Effect of Sericin Treatment on Dyeability of Cotton Fabric Using Natural Dye

By
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CERTIFICATE – I

This is to certify that this thesis entitled, **"Effect of sericin treatment on dyeability of cotton fabric using natural dye"** submitted for the degree of **Master of Science**, in the subject of **'Textile and Apparel Designing'** to the CCS Haryana Agricultural University, is a bonafide research work carried out by **Babita Bhandari (**Admission No. **2012HS25M)** under my supervision and that no part of this dissertation has been submitted for any other degree.

The assistance and help received during the course of investigation have been fully acknowledged.

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CERTIFICATE – II

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CHAPTER-I

INTRODUCTION

Love for colour is a natural instinct and every individual has its own choice and liking for it. Colour is visual perceptual property corresponding in humans to the categories called red, yellow, blue and others. It is a sensation that arises from the activity of retina of the eye and results in a specific response to radiate energy of certain wavelength and intensity. Thus it is a quality of an object with respect to light. (Mizzarini *et al.*, 2002).

Dyes are the coloured substances which are capable of imparting colours to the matrix which may be fiber, paper or any object. They must have fixing tendency on a fabric that is impregnated with their solution and the fixed dyes must be fast to light as well as resistant to action of water, dilute acids, alkalies, various organic solvents used in dry cleaning, soap solutions, detergent etc. (Shukla, 1992).

Natural colourants derived from flora and fauna are believed to be safe because of their non-toxic, non-carcinogenic and biodegradable nature. The natural dyes are eco-friendly and user-friendly. These are obtained from the parts of the plants namely, leaves, fruits, flowers, seeds, bark, rind, roots, husk, nuts, shoots, insects and minerals etc. Natural dyes are known for their use in colouring of food substrate, leather, wood as well as natural fibers like wool, silk, cotton and flax as major areas of application since ancient times. Natural dyes produce a wide range of shades which are soothing and soft. Since the advent of widely available and cheaper synthetic dyes in 1856 having moderate to excellent colour fastness properties, the use of natural dyes having poor to moderate wash and light fastness had declined to a great extent. However, the health hazards associated with the use of synthetic dyes and also the increased environmental awareness have revived the use of natural dyes during the recent years (Samanta and Agarwal, 2009). India is still a major producer of most natural dyed textiles (Vankar, 2007). Production of synthetic dyes is dependent on petrochemical source and some of synthetic dyes contain toxic or carcinogenic amines which are not ecofriendly (Hunger, 2003). Moreover, the global consumption of textiles is estimated at around 30 million tons, which is expected to grow at the rate of three percent per annum. The colouration of this huge quantity of textiles needs around 700,000 tons of dyes which causes release of vast amount of unused and unfixed synthetic colourants into the environment. (Samanta and Agarwal, 2009).

As renewable raw materials for textile industry, natural dyes and fibers, especially cotton are gaining considerable importance due to recent environment conservation regulations. Cotton accounts for 40 percent of the total global fibre production and is the most important fibre in the world. Cotton as a crop as well as a commodity plays an important role in the agrarian and industrial activities of the nation and has a unique place in the economy of our country. Cotton popularly known as 'White Gold' is grown mainly for fiber. India has been a traditional home of cotton and cotton textiles. It provides livelihood to 60 million people who depend on cotton cultivation, processing, trade and textiles.

Dominance of cotton in textile industry is due to its varied advantages such as ability to withstand the rough laundering treatments especially under alkaline conditions, good perspiration absorption characteristics, comfort during wear and the ability to take up the wide range of dyestuffs which make cotton ideal for apparel use. Limitations of cotton as textile fibre includes poor colour fastness, proneness to microbial attack causing odour, discolouration, unhygienic conditions, flammability, low UV resistance, high quantity of salt requirement for certain important dyeing processes etc. Cotton can be modified by number of techniques to improve performance characteristics such as dye fixation, soil repellency, crease resistance and flame retardancy. Chemical modification of cotton will improve their dye ability but at the same time it will pollute the environment to greater level. Modification can also be possible with the help of biocatalysts (enzyme) and biopolymers like sericin in an environmentally benign route. Sericin is one of such biomaterials which can be effectively used in dyeing process.

Silk derived from the silkworm, *Bombyx mori* is composed of two major proteins, fibroin and sericin. Fibroin is a fibrous protein, present as a delicate twin thread linked by disulfide bonds, enveloped by successive sticky layers of sericin that help in the formation of a cocoon. Sericin or silk glue is a globular protein (Poza *et al*., 2002; Wu *et al*., 2007), that constitutes 25 to 30 percent of silk proteins and it also has some impurities such as waxes, fats and pigments. Sericin is yellow, brittle and inelastic substance. It consists of 18 amino acids (Takasu *et al*., 2002), most of which have strong polar side chains such as hydroxyl, carboxyl and amino groups (Zhang, 2002). Its high hydrophilicity arises from the high content of serine and aspartic acid, approximately 33.4 and 16.7 percent sericin, respectively (Tsubouchi *et al*., 2005; Zhang, 2002; Zhaorigetu *et al*., 2001).

Sericulture is an agro-industry, the end product of which is silk. Sericulture involves three activities viz., mulberry cultivation, silkworm rearing, reeling of the silk from the cocoons formed by the worms. The first two activities are basically agriculture in nature and the later is an industry of different financial investments. Sericin, the second major component of the cocoon is separated from fibroin by a degumming process and the sericin is mostly discarded in the wastewater. It is estimated that out of the 1 million tons (fresh weight) of cocoon production worldwide or about 400,000 tons of dry cocoon, approximately 50,000 tons of sericin could be recovered from the waste solution (Kim, 2007; Zhang, 2002). Sericin is at present an unutilized by-product of the textile industry and the discarded degumming wastewater also ultimately leads to environmental contamination due to the high oxygen demand for its degradation by microbes (Fabiani *et al*., 1996). Sericin could represent a significant source of profit for silk cultivators if they utilize it in textile processing or sell to the industry. Recovered sericin could be used as a 'value-added' product for many purposes and sericin-derived products (Vaithanomsat and Kitpreechavanich, 2008) which would also be beneficial in terms of the economy and the environment.

Sericin has been used for cosmetics, medical, polymer materials and other applications due to its antioxidative, antimicrobial, anti-wrinkle, wound healing, UV resistant, moisture-absorbing and desorbing properties etc. (Fabiani *et al*., 1996; Rigueiro *et al*., 2001; Shen *et al*., 1998; Wu *et al*., 2007).

With its unique properties, sericin can be used in the surface modification of fibers and fabrics. In fact, it is used as a coating material for cellulose fibers and the treated textiles exhibit a decrease in free formaldehyde content, resistance to electricity, skin irritation and allergic reactions with increased water retention and only a negligible decrease in the textile tensile strength. Sericin is also impregnated into polyester fabric to overcome polyester hydrophobicity and to improve UV absorption properties of the sericin treated fabrics. It would also significantly reduce the environmental impact of silk production processes (Fersi *et al*., 2005) and help sustainable development.

