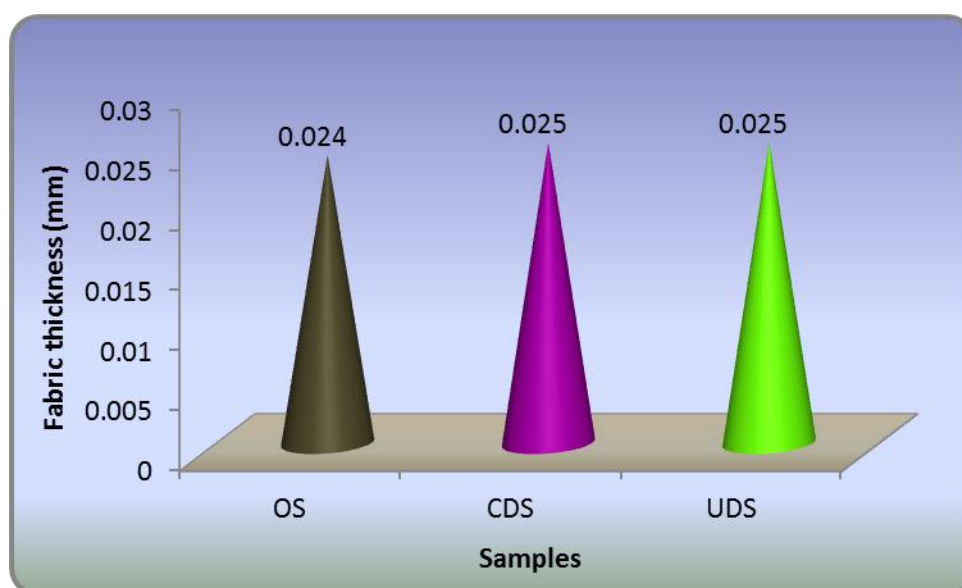
The fabric thickness and analysis of variance of the samples OS, CDS and UDS are shown in Table 11 and Figure 7.

**Table 11**  
**Fabric thickness**

S.No	Sample	Mean value (mm)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	0.024			0.782 <sup>ns</sup>
2	CDS	0.025	0.001	4.16	
3	UDS	0.025	0.001	4.16	

ns – Not significant

**Figure 7**  
**Fabric thickness**



From Table 11 and Figure 7, it is clear that the thickness of sample CDS and PDS increased when compared to original. The increase in fabric thickness was similar for both CDS and UDS. The increase in fabric thickness when compared with the original was found to be not significant.

#### 4.3.2.3 Fabric Strength (Warp)

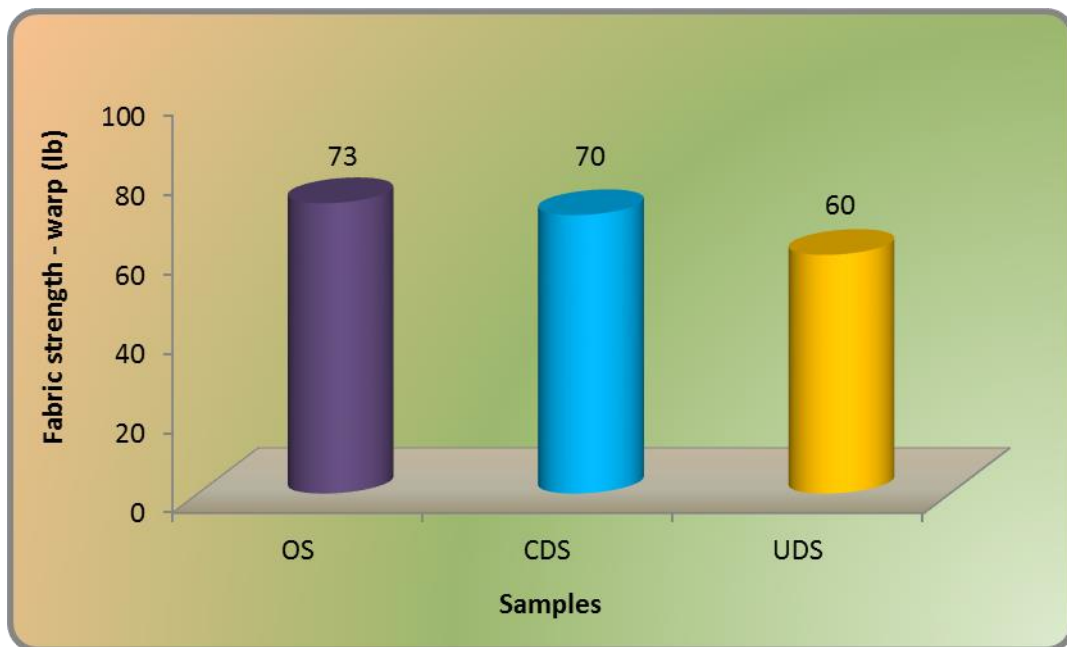
Fabric strength and analysis of variance of the samples OS, CDS and UDS in warp direction is presented in Table 12 and Figure 8.

**Table 12**  
**Fabric strength (Warp)**

S.No	Sample	Mean value (lb)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	73			198.571**
2	CDS	70	3	4.10	
3	UDS	60	13	17.8	

\*\* - Significant at 1% level

**Figure 8**  
**Fabric strength (Warp)**



From Table 12 and Figure 8, it is clear that the fabric strength of sample CDS and UDS decreased when compared to original. There is a significant difference at 1% level when compared between the samples.