It is also used as a coating material for natural and artificial fibers which can prevent abrasive skin injuries, the development of rashes and antibacterial for the products such as diapers, diaper lines and wound dressing (Yamada *et al*., 1998; Sarovart *et al*., 2003). It has also been used as finishing agent for natural (Kongdee *et al*., 2005) or man-made textiles (Lee *et al*., 2004) with good results in terms of moisture absorption, softness, comfort and antistatic properties. But not much work has been done in India on natural dyes using sericin as treatment agent. Hence the work was planned to make the process of natural dyeing truly ecofriendly taking into consideration the growing environmental consciousness and to improve the dye uptake of cotton fabric by treating with sericin. The present study has been taken with the following objectives:

1. To optimize the conditions for sericin treatment and apply on cotton fabric.
2. To dye the treated fabric with natural dye and assess the colour fastness properties.
3. To study the physical properties of dyed fabric.

CHAPTER-II

REVIEW OF LITERATURE

The review of literature reveals that though ample work has been done on dyeing using natural dyes in India and abroad, but scanty work could be explored related to treatments given to fabric for improving dyeability of natural dyes. An attempt has been made here to present a brief resume of the available literature on the issues relevant to the present study under the following subheads.

2.1 General characteristics of cotton and sericin

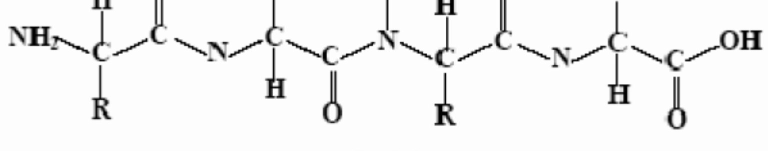
2.2 Effect of sericin treatment on dyeability and properties of textiles

2.3 Effect of different treatments on dyeability of cotton

2.1 General Characteristics of Cotton and Sericin

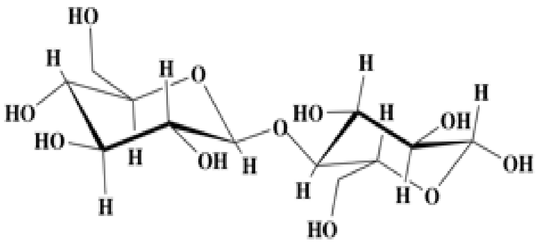
Hollen and Saddler, 1988 stated that the chemical reactivity of cellulose is related to three hydroxyl groups of the glucose unit. These groups readily react with moisture, dyes and many finishes. Cotton, a cellulosic fibre is medium strength fibre having the breaking tenacity of 3.5-4.0 g/d. It is stronger when wet. Cotton makes very comfortable contact with skin because of its high absorbency and its good heat conductivity. It has moisture regain of 7 percent and elastic recovery is moderate. It is harmed by acids and resistant to organic solvents hence can be safely dry-cleaned.

Zhang, 2002 reviewed that the application areas of sericin differ with respect to its molecular weight. It has been reported that low molecular weight sericin peptides (less than 20 kDa) or sericin hydrolysates are used in cosmetics including skincare and hair care products, health products and medications. On the other hand, high molecular weight sericin peptides (greater than 20 kDa) are mostly used as medical biomaterials, degradable biomaterials, compound polymers, functional bio-membranes, hydrogels and functional fibers and fabrics. Sericin can be cross-linked, copolymerized and blended with other macromolecular materials for production of materials with improved properties, especially artificial polymers. It is also used as an improving reagent or a coating material for natural and artificial fibers, fabrics and articles.



Sericin

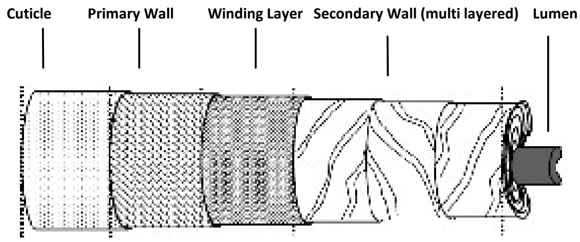
Anonymous, 2003 reported that after scouring and bleaching, cotton is 99% cellulose. Cellulose a macromolecule is a polymer made up of long chain of glucose molecules linked by C-1 to C-4 oxygen bridges with elimination of water (glycoside bonds). The anhydroglucose units are linked together as beta-cellobiose; therefore, anhydro-beta-cellobiose is the repeating unit of the polymer chain. The cellulose chains within cotton fibers tend to be held in place by hydrogen bonding. These hydrogen bonds occur between the hydroxyl groups of adjacent molecules and are most prevalent between the parallel, closely packed molecules in the crystalline areas of the fiber. The three hydroxyl groups, one primary and two secondary, in each repeating cellobiose unit of cellulose are chemically reactive. These groups can undergo substitution reactions in procedures designed to modify the cellulose fibers or in the application of dyes and finishes for crosslinking. The hydroxyl groups also serve as principal sorption sites for water molecules.



Cellobiose Repeat Unit

Brahma, 2006 noticed that sericin is a macromolecular protein created by silkworms in the production of silk and constitutes 25-30 % of silk protein; the sericin molecular weights range from 30,000 to 300,000 Da. This protein that cements the two fibroin filaments is removed during degumming process of raw silk production. The sericin recovered from the degumming liquor finds applications in creams, shampoos and as moisturizing agents. Recently, it has been found that poly ethylene terepthaline fabrics treated with 4 percent sericin (w/v) showed 51 percent reduction of *Proteus vulgaris* and 38 percent reduction of *S. aureus*.

Chettra, 2006 envisaged that raw cotton consists of 90-95% cellulose, but also consists of waxes, pectins and other plant material. It is these minor constituents which are removed during mercerization to give a soft, clean, white and absorbent fibre. The cuticle layer in cotton fibre is impermeable to water and aqueous solutions, therefore it is removed during processing to aid the uptake of solutions and dyes.



Layered structure of cotton fibre

Anghileri ***et al.,*** 2007 determined chemical properties of oxidized sericin by amino acid analysis. The amino acidic pattern of sericin is dominated by the presence of hydroxyl (serine, threonine, tyrosine), acidic (aspartic acid, glutamic acid) and basic (lysine, histidine, arginine) amino acid residues, which totally account for about 73 %. Glycine and alanine are minor components with a total concentration of only 20 %. Other amino acids (proline, methionine, isoleucine, leucine, phylalanine, cysteine, valine and tryptophane) are present in very small amounts. It was concluded that sericin, either obtained directly from the cocoons or recovered from degumming wastewater can be considered as a valuable natural polymer worth of being used for a wide range of applications, including those related to biomaterials.

Wu ***et al.,*** 2008 visualized that in sericin the amount of the hydrophilic amino acids was up to 76% which indicated water absorbability and good solubility. Some of the amino acid residues of sericin macromolecules have polar side groups whereas others have non-polar side groups.

Kurlageri, 2009 stressed that cotton, being famous for its purity and user friendly properties is the principle clothing material of the world. It is universally known that this fiber absorbs and releases perspiration quickly thus keeping the body temperature cool; being cellulose by source possesses the most advantageous characteristics like excellent wicking, good conductor, graceful drapability and user friendly.

Aramwit ***et al.,*** 2012 considered that sericin is a mixture of proteins with different molecular properties. It exists in a wide range of molecular weights, from 10 to over 400 kDa, depending on the extraction methods, temperature, pH and processing time. Sericin with a low molecular weight, commonly less than 20 kDa is soluble in cold water and can be recovered during the early stages of raw silk production, whereas higher molecular weight sericin is soluble in hot water and can be obtained from the later stages.

2.2 Effect of Sericin Treatment on Dyeability and Properties of Textiles

There are many approaches for increasing colour yield during dyeing. These could be simple changes to process parameters such as temperature and time, pretreatment of the substrate and addition of auxiliaries. The process gains popularity when the increase in colour yield is brought about by using a byproduct or waste product like sericin.

Xie ***et al.,*** 2002 coated sericin on polyester fabrics using epoxy compounds as crosslinking and some properties of the fabrics were measured. The results showed that sericin can be evenly coated on the polyester fabrics after being treated by base deweighting finishing. The absorbent quality of the coated fabrics improved greatly, moisture regain increased 95.24% and air permeability rose 24.79%.

Kongdee ***et al.,*** 2005 noticed the effect of silk sericin on cellulose fiber surface. The sericin was coated onto the cotton surface as a film. It was found that increasing sericin content in the finishing solution increased the amount of coated sericin, gave greater depth of colour and reduced free formaldehyde content in treated samples. The sericin content was found to have a negligible influence on tensile strength and crease recovery angle. With increasing sericin content, electrical resistivity of the samples dramatically decreased and water retention increased, indicating that sericin-treated fabrics may be comfortable to wear because of its maintenance of moisture balance with respect to human skin.

Wang *et al.,* 2006 studied the influence of the sericin modification on the dyeing performance of wool fiber cross-linked by DE agent on Lanasol reactive dyes and Lanaset dyes. The results indicated that the dyeing ability of the sericin cross-linked wool fiber improved obviously indicating that the dyeing temperature decreased, the dye-uptake increased, the washing fastness improved and the effect was better than that of wool fiber modified by DE cross linking agent only.

Kongdee and Chinthawan, 2007 investigated the modification of cotton fibre with silk sericin in a pad-dry-cure method. By using glutaraldehyde and dimethylol-dihydroxy-ethylene urea as crosslinking agents, sericin was successfully bound to cotton fibers as seen from infra-red absorption band at 1515, 1546 and 1687 cm^{-1}which corresponded to –NH and –C=O of sericin respectively and the band at 1747, 1789 and 1750 cm^{-1} indicated –C=O of the regenerated functional groups. The examination of the modified fibers by scanning electron microscope showed round fibers and deposited sericin on cotton fiber surfaces. The modification of cotton fibers with sericin using these crosslinking agents was clearly proved by dyeing with an acid dye. In sample treated with sericin, the increase in the colour strength, b-values and the decrease in L-values were the proof of the reaction between cellulose and sericin.

Gulrajani *et al*., 2008 developed a process for the recovery and production of sericin powder from industrial degumming liquor and utilized it as value added finishing agent for textiles. The polyester fabric was pre-treated with 15% sodium hydroxide (owf) keeping material to liquor ratio 1:40 at 60°C for 30 minutes and a weight loss of 5 percent was recorded. This weight loss was expected to give sufficient number of hydroxyl and carboxyl end groups so that further treatment can be carried out. The pre-treated fabrics were padded (80% expression) with the sericin solution (20 g/l) along with glutaraldehyde (1% v/v) magnesium chloride (1% w/v), acetic acid (0.1% v/v) in a laboratory padding mangle by 2-dip 2-nip process. The padded fabric was dried and cured. The cured samples were washed at 60°C and dried. It has been found that the original polyester fabric has a moisture content of 0.82%. On pre-treatment with sodium hydroxide, the moisture content increased up to 1.47% whereas at 20 g/l of sericin concentration in padding liquor gave a moisture content of 2.09%. This may be due to the increase in moisture content of the fabric in the presence of sericin on the fabric surface.

Haggag *et al.,* 2009 treated wool fabric with hydrogen peroxide/sodium sulphite/sericin system to improve its printability with acid, reactive and basic dyes. The treated printed fabrics showed higher degree of colour intensity compared to the untreated printed ones. Sericin was found to be effective in enhancing the printability of wool fabrics with acidic, reactive and basic dyes. The fastness properties of the treated fabrics after being printed with different classes of dyes were significantly improved. This system would be an ecologically acceptable alternative to the chlorination process which is used in preparation of wool for printing. The treatment of wool with the aforementioned system has a positive impact on some of the physico-mechanical properties such as resistance to felting, shrinkage and pilling as well as wettability. Similar results were encountered when sericin was added to the printing paste.

Mairal and Hardiant, 2009 utilized silk degumming waste liquor containing sericin as the medium for dyeing 100% polyester fabric and modified the polyester to have higher moisture regain. Conventional high temperature high pressure (HTHP) and thermosol (pad-dry-cure) dyeing methods were used to dye the 100% polyester fabric. The aim of the study was to study the dyeability of 100% polyester fabric modified and dyed in silk degumming waste liquor with disperse dyes. Moisture regain of the polyester fabric modified in the silk degumming waste liquor was greater than that of the control fabric. This is possibly because the sericin in the dye bath is absorbed by the polyester fibres and therefore the moisture regain of the polyester increased which gave an indication of reduction in its hydrophobic character. Tensile strength of the polyester sample treated in sodium hydroxide decreased as compared to the untreated polyester. The decrease in tensile strength was due to the hydrolysis of the polyester by the alkaline treatment. The decrease in tensile strength of samples treated in dyeing bath containing enzyme was probably due to the fact that enzyme also attacked some portion of the polyester fibre causing the reduction of the tensile strength.

Chu *et al.,* 2010 treated raw bamboo fiber with sericin protein in order to improve the finishing effect. It was selectively oxidized by $NaIO_4$ (sodium periodate) and then treated with sericin solution. The morphology and structure of sericin proteinic raw bamboo fiber were analyzed by means of Scanning Electron Microscope, X-ray diffraction and thermal analysis. The treatment results showed that sericin protein could be directly coated onto the oxidized raw bamboo fiber without any other reagent and a covalence was formed between amino and aldehyde group. After being treated with sericin protein solution, the thermal stability and crystallinity of the oxidized raw bamboo fiber improved and the surface became smoother.

Jassim and Alsaree, 2010 examined silk sericin for antibacterial property by growing spores colonies and tensile strength tests before and after treatment of the sample with 2% sericin solution and burying in the garden sand for 7 days. The results showed that the spores colonies are reduced from 146 before treatment with sericin to 29 only after treatment. Also the tensile strengths increased after treatment with 2% sericin. The *micrococcus leuteus* used to detect the effect of 10-20 mg/ml sericin to inhibit this kind of bacteria, by using well diffusion method seeded nutrient agar, then incubated at (35-37)°C for 24 hours. The zones of inhibition were observed between 9-12 mm in diameter. The study suggested that use the silk sericin for medical applications after isolation and identification of some pathogenic bacteria like *Pseudomonas aeruginosa, Staphyllococcus aureous* and *Escherchia coli* to produce medical bandages, mouth wash, antibacterial soaps and tooth-paste.