#### 4.3.2.4 Fabric Strength (Weft)

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

**Table 13**

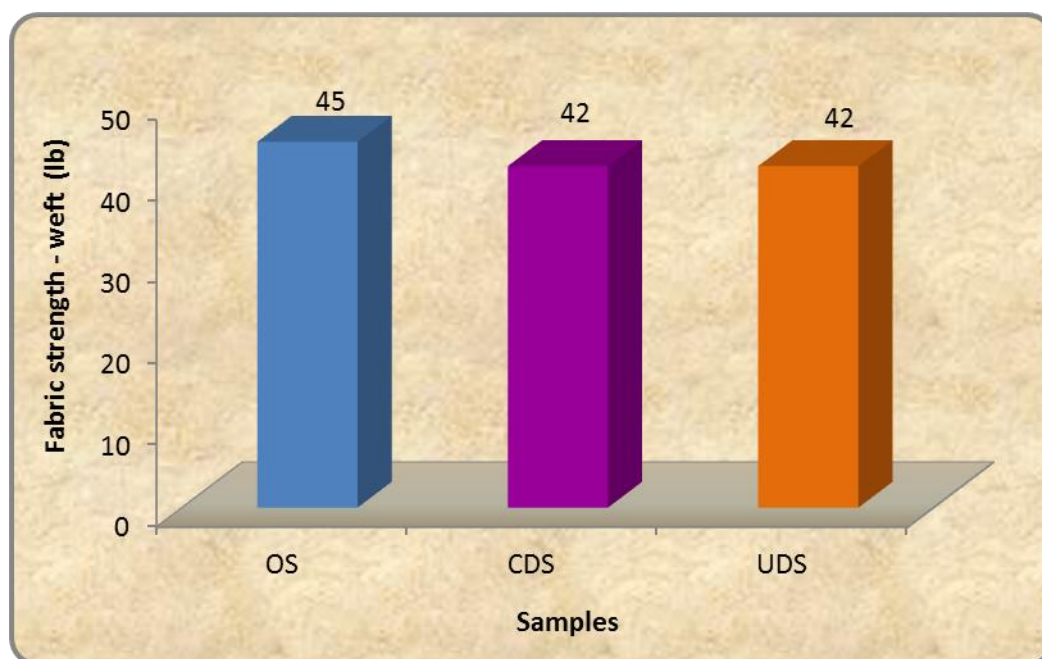
**Fabric strength (Weft)**

S.No	Sample	Mean value (lb)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	45			30.250**
2	CDS	42	3	6.66	
3	UDS	42	3	6.66	

\*\* - Significant at 1% level

**Figure 9**

**Fabric strength (Weft)**



From Table 13 and Figure 9, it is clear that the fabric strength of sample CDS and UDS decreased when compared to original. The decrease in fabric strength was similar both CDS and UDS (6.66%). There was a significant difference at 1% level when compared with the original sample.

**4.3.2.5 Fabric Elongation (Warp)**

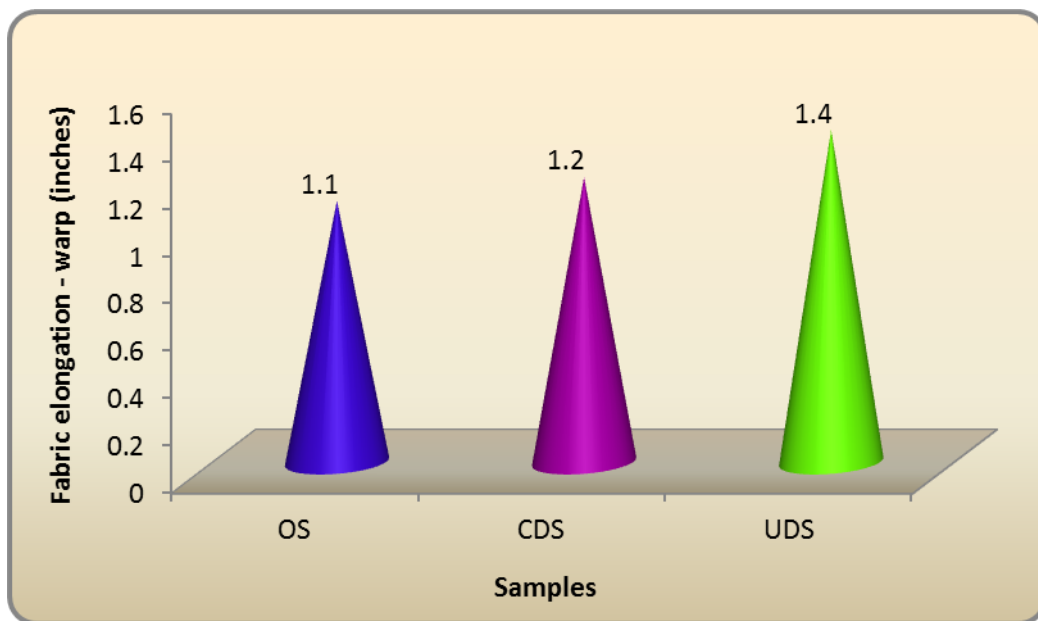
Fabric elongation and analysis of variance of the samples OS, CDS and UDS in warp direction are shown in Table 14 and Figure 10.

**Table 14**  
**Fabric elongation (Warp)**

S.No	Sample	Mean value (inches)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	1.1			9.243**
2	CDS	1.2	0.1	9.09	
3	UDS	1.4	0.3	27.27	

\*\* - Significant at 1% level

**Figure 10**  
**Fabric elongation (Warp)**



From Table 14 and Figure 10, it is clear that the elongation of CDS and UDS sample increased when compared to original. The increase in fabric elongation was maximum in sample dyed with ultrasonic method. The increase in elongation was significant at 1% level when compared between the samples.