Cheng *et al.*, 2011 discussed the dyeing behaviour and wearability of sericin protein treated flax fabric. The results showed that the crease resistance of oxidized flax was significantly increased compared with that of the untreated flax. The sericin macro-molecule and the flax cellulose macro-molecule formed chemical cross linking after the oxidized flax fabric was treated by sericin and the crease resistance was further increased. The air permeability, moisture regain and dyeing behaviour of the sericin treated flax fabric increased slightly compared with the oxidized flax.

Kurioka and Shiozaki, 2011 treated cotton fabrics with sericin (1.2%) solution containing citric acid (9%) as a cross-linkage agent to improve their hydrophilic and thermal properties. The treated fabrics absorbed more water than the non-treated fabrics and the water diffusion time decreased. The moisture content and maximum absorption coefficient of the treated fabrics were much less than of untreated fabrics. Moreover, the q-max, thermal conductivity and air permeability resistance values were higher.

Rajendran *et al.*, 2011 developed an effective technology for the extraction of sericin from the cocoons of *Bombyxmori* silk worms. Sericin was extracted with ice cold ethanol to obtain crude extract. Sericin extract was coated onto cotton fabric by pad–dry–cure method. FTIR characterization of the sericin-coated cotton fabric showed distinct amide peaks. The antimicrobial activity of the sericin thus extracted was assessed by both qualitative (agar diffusion and parallel streak method) and quantitative (percentage reduction test) methods. An inhibition zone of 28 mm and 30 mm (for *E. coli* and *S. aureus*) by agar diffusion method and of 40 mm and 42 mm by parallel streak method were obtained. Quantitative assessment by percentage reduction test showed a reduction percentage of 89.4% and 81% for *S. aureus* and *E. coli* respectively. Results suggested that sericin might be a valuable ingredient for the development of antimicrobial textiles.

Tao, 2011 finished the knitted cashmere with sericin solutions in order to improve its pilling resistance. The relationship between the finishing processing parameters and the pilling resistance of the fabric was analyzed and the laundry resistance of the knitted cashmere before and after finishing was compared. The results showed that when the concentration of sericin solution 8% (owf), pH value 6, temperature 50°C and impregnating time 30 minutes, the best pilling resistant result of the cashmere was achieved as pilling resistance raised by 1 grade than that of the control. The cashmere also exhibited good laundry resistance, its pilling resistant grade remained the same after washing 20 times. The comparison of moisture regain, shrinkage and bursting strength of the cashmere before and after finishing showed that all of these properties of the finished cashmere improved except that the hand feel decreased slightly.

Xing *et al.,* 2011 fixed sericin onto the surface of cotton fabric in the presence of poly-carboxylic acids citric acid (CA) and butane tetra carboxylic acid (BTCA) at high temperature using a pad-dry-cure process. The effect of catalyst concentration, pH value, curing temperature and curing time on the finish were investigated. The optimized finishing conditions for cotton fabric were obtained. The weight gain of treated fabric with BTCA as crosslinking agent was higher than CA. The results showed that wrinkle recovery angle evidently increased and the wrinkle recovery angle of BTCA combined sericin treated fabric was higher than CA. The breaking strength, moisture regain and whiteness of the treated fabric slightly decreased while the air permeability of cotton fabrics did not change.

Chen *et al.,* 2012 stated that the silk sericin is the main residue in silk production which is low in cost and effective for removal of acidic dyes from water. In this study, sericin was characterized with various techniques including SEM (scanning electron microscope), X-Ray Diffraction, N_2 physisorption, FTIR (fourier transformed infrared spectroscopy) and XPS (X-ray photoelectron spectroscopy). Dye adsorption by sericin biosorbent was investigated with the acid yellow (AY), methylene blue (MB) and copper (II) phthalocyanine-tetrasulfonic acid (CuPc) dyes from water. Sericin displayed large capacity for AY and CuPc adsorption with adsorption capacities of 3.1 and 0.35 $mmol.g^{-1}$ respectively but it did not adsorb methylene blue dye. This selectivity is due to the basicity of amide groups in sericin biosorbents.

Khalifa *et al.,* 2012 extracted sericin from silkworm cocoons in order to obtain the optimum yield of sericin and optimized the parameters for a favorite sericin extraction. Then, the extracted sericin was fixed onto wool and cotton fabrics with an aim to modify some of their properties. The tests of treated fabrics showed that sericin had an affinity for wool. This affinity for wool fiber was obtained in defined conditions with about 48% exhaustion rate for sericin concentration of 2.5% (w/w). Compared to different sericin concentrations (0%, 2.5%, 5%, 10% and 20%), 5% of sericin (w/w) improved the wool samples touch until a score of 4 points, as well as the water absorption with a profit of 70.75%. The samples also showed an improved antibacterial activity. It revealed the multi-functionality of sericin as a finishing agent which improved both absorption and hand with an acceptable change in colour.

Zhou *et al.,* 2012 reported the adsorption behaviour of annatto dye on cotton fabrics pre-treated by sericin. It was found that the sericin largely developed the positive charge on cotton fibers following a decrease in the pH. Adsorption studies of the annatto dye on the modified cotton fibers showed that sericin enhanced the adsorption capacity of the annatto dye on cotton fibers. The results revealed that the uptake of annatto dye on modified cotton fibers occurred via electrostatic attractions between the anion of the dye and the cationic segments on the modified cotton fibers.

2.3 Effect of Different Treatments on Dyeability of Cotton

The environment friendly natural dyes are enjoying resurgence in popularity because of concern with the carcinogenic, mutagenic and sensitizing characteristics of some synthetic dyes. The ban on certain synthetic dyes has stimulated the entry of the golden era of natural dyes.

Saxena *et al.*, 1997 developed the method of application of lac dye on cotton pre-treated with chitosan (a naturally occurring polymer). The pre-treated cotton fabric was dyed using alum, ferrous sulphate and tannic acid as mordants. The dyed samples exhibited medium light fastness, good rubbing and perspiration fastness but poor wash fastness. The wash fastness improved considerably when the dyed fabric was treated with DMDHEU.

Rastogi *et al.,* 2000 used lac dye on cationised cotton fabric. Maximum colour yield was obtained in the fabric treated with 5 percent Discofix DBA at pH 4.0 and exhibited good fastness to washing and perspiration, fair to good to light and excellent fastness to dry and wet rubbing.

Eom *et al.*, 2001 cationized the cotton fabric before dyeing with natural colourant, namely Redwood, Gromwell, Cochineal, Goldthread and *Amur* cork tree for improving its dye ability. The K/S value of cationized cotton fabric dyed with Redwood and Cochineal was higher than that of untreated one. In case of Gromwell, though the K/S value of the cationized fabric was higher than that of untreated one yet the fabric showed poor levelness. Cationizaion of cotton had no effect on the dye ability of Goldthread and Amur cork tree. It was observed when the concentration of sodium hydroxide exceeds beyond a certain level, sodium hydroxide hydrolyzes the cationizing agent and the K/S value of dyed materials decreased.

Teli *et al.,* 2004 subjected cotton to swelling treatment using three different agents like sodium hydroxide, ethylene diamine and morpholene. These swollen cotton samples were treated with acid and neutral cellulose enzyme and dyed with aqueous extract of madder. The K/S values of the samples indicated that the enzyme treatment followed by swelling showed a higher depth of dyeing as compared to swelling treatment followed by enzyme treatment. The wash and light fastness of the treated samples were found to be good to excellent.

Kim, 2006 dyed cotton fabrics successfully by green tea extract upon chitosan mordanting. To increase the affinity of cotton fiber to the polyphenolic components in the green tea extract, a natural biopolymer chitosan was used as mordanting agent. The effects of chitosan concentration in mordanting on the dyeing characteristics and the UV protection property were examined. Chitosan mordanted green tea dyed cotton showed better dyeing characteristic and higher UV protection property compared with the unmordanted green tea dyed cotton. As the chitosan concentration increased, the dyeing efficiency and the UV protection property also increased.

Kamel *et al.,* 2007 studied the dyeing of cationized cotton fabrics with lac using both conventional and ultrasonic techniques. The effects of dye bath pH, salt concentration, ultrasonic power, dyeing time and temperature were studied and the resulting shades obtained with ultrasonic and conventional techniques were compared. Colour strength values obtained were found to be higher with ultrasonic than with conventional heating. The results of fastness properties of the dyed fabrics were fair to good.

Vankar *et al.,* 2007 studied two step ultrasonic dyeing of cotton and silk fabrics with natural dyes, *Terminalia arjuna*, *Punica granatum* and *Rheum emodi* were developed in which an enzyme was complexed with tannic acid first as a pretreatment. This was found to be comparable with one step simultaneous dyeing. The effectiveness of three enzymes protease-amylase, diasterase and lipase was determined. The enzymatic treatment gave cotton and silk fabrics rapid dye adsorption kinetics and total higher adsorption than untreated samples for all the three dyes. The CIE Lab values also showed improvement by enzymatic treatment. The tannic acid-enzyme-dye combination method offers an environmentally benign alternative to the metal mordanted natural dyeing.

Vankar *et al.,* 2008 studied the production of anthraquinone reddish orange dyes in roots, stem and leaves, which has been used for dyeing textiles since ancient times from *Rubia cordifolia*. Commercial sonicator dyeing with *Rubia* showed that pretreatment with biomordant, *Eurya acuminata DC var euprista Karth* of *Theaceae* family having local name, *Nausankhee* (*Apatani* tribe), *Turku* (*Nyishi* tribe) in 2% concentration showed very good fastness properties for dyed cotton using dry powder as 10% of the weight of the fabric. Use of biomordant replaces metal mordants thus making natural dyeing ecofriendly. For acidic extraction of dyes from fibres, ethanol was used. Due to its higher boiling point than methanol it evaporates slower from the extraction solution enabling a more efficient extraction of dyes.

Haji, 2012 extracted a natural cationic dye berberine, with excellent antibacterial activity from the roots of *Berberis vulgaris* and applied on cotton fabric. As, there is no affinity of cationic dyes for cotton fiber, hence to improve the dyeability of cotton fiber, plasma treatment and acrylic acid grafting, using plasma technology for pretreatment were employed. The samples dyed after acrylic acid grafting showed the highest antibacterial activity. Oxygen plasma treatment of cotton fabric improved the graft yield of acrylic acid on the fiber. Grafting of acrylic acid on cotton fiber is affected by time of plasma treatment, concentration and grafting time. Grafting of acrylic acid onto cotton fiber improves the absorption of berberine natural dye on it, because of ionic interactions between the dye and the modified fiber. The dyed fabric showed good fastness properties, besides excellent antibacterial activity against both gram-negative and gram-positive bacteria.

Sundrarajan *et al.,* 2012 modified woven cotton fabric with enzymes and chitosan in order to enhance the dye ability and reduce pollution load. Cellulase, an eco friendly biodegradable, non toxic and environmentally benign enzyme removed protruding fibers, fuzz on the surface of fabric by the process called bio polishing. It improved fabric hand, feel, appearance and also dye uptake. Chitosan; a biopolymer present as chitin in crab shell was prepared and crosslinked with the fabric using citric acid and sodium hypophosphite at 110°C. Modifications, evidenced by morphology changes inferred from SEM analysis. FTIR studies confirmed the fixation of chitosan on fabric. Unmodified and modified fabrics were dyed with natural dye *curcuma longa* (turmeric). Dye uptake and K/S values of dyed fabric were evaluated. It was revealed that the eco friendly modifiers i.e. enzyme and chitosan enhanced the dyeability and washing fastness of dyed fabric.

Ratnapandian, 2013 demonstrated that chitosan could be utilized to improve dye uptake by cotton fabrics during padding with natural dyes. Incorporating 0.05% chitosan achieved a maximum increase of 37.9% in the depth of shade obtained from the Acacia plant family. Excess chitosan limited the reactivity between the dye-mordant complex and the fabric, consequently reducing the final colour yield. The propensity of chitosan to increase the depth of shade was more pronounced when iron sulfate was the mordant. The fastness properties of the dyed fabrics were not affected by chitosan. All samples dyed in the presence of chitosan showed excellent antimicrobial properties. Thus such materials dyed in the presence of chitosan have the potential for application in medical textiles.

Saleh *et al.,* 2013 modified the cotton by cationization followed by dyeing with acacia bark. Cationization of cotton fabric for achieving electropositive charge and better adsorption properties was carried out with cationic compound produced from extractable solution of chicken feather. The cationized cotton fabric was dyed using acacia bark. The application of the extractable solution of chicken feather with cotton was studied. Color measurements were carried out to evaluate the shade obtained. The dyed fabrics were subjected for analysis in terms of K/S, CIE L* a* b*, hue and chroma as well as their fastness properties. The results indicated that the dye extraction was more effective in alkaline medium. The colour measurement values were found to be better with cationization than the untreated samples. The fastness properties improved from fair for untreated samples to good with the cationized fabrics.