#### 4.3.2.6 Fabric Elongation (Weft)

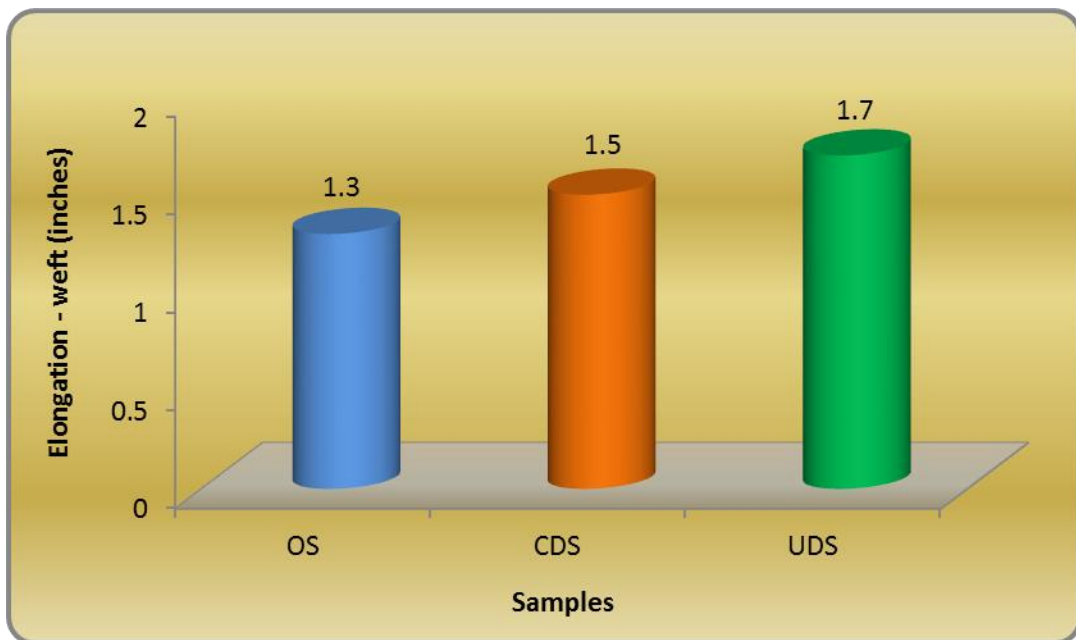
Fabric elongation and analysis of variance of the samples OS, CDS and UDS in weft direction is shown in Table 15 and Figure 11.

**Table 15**  
**Fabric elongation (weft)**

S.No	Sample	Mean value (inches)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	1.3			24.636**
2	CDS	1.5	0.2	15.38	
3	UDS	1.7	0.4	30.76	

\*\* - Significant at 1% level

**Figure 11**  
**Fabric elongation (Weft)**



From Table 15 and Figure 11, it is clear that the fabric elongation of sample CDS and UDS increased when compared to original. The increase in fabric elongation was found to be maximum in sample dyed with ultrasonic method. The obtained result proves that dye up take was higher in ultrasonic method than conventional method. There was a significant difference in elongation at 1% level when compared between the samples.

#### 4.3.2.7 Fabric Stiffness (Warp)

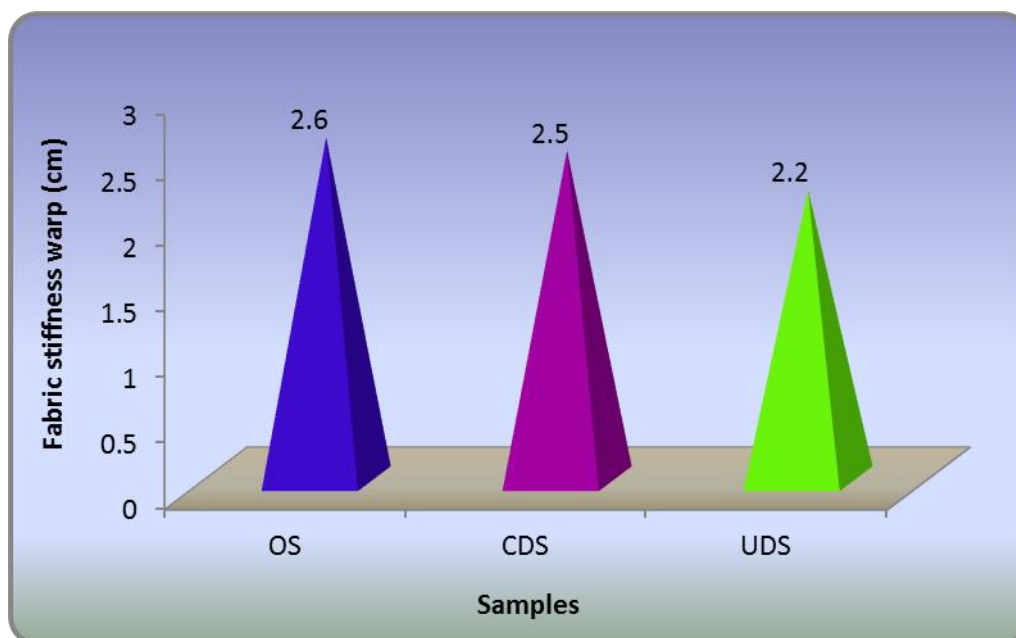
Fabric stiffness and analysis of variance of the samples OS, CDS and PDS in warp direction are shown in Table 16 and Figure 12.

**Table 16**  
**Fabric stiffness (warp)**

S.No	Sample	Mean value (cm)	Loss or gain over original	% loss or gain over original	'F' Value
1	OS	2.6			8.5079**
2	CDS	2.5	0.1	3.84	
3	UDS	2.2	0.4	15.3	

\*\* - Significant at 1% level

**Figure 12**  
**Fabric stiffness (warp)**



From Table 16 and Figure 12, it is clear that the stiffness of samples CDS and UDS decreased when compared with original. The decrease in fabric stiffness was found to be maximum in sample dyed by ultrasonic method (15.3%). There is a significant difference in stiffness at 1% level when compared between the samples.