CHAPTER-III

MATERIAL AND METHODS

The present research was carried out to investigate the effect of sericin treatment on dyeability of cotton fabric using a natural dye at Department of Textile and Apparel Designing, I.C. College of Home Science, CCS HAU, Hisar. The methods and techniques adopted for the fulfillment of the objectives of the study are presented under the following headings:

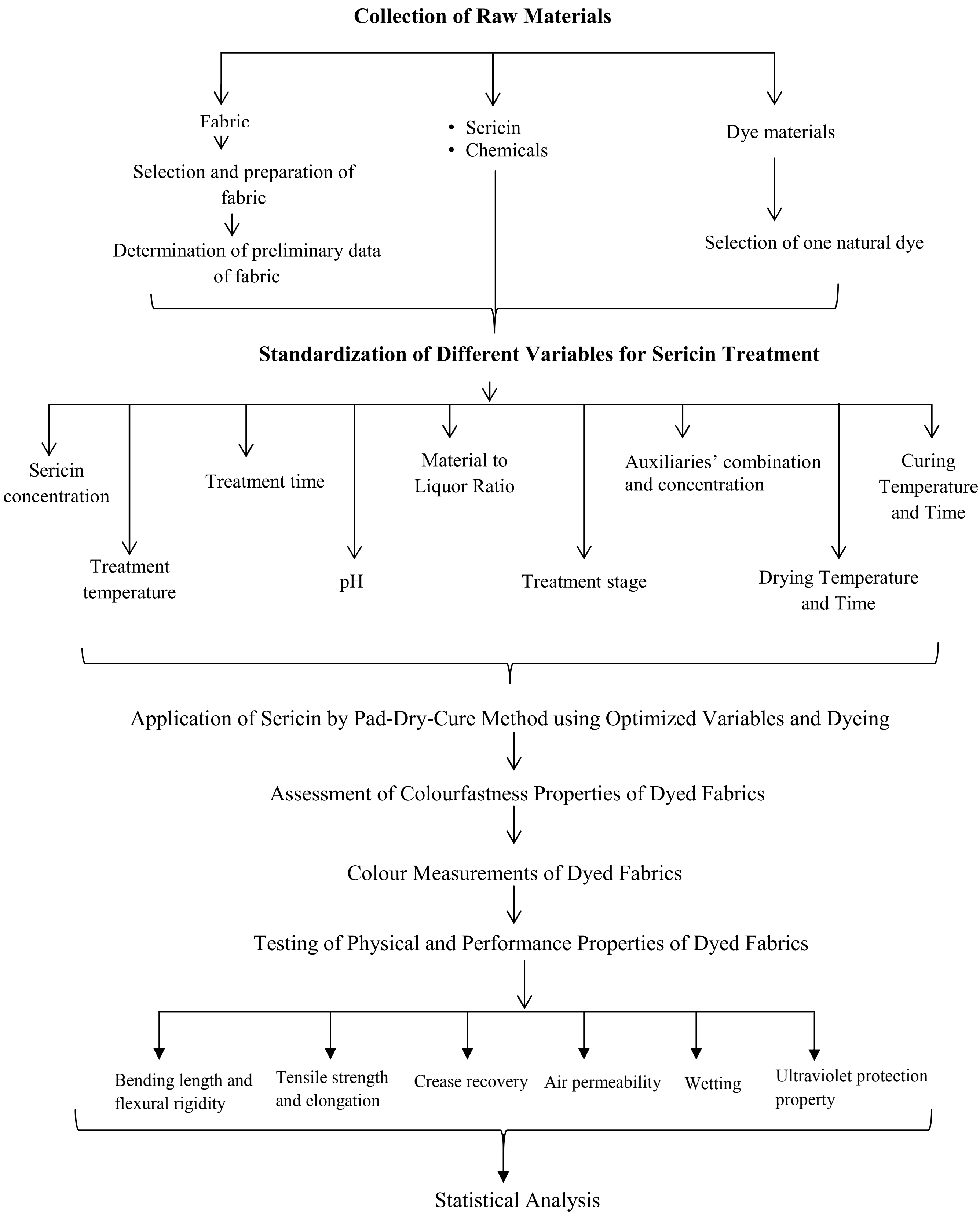
3.1 Collection of Raw Materials
3.2 Chemicals and Instruments Used in the Study
3.3 Preparation of Textile Material
3.4 Determination of Preliminary Data of the Fabric
3.5 Standardization of Different Variables for Sericin Treatment
3.6 Application of Sericin with Optimized Variables and Dyeing
3.7 Assessment of Colorfastness Properties of Dyed Fabrics
3.8 Colour Measurements of Dyed Fabrics
3.9 Testing of Physical and Performance Properties of Dyed Fabrics
3.10 Statistical Analysis

3.1 Collection of Raw Materials

3.1.1 Textile material: Three white cotton fabrics of medium weight were collected from local market. Medium weight bleached cotton fabric which was most suitable for apparel use was selected on the basis of maximum dye absorption and visual basis by advisory committee members from the major subject. To confirm that the fabric procured for the study is pure cotton, burning, microscopic and chemical test were conducted.

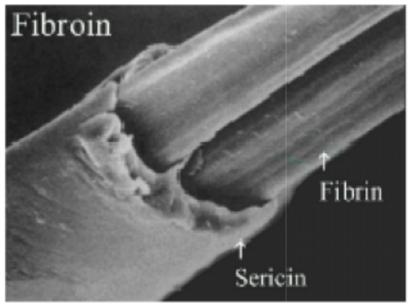
3.1.2 Dye material: Four plant materials which were easily available and in abundance *i.e. Kachnar* bark*, Manjistha* root, *Neem* leaves and Safflower flowers were collected and tried for the study. The dye materials were examined carefully to remove extraneous matter. These were dried in shade and ground to make powder. The selected fabric was treated with sericin and dyed with all the four dyes using standardized dyeing procedures (Senthilkumar *et al*., 2002; Marie *et al*., 2011; Rose, 2002; Teli *et al*., 2004). On the basis of improvement in percent dye absorption and washing fastness of dyed fabrics upon sericin treatment, one dye *i.e.* '*manjistha*' was selected for the present study. *Manjistha* or Indian madder (*Rubia cordifolia*) belongs to the family '*Rubiaceae'*. It is a prickly creeper, up to 10 m long, perennial, herbaceous climbing plant with very long roots, cylindrical, flexuous with thin red

RESEARCH DESIGN



bark. The valuable portion of this plant is entirely its root, which is usually of considerable length but does not exceed ordinary slate pencil in thickness. *Manjistha* or Madder is a source of natural dye producing variety of anthraquinone pigments in its roots and also in rhizomes. The main colouring matter present in the roots of madder is a mixture of purpurin (C.I. 75410), munjistin (C.I. 75370), xanthopurpurin (C.I. 75340), pseudopurpurin (C.I. 75420) and nordamncanthal. Madder is a very famous dye used for dyeing wool, silk and cotton fibers (Anon 1972; Bechtold 2009; Color Index 1971; Gupta *et al*., 2001a, 2001b).

3.1.3 Procurement of Sericin Powder: Sericin, an important component of the silk fibre making up around 25 percent of the weight of raw silk, is known as 'silk gum', which holds a number of other components such as waxes, fats and pigments. Sericin, a yellow coloured, brittle and inelastic substance has been proven to enhance adsorption capacity of dyes (Zhou *et al*., 2012). Sericin powder used for the study was procured from M/s Swapnaroop, Aurangabad, Maharashtra.



3.2 Chemicals and Instruments Used in the Study

3.2.1 Selection of chemicals: Toxic and banned chemicals were excluded from experimental work. Different laboratory grade chemicals were selected for the study on the basis of available literature, easy availability, cost effectiveness and ecofriendly nature.

Chemicals used for different purposes:

Sr. No.	Chemicals	Purpose
1.	Acetic acid	To control pH
2.	Anhydrous sodium carbonate	For preparation of washing liquor
3.	BTCA	As a cross-linking agent in sericin treatment
4.	Citric acid	As a cross-linking agent in sericin treatment
5.	Disodium hydrogen orthophoshphate dehydrate	For preparation of perspiration liquor
6.	Hydrochloric acid	To control pH
7.	L-Histidine monohydrochloride monohydrate	For preparation of perspiration liquor
8.	Magnesium chloride	As a catalyst in sericin treatment
9.	Sodium chloride	For preparation of perspiration liquor
10.	Sodium hydroxide	To control pH and for desizing of cotton fabric
11.	Sodium hypophosphite	As a catalyst in sericin treatment
12.	Sodium sulphite	Scouring of cotton fabric
13.	Sulphuric acid	Desizing of cotton fabric

3.2.2 Instruments used in the study:

Sr. No.	Instruments	Purpose
1.	Air permeability tester	To test air-permeability
2.	Centrifuge machine	To centrifuge the dye solutions
3.	Crease recovery tester	To determine the crease recovery angle of fabric samples
4.	Dessicators	To condition the fabric samples
5.	Digital chrockmeter	To determine rubbing fastness
6.	Digital pH meter	To measure pH
7.	Digital tensile tester	To determine the tensile strength and elongation of fabrics
8.	Electronic weighing balance	To weigh fabrics, dyes, sericin and auxiliaries
9.	Exposure rack	To determine light fastness
10.	Fabric thickness gauge	To determine thickness of fabric
11.	Grey Scale	To evaluate the fastness of dyed samples
12.	Grinder	To grind the plant material
13.	Laundrometer	To determine the washing fastness of dyed samples
14.	Padding mangle	For giving sericin treatment
15.	Perspirometer	To determine the perspiration fastness
16.	Pick glass	To determine the fabric count
17.	Precision scale for GSM	To determine the weight per unit area of fabric
18.	Serological water bath	To extract the dye and dye the fabric
19.	Stiffness tester	To determine the bending length
20.	Spectrophotometer SS5100A	To measure the colour of dyed fabrics
21.	Spectro-310 Spectrophotometer	To measure the optical density of dye solutions fastness
22.	UV Transmission Analyser	To test UPF value of samples

3.3 Preparation of Textile Material

The textile material must undergo preparation to ensure complete wetting, uniform absorbency of the dye and to remove colour or yellowness and any other impurity from the material that could interfere with colouration of the material.

The methods adopted for fabric preparation were:

3.3.1 Desizing of the fabric: Desizing was done to remove starch and any other sizing material present in the fabric. The cotton fabric was desized in a solution containing 1 percent sulphuric acid (H_2SO_4) at 50°C with MLR 1:40 for 60 minutes. The fabric was rinsed thoroughly to remove loose starch and other residues, if left. (Modi and Grade, 1975).

3.3.2 Scouring of the fabric: Scouring of the cotton fabric was done for the removal of natural and added impurities like oils, fats, waxes and other adventitious dirt that may have been added to the fabric. The fabric was weighed and soaked prior to introduction in the scouring bath. The fabric was squeezed thoroughly and was treated in water solution containing 1 percent soap, 3 percent soda ash and 0.5 percent sodium sulphite at boiling temperature (100°C) with MLR 1:40 for 60 minutes. The fabric was rinsed thoroughly to remove any residues, if left and dried on a flat surface. (Modi and Grade, 1975).

3.4 Determination of Preliminary Data of the Fabric

The preliminary data of the fabric to be treated and dyed was obtained under three parameters i.e. fabric count, thickness and weight. The fabric samples were conditioned prior to determination of fabric dimensions under standard test conditions i.e. relative humidity 65± 2 percent and temperature 27°C ± 2°C.

3.4.1 Fabric count: Fabric count is the number of warp yarns (ends) and weft yarns (picks) per inch in the woven fabric. Paramount pick glass with pointer was used to determine the fabric count of the woven cotton fabric using ASTM-D123 test method. It was determined by counting the number of threads per square inch in the warp and weft directions at five different places in the woven cotton fabric. An average of five readings was taken as the fabric count.

3.4.2 Fabric weight: The weight of the fabric is defined as weight of a known area of the material and then computing the weight per unit area. Samples were cut at random from fabric with the help of round cutter for GSM. The individual samples were suspended on the clamp of the pointer beam of the Paramount Precision Scale for GSM using ASTM-D3776-90 test method. The movement of the pointer beam was controlled with the help of break knob provided at the centre of the unit. The weight per unit area was read directly from the Quadrant scale from 0-250 GSM. An average of five readings was calculated.

3.4.3 Fabric thickness: Fabric thickness is defined as 'the distance between two parallel surfaces while exerting a specified pressure on the material' by the pressure foot of the tester. Paramount thickness tester was used to determine the thickness of the fabric using ASTM-D1777-60 Test method. A specimen was placed on the flat surface below pressure foot of the instrument without any folds and wrinkles. The pressure foot was lowered upon the specimen gently until the pointer of the dial meter stopped moving further and the reading on the dial gauge was recorded in mm. An average of five readings was calculated as the fabric thickness.

3.5 Standardization of Different Variables for Sericin Treatment

Experiments were carried out to optimize sericin concentration, treatment temperature, treatment time, pH, material to liquor ratio (MLR), treatment stage, combination and concentration of auxiliaries, drying temperature and time, curing temperature and time. Different ranges of variables were tried for optimization on the basis of available review. The optimization of the variables was done on the basis of maximum percent dye absorption and better wash fastness exhibited by the fabrics treated with sericin and dyed with selected natural dye.

The standardization of variables was done under following sub sections:

3.5.1 Optimization of sericin concentration
3.5.2 Optimization of treatment temperature
3.5.3 Optimization of treatment time
3.5.4 Optimization of treatment pH
3.5.5 Optimization of treatment stage
3.5.6 Optimization of material to liquor ratio
3.5.7 Optimization of auxiliaries' combination and concentration
3.5.8 Optimization of drying temperature and time
3.5.9 Optimization of curing temperature and time

3.5.1 Optimization of sericin concentration: For determining the optimum concentration of sericin, five padding baths were prepared containing five different concentrations of sericin i.e. 0.25, 0.50, 0.75, 1.00 and 1.25 percent (1 percent means 1 g of sericin powder/100 gm of fabric) whereas other variables were kept constant i.e. treatment temperature: 60°C, treatment time: 45 minutes, pH: 7, material to liquor ratio: 1:40, cross-linking agent: 3% and catalyst: 1.5%. The application was carried out by pad-dry-cure method in a laboratory padding mangle. The pressure of 2 kg/cm was maintained and the 70-75% expression was achieved. The padded and squeezed cotton fabric samples were dried at 80°C for 6 minutes and subsequently cured at 160°C for 3 minutes. Sericin treated samples were dyed using natural dye as per standardized dyeing procedure given below:

Five percent of selected natural dye material was boiled and extracted for one hour in aqueous medium, cooled and filtered. The filtrate was used as dye solution for dyeing. The sericin treated cotton samples were dyed keeping material to liquor ratio 1:40. Temperature of the dye bath was raised to 90°C and maintained for 45 minutes. The dyed fabrics were rinsed with water followed by soaping with 2g/l neutral detergent at 60°C. The samples were washed thoroughly with cold water, squeezed and dried (Teli *et al*., 2004). Before and after dyeing, 1 ml of dye solution was taken out from the dye bath, diluted to 100 times, centrifuged at 2500 rpm for 20 minutes and optical density (O.D.) of the solution was taken on a spectrophotometer at 400 nm wave length.