#### 4.3.2.8 Fabric Stiffness (Weft)

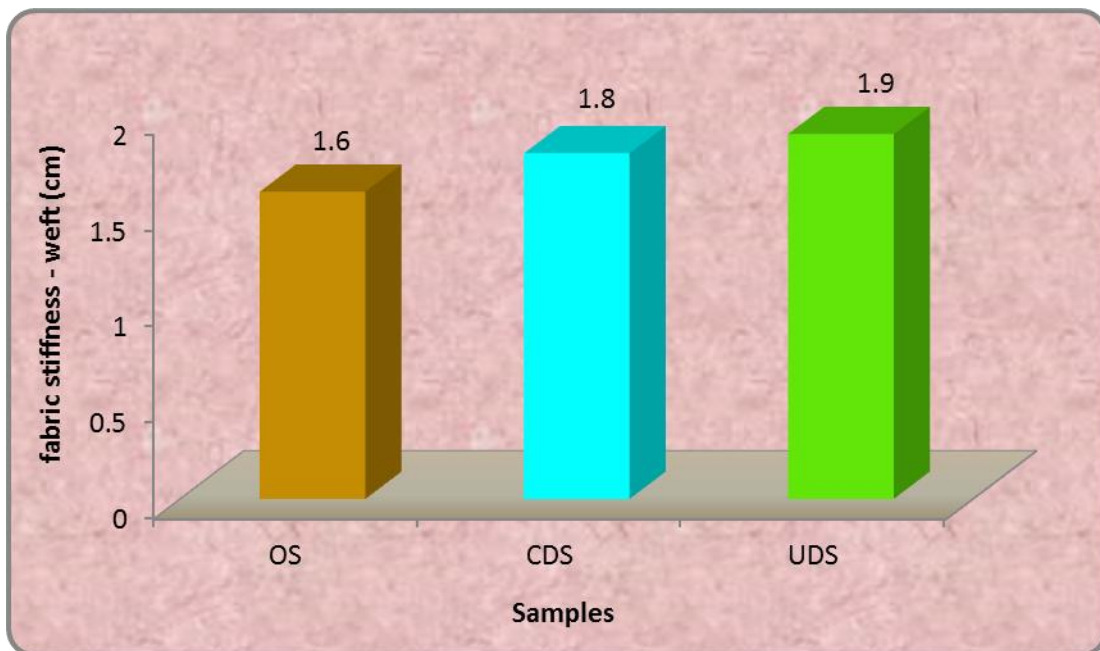
Fabric stiffness and the analysis of variance of the samples OS, CDS and UDS in weft direction are presented in Table 17 and Figure 13

**Table 17**  
**Fabric stiffness (Weft)**

S.No	Sample	Mean value (cm)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	1.6			9.1765**
2	CDS	1.8	0.2	12.5	
3	UDS	1.9	0.3	18.75	

\*\* - Significant at 1% level

**Figure 13**  
**Fabric stiffness (Weft)**



From Table 15 and Figure 13, it is clear that the fabric stiffness of sample CDS and UDS increased when compared to original. The increase in fabric stiffness was higher in UDS sample than when compared to conventional dyed sample. Statistical analysis proved that there is a significant difference at 1% level when compared between the samples.

#### 4.3.2.9 Crease recovery

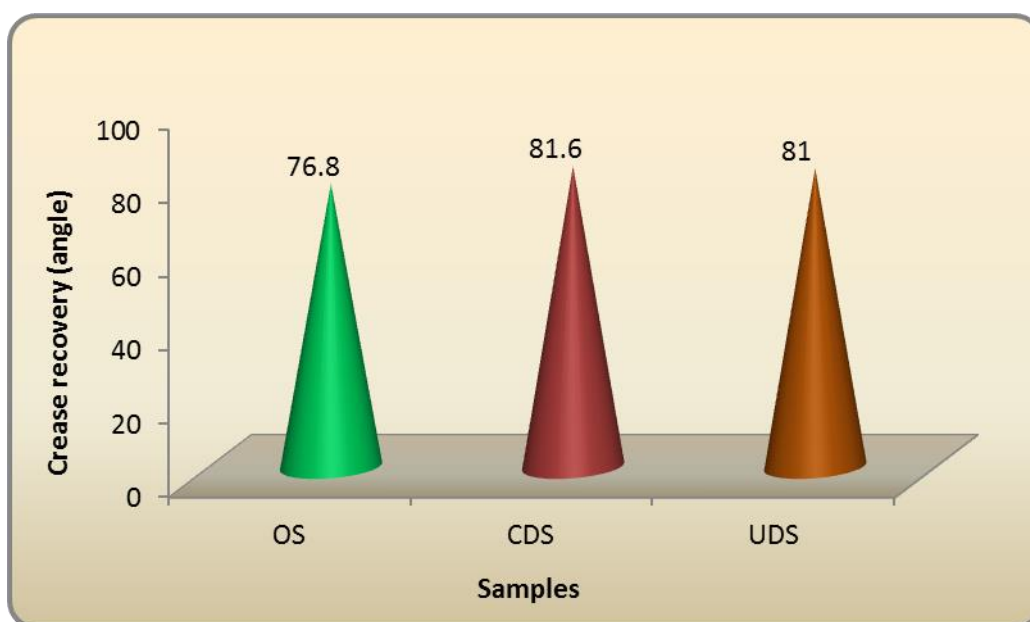
Crease recovery and the analysis of variance of the samples OS, CDS and UDS are shown in Table 18 and Figure 14.

**Table 18**  
**Crease recovery**

S.No	Sample	Mean value (angle)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	76.8			4.366*
2	CDS	81.6	4.8	6.25	
3	UDS	81	4.2	5.46	

\* - Significant at 5% level

**Figure 14**  
**Crease recovery**



From Table 18 and Figure 14, it is clear that the crease recovery of sample CDS and UDS increased when compared to original. The increase in fabric crease recovery was maximum in sample dyed with conventional method. Statistical analysis proved significant difference at 1% level when compared between the samples.

#### 4.3.2.10 Drapability Test

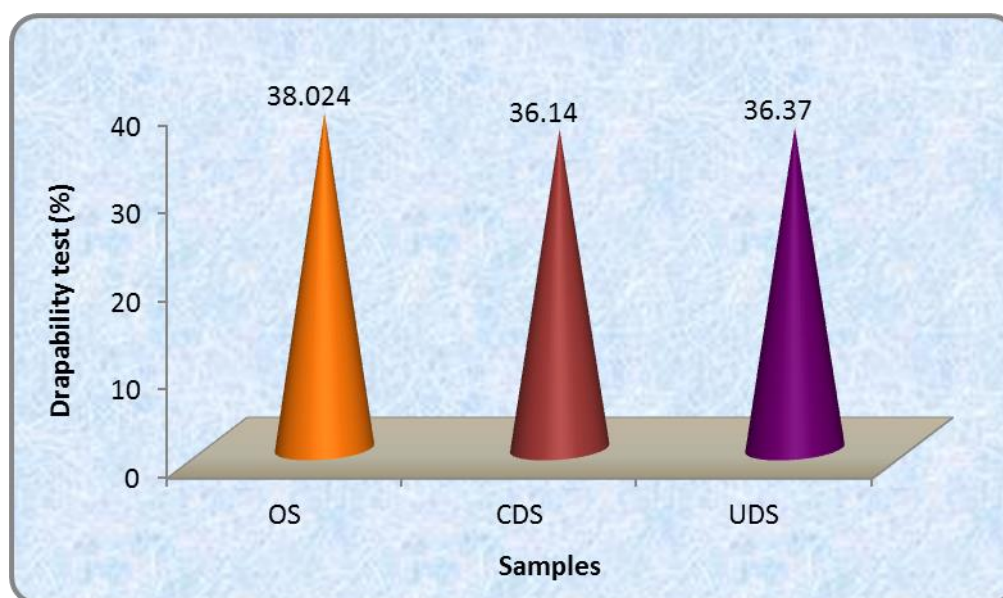
Drapability test and analysis of variance of the sample OS, CDS and PDS are shown in Table 19 and Figure 15.

**Table 19**  
**Drapability**

S.No	Sample	Mean value (%)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	38.02			69.5889**
2	CDS	36.14	1.884	4.95	
3	UDS	36.37	1.648	4.33	

\*\* - Significant at 1% level

**Figure 15**  
**Drapability**



From Table 19 and Figure 15, it is clear that the drapability of samples CDS and UDS has increased when compared to original. There is a significant difference in drapability at 1% level when compared between the samples.

#### 4.3.2.11 Capillary Rise Test

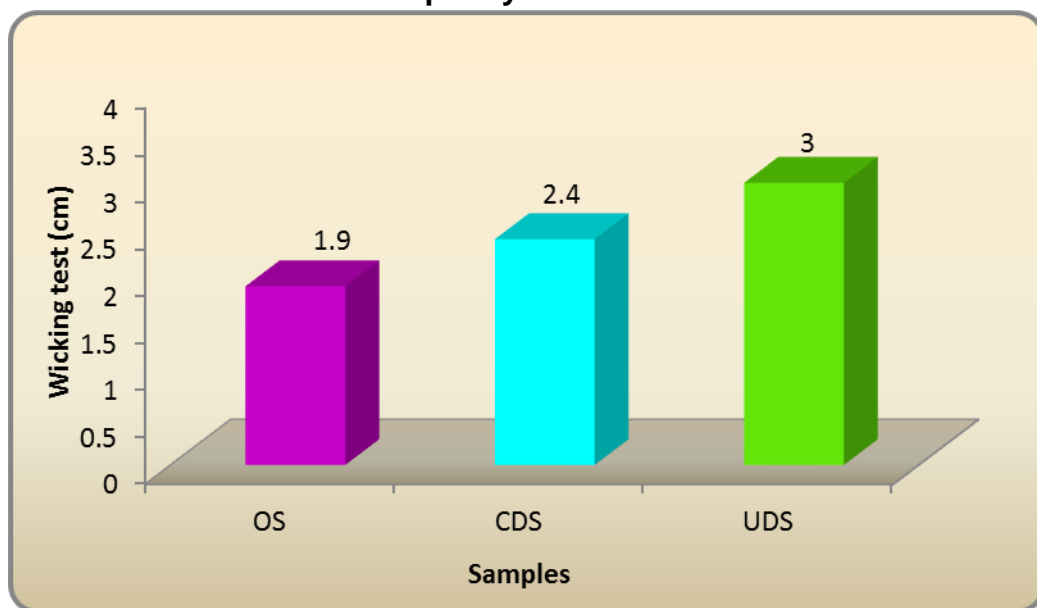
Wicking test and the analysis of the variance of the sample OS, CDS and UDS are shown in Table 20 and Figure 16.

**Table 20**  
**Capillary Rise Test**

S.No	Sample	Mean value (cm)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	1.9			100.468**
2	CDS	2.4	0.5	26.31	
3	UDS	3	1.1	57.8	

\*\* - Significant at 1% level

**Figure 16**  
**Capillary Rise Test**



From Table 20 and Figure16, it is clear that the absorbency increased for both CDS and UDS when compared to original. There was a significant difference in absorption at 1% level between the conventional and ultrasonic dyed samples.

#### 4.3.2.12 Sinking Test

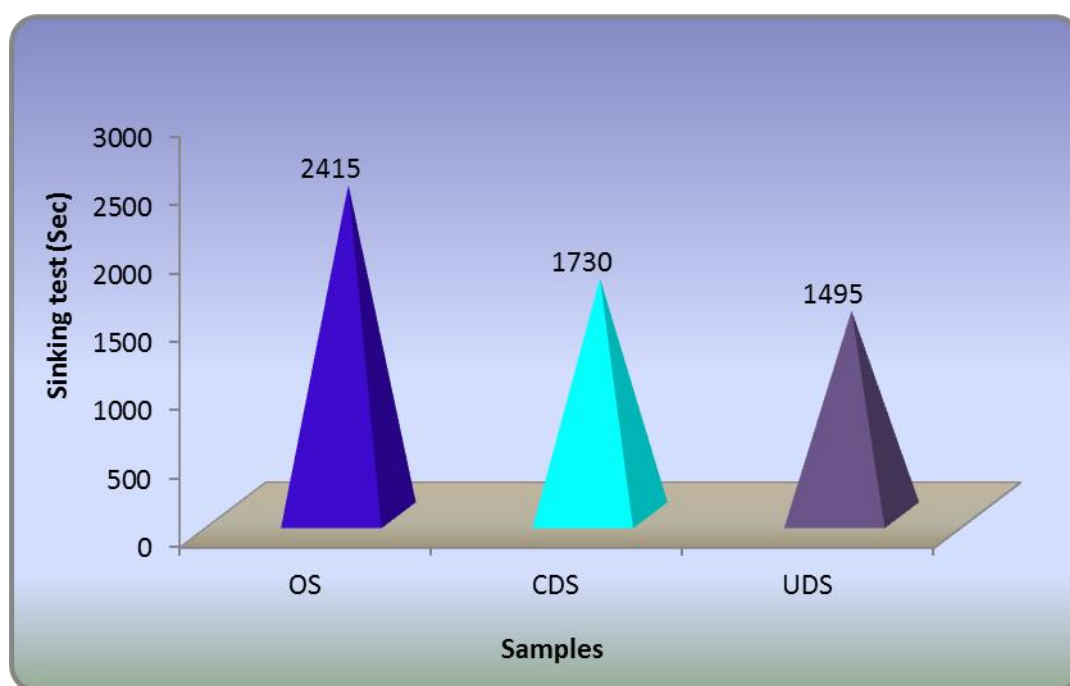
Sinking test and the analysis of the variance of the sample OS, CDS and UDS are shown in Table 21 and Figure 17.

**Table 21**  
**Sinking test**

S.No	Sample	Mean value (sec)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	2415			345.302**
2	CDS	1730	685	28.36	
3	UDS	1495	920	38.09	

\*\* - Significant at 1% level

**Figure 17**  
**Sinking Test**



From Table 21 and Figure17, it is clear that the time taken by samples CDS and UDS to sink is decreased when compared with the original. The results indicate that the absorbency of the CDS and UDS were increased than the original. There was a significant difference in absorption at 1% level between the conventional and ultrasonic methods.

#### 4.3.2.13 Drop Test

Drop test and the analysis of the variance of the samples OS, CDS and PDS are shown in Table 22 and Figure18.

**Table 22**

**Drop test**

S.No	Sample	Mean value (sec)	Loss or gain over original	% loss or gain over original	'F' value
1	OS	26.35			147.03**
2	CDS	22.78	3.57	13.54	
3	UDS	15.43	10.91	41.42	

\*\* - Significant at 1% level

**Figure 18**

**Drop test**

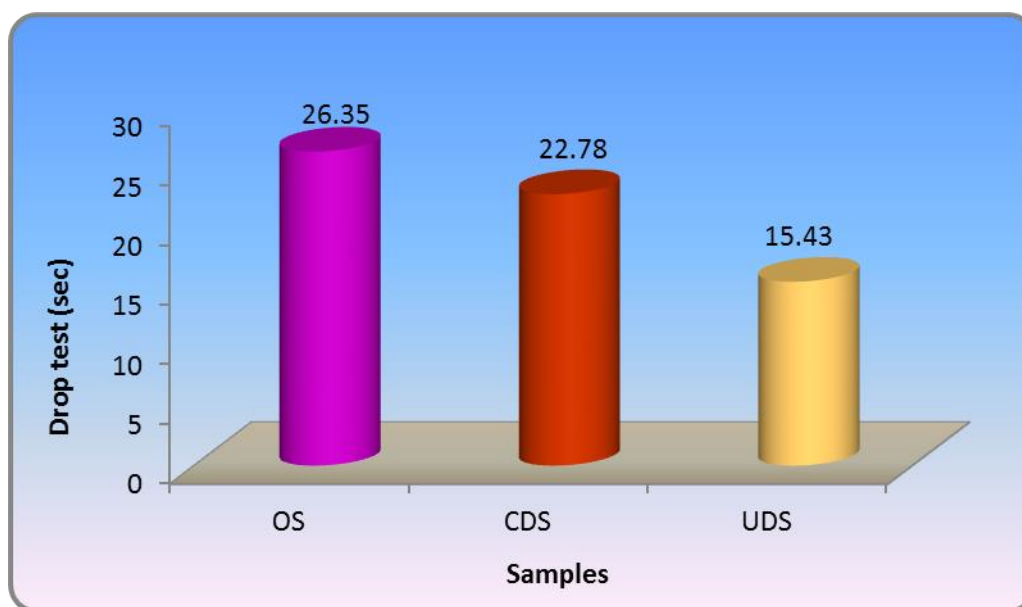


Table 22 and Figure 18 indicates that the absorbency of the dyed samples increased when compared over the original. Maximum increase in absorbency was noted in UDS sample. Statistical analysis proved that there is a significant difference at 1% level when compared between the samples.

#### 4.4 COLOUR FASTNESS TEST

Colour fastness of the dyed samples to sunlight, crocking and washing were determined and the results are presented in Table 22.

**Table 23**

**Colour Fastness to Sunlight, Crocking and Washing**

S.No.	Sample	Sunlight		Washing		Crocking			
		Colour change	Staining	Colour change	Staining	Dry		Wet	
						Colour change	Staining	Colour change	Staining
1.	CDS	4	4	4	4	5	4	4	5
2.	UDS	5	4	5	5	5	5	5	4

5 – Excellent ; 4 – Very Good

From the 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 UDS samples, UDS showed excellent colour fastness to all the colour fastness tests.

#### 4.5 Evaluation of UV Protection property

The UV- A blocking, UV- B blocking, UPF values and protection class of CDS and UDS samples were presented in Table 24 and Appendix 3.

**Table 24****UPF Values and Protection Class of Silk**

S.No	Dyed Samples	UV- A Blocking (%)	UV-B Blocking (%)	UPF	UV Protection Class
1	CDS	94.16	94	15	Good
2	UDS	94.61	95.53	20	Good

The results of per cent transmittance (Appendix 3), showed that the CDS and UDS have low transmittance value. The UV transmittance of the CDS was in the range of about 5% to 8% in the UV- B band (290- 320 nm) and 8% to 12% in the UV- A band (320- 400 nm). For UDS the UV- B band is in the range of 3% to 5% and the UV-A band is in the range of 5%- 11%. It indicates that the resistance of the CDS to UV rays was poor, while the UV transmittance of the UDS appeared to be lower than 5% in the UV- B region. Generally the UV protection of the fabric is evaluated as good when the UV transmittance is less than 5% (Feng *et al.*, 2007).

It can be observed that UV-A blocking for CDS and UDS were 94.16% and 94.61% respectively. The UV-B blocking was found to be 94% for CDS and 95.53% for UDS. It shows that UV- B blocking of UDS was greater than CDS. The samples dyed using both the techniques showed UPF values between 15- 24, which can be rated as offering 'Good' UV protection. When compared between CDS and UDS, UDS exhibited higher UV protection than CDS.

## 5. SUMMARY AND CONCLUSION

Synthetic dyes are known to be a major source of environmental pollution and their excessive use has led serious health hazards and disturbances in eco-balance of nature. In the current scenario, the use of non-allergic, antimicrobial and eco-friendly dyes in textiles has attracted the attention of people throughout the world. Natural dyes extracted from plants, animals or minerals without chemical processing show better compatibility with environment due to their non-toxic nature and having renewability potential. These are particularly suitable for people with dermatological disorders and allergies to chemicals in synthetic components used for textile finishing. Despite so many benefits associated with the use of natural colourants, textile industries, particularly in developing or underdeveloped countries hesitate to use natural dyes due to their low colour strength, limited availability and high cost in these regions. Most of the natural colourants are commonly obtained from floral parts of plants.

Scientists working in the field of natural colourant technology are now focusing to improve extraction of colourant from plant material and its application onto surface modified fabric. The utilization of ultrasonic radiations in natural dye extraction has been the subject of many recent studies. Ultrasound can enhance a wide variety of physical and chemical process, mainly due to the cavitation effect in liquid mediums that enhances the better leaching of natural dye materials from plant cell wall membranes and mass transfer of dye extract to the solvent. Exploration of natural colourant sources has been the subject of many studies from last few decades. Despite all the efforts, scientist have explored about 500 plants out of hundreds of thousand species found on the earth. Keeping in view the above mentioned facts, ultrasonic extraction method of colourant from flowers of *Hibiscus hispidissimus* was studied.

Hence the present study **“Efficacy of Ultrasonic Assisted Dye Extraction from Hibiscus *hispidissimus* for UV Protection”** was carried out with the following objectives:

## Objectives

- To extract dye by aqueous and ultrasonic method
- To optimize different parameters for dye extraction and dyeing
- To assess the UV protection property of the dyed fabric.

## Methodology Adopted

- Natural dye was extracted from dried *Hibiscuss hispidissimus* powder by conventional and ultrasonic method.
- Optimization of dye extraction - Various parameters such as solvent, dye concentration, pH, temperature and Mass liquor ratio were optimized for the extraction of dye.
- Optimization of dyeing - Pilot study was carried out to select suitable mordant. Orange peel, pomegranate rind, myrobolan and alum, were used as mordant for the pilot study. Suitable mordant was selected based on the shade and depth of colour produced. Dyeing parameters such as time, dye concentration, mordanting techniques were optimized for effective dyeing of the fabric.
- Dyeing by conventional and ultrasonic method - The selected silk fabric was dyed at 90°C for 120 min, with material liquor ratio of 1:20 for conventional method and in ultrasonic method the silk fabric was dyed at 70°C for 90 min, with material liquor ratio of 1: 20.
- The dyed fabrics were evaluated both subjectively and objectively.
- The dyed fabric was assessed for UV- Protection property.

## FINDINGS

- Water was found to be the suitable solvent for dye extraction by both aqueous and ultrasonic method.
- Dried *Hibiscuss hispidissimus* powder at a concentration of 3 per cent was found to be the optimum concentration for both type of dye extraction.

- A contact time of 120 min for conventional method and 90 min for ultrasonic method resulted in maximum dye extraction
- Dye extraction was found to be maximum at 90°C for conventional method and 70°C for ultrasonic method
- The optimum pH for dye extraction was found to be 7 for both the methods
- The material liquor ratio selected for dye extraction was 1:20
- Dyeing time of 90 minutes resulted in maximum dye absorption by the fabrics in both the methods
- The maximum dye absorption by the fabric was found at a dyeing temperature of 90°C for conventional and 70°C for ultrasonic method
- The fabric mordanted with orange peel by pre-mordanting technique produced better shade
- The results of visual evaluation of dyed fabric revealed that the sample dyed using ultrasonic extract was rated excellent in appearance, brighter in shade and evenly dyed
- Fabric thickness was found to be increased in all the dyed samples
- Fabric strength was found to be decreased in samples dyed with conventional and ultrasonic methods
- Maximum increase in fabric elongation was noticed in UDS sample in both warp and weft directions.
- Increase in fabric weight was recorded in all the samples.
- UDS sample exhibited excellent colour fastness to sunlight, washing and crocking followed by CDS sample
- The silk fabric dyed by ultrasonic method was found to exhibit higher UV protection property than the fabric dyed by conventional method.

## CONCLUSION

Natural dyes provide an environmentally safe option for coloring of textile and other materials. It was found that the application of ultrasound can increase the extraction of dyes from different parts of various plant resources. Extraction was done using ultrasound as well as conventional methods and the extraction efficiency was compared. The reason for the improvement could be due to better leaching of natural dye material from plant cell membranes and mass transfer to solvent assisted by acoustic cavitation provided by ultrasound. This novel technique can be employed effectively for the extraction of coloring matter from various plant resources. This process provides effective utilization of natural resources as eco-friendly method in current situation of global environmental concern. In this study, it was observed that silk fabrics dyed with dried *Hibiscus hispidissimus* powder extract have “good UV protection” properties. The degree of protection imparted after dyeing 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 ultrasonic method were good to very good. Therefore, it can be concluded that Ultrasonic dyeing with *Hibiscus hispidissimus* extract can be useful in developing UV-protective silk fabrics.

### Scope for Further Study

- Natural dye from *Hibiscus hispidissimus* could be extracted using microwave radiation technique
- Enzymes could be exploited for the extraction of dye from *Hibiscus hispidissimus*
- Different mordant could be tried for dyeing

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**APPENDIX – 1**

**Original and Dyed Samples**



**Original Sample**



**Conventional dyed Sample**



**Ultrasonic Dyed Sample**



## APPENDIX - 3

### Labsphere Transmittance Analyzer UPF Report

Sample: Conventional Treated  
Description: Sample with green colour  
  
Operator: PSG  
Client: N.M.JINSIYA  
Comment: MSC - TT & FA AVINASHILINGAM UNIV  
Date: 3/12/2015 9:58:10 AM  
Unit serial number: 1014134754  
Solar Irradiance Profile: AATCC 183:2010  
Wavelength Range: 280-400 nm

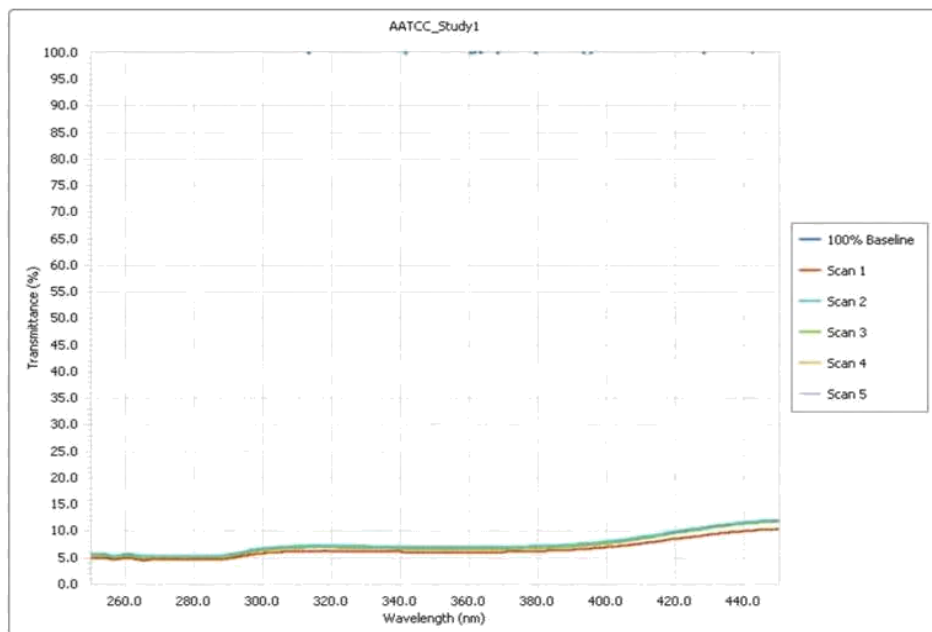
	UPF	T(UVA)	T(UVB)
Number of Scans:	5	5	5
Mean:	14.88	6.84%	6.00%
STD:	0.81	0.35%	0.30%
COV:	5.45%	5.17%	5.06%

UV-A Blocking 93.16%

UV-B Blocking 94.00%

UPF Rating: 15

Scan	UPF	Critical Wavelength
Scan 1	16.23	388
Scan 2	14.02	388
Scan 3	14.70	387
Scan 4	14.72	387
Scan 5	14.75	387



## Labsphere Transmittance Analyzer UPF Report

**Sample:** Ultrasonic Treated  
**Description:** Sample with green colour  
  
**Operator:** PSG  
**Client:** N.M.JINSIYA  
**Comment:** MSC - TT & FA AVINASHILINGAM UNIV  
**Date:** 3/12/2015 9:58:10 AM  
**Unit serial number:** 1014134754  
**Solar Irradiance Profile:** AATCC 183:2010  
**Wavelength Range:** 280-400 nm

	UPF	T(UVA)	T(UVB)
Number of Scans:	5	5	5
Mean:	19.84	5.39%	4.47%
STD:	2.08	0.55%	0.49%
COV:	10.50%	10.15%	10.96%

**UV-A Blocking:** 94.61%  
**UV-B Blocking:** 95.53%  
**UPF Rating:** 20

Scan	UPF	Critical Wavelength
Scan 6	19.90	387
Scan 7	17.76	387
Scan 8	17.80	387
Scan 9	21.37	387
Scan 10	22.39	387