Percent dye absorption of the samples was calculated using the following formula:

$$\text{Percent dye absorption} = \frac{\text{O.D. before dyeing} - \text{O.D. after dyeing}}{\text{O.D. before dyeing}} \times 100$$

Percent dye absorption and wash fastness of the samples was noted and optimum sericin concentration was selected on the basis of maximum dye absorption and better wash fastness.

3.5.2 Optimization of treatment temperature: The treatments were carried out at five different temperatures viz. 40, 50, 60, 70 and 80°C using optimized sericin concentration while other variables were kept constant i.e. treatment time: 45 minutes, pH: 7, material to liquor ratio: 1:40, cross-linking agent: 3% and catalyst: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes. Sericin treated samples were dyed using selected dye as per standardized procedure as explained under section [3.5.1].

The temperature reflecting the maximum percent dye absorption and wash fastness was taken as the optimum treatment temperature.

3.5.3 Optimization of treatment time: Sericin treatments were given to fabric samples for five time durations i.e. 15, 30, 45, 50 and 60 minutes using optimized sericin concentration and treatment temperature while other variables were kept constant i.e. pH: 7, material to liquor ratio: 1:40, cross-linking agent: 3% and catalyst: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes. Treated samples were dyed using selected natural dye as explained under section [3.5.1]. The optimum treatment time was selected on the basis of maximum percent dye absorption and better wash fastness property.

3.5.4 Optimization of treatment pH: To optimize treatment pH, sericin solutions were set to five different pH i.e. 5, 6, 7, 8 and 9. The cotton fabric samples were treated at optimized sericin concentration, treatment temperature and time whereas other variables were kept constant including material to liquor ratio: 1:40, cross-linking agent: 3% and catalyst: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes. The samples were dyed at optimized conditions with the selected natural dye as mentioned under section [3.5.1]. Optimum treatment pH was selected on the basis of maximum dye absorption and better wash fastness rating.

3.5.5 Optimization of treatment stage

Sericin treatment was given at three stages i.e. pre, simultaneous and post treatment stage using optimized sericin concentration, treatment temperature, treatment time and pH whereas other variables were kept constant including material to liquor ratio 1:40, cross-linking agent: 3%, catalyst: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes.

i. Pre-treatment: The optimum concentration of sericin was dissolved in distilled water keeping material to liquor ratio 1:40 and the desized and scoured sample was added to it. The sericin treatment was carried out at optimized temperature for optimized time at optimized pH. The samples were passed through the padding mangle and samples were dried and cured. Treated samples were dyed with selected natural dye using standardized procedure as given under section [3.5.1].

ii. **Simultaneous-treatment:** Sericin and dye was applied simultaneously in the same bath. The optical density of the original extracted dye liquor was recorded and samples were dyed in dye liquor at 60°C for 15 minutes. Required amount of sericin and auxiliaries were added to the dye solution and dyed for further 30 minutes with occasional stirring using optimized dyeing conditions as explained under section [3.5.1]. The samples were passed through the padding mangle and were dried at 80°C for 6 minutes and cured at 160°C for 3 minutes. The optical density of the dye liquor was again recorded and sample was washed, rinsed and dried in shade.

iii. **Post-treatment:** The sample was first dyed with the dye material at optimum dyeing conditions as explained under section [3.5.1]. Sericin and auxiliaries were added in the same bath after exhausting the dye. The sericin treatment was given for optimized time, temperature and pH. Samples were passed through laboratory padding mangle and dried at 80°C for 6 minutes and cured at 160°C for 3 minutes, subsequently sample was rinsed and allowed to dry.

One stage of treatment was selected on the basis of maximum dye absorption and wash fastness for further work.

3.5.6 Optimization of material to liquor ratio (MLR): Five treatment baths were prepared for the optimization of material to liquor ratio. For this purpose treatment was given keeping M:L ratio 1:20, 1:30, 1:40, 1:50 and 1:60 at optimum sericin concentration, treatment temperature, treatment time, pH and stage of application while other variables were kept constant *i.e.* cross-linking agent: 3%, catalyst: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes. Treated samples were then dyed with natural dye as per procedure given under section [3.5.1] and optical density was recorded.

Material to liquor ratio which gave maximum percent dye absorption and better wash fastness was selected for further research work.

3.5.7 Optimization of auxiliaries' combination and concentration

i. **Optimization of auxiliaries combination**: To improve the durability of sericin treatment, three non-formaldehyde released crosslinking agents i.e. 1, 2, 3, 4-butane tetra carboxylic acid (BTCA), citric acid (CA) and gluteraldehyde and two catalysts i.e. magnesium chloride and sodium hypophosphite were tried for the study. Using optimum sericin concentration, treatment temperature, treatment time, pH, stage of application and material to liquor ratio, three combinations of crosslinking agent and catalyst on the basis of reviews were tried i.e. 1, 2, 3, 4-butane tetra carboxylic acid

(BTCA) and sodium hypophosphite (Xing *et al.,* 2011), citric acid and sodium hypophosphite (Xing *et al.,* 2011), gluteraldehyde and magnesium chloride (Kongdee and Chinthawan, 2007) keeping other variables constant. Treated samples were dyed with selected dye as per procedure given under section [3.5.1] and percent dye absorption was calculated. One combination of auxiliaries i.e. cross-linking agent and catalyst was selected on the basis of percent dye absorption and wash fastness keeping cost and availability under consideration.

ii. Optimization of auxiliaries concentrations

a. Concentration of cross-linking agent: Different concentrations of selected cross-linking agent i.e. 1, 2, 3, 4 and 5 percent on the weight of fabric were taken for sericin treatment using optimized sericin concentration, treatment temperature, treatment time, pH, stage of application, material to liquor ratio and selected catalyst while other variables were kept constant including catalyst concentration: 1.5%, drying temperature: 80°C, drying time: 6 minutes, curing temperature: 160°C, curing time: 3 minutes. Afterwards dyeing was carried out as per procedure given under section [3.5.1]. Optimum concentration of cross-linking agent was decided on the basis of maximum dye absorption and better wash fastness.

b. Concentration of catalyst: Different concentrations of selected catalyst i.e. 0.5, 1, 1.5, 2 and 2.5 percent (o.w.f.) were added in five padding baths using optimized sericin concentration, treatment temperature, treatment time, pH, stage of treatment, material to liquor ratio and concentration of cross linking agent while other variables were taken as constant. The samples were passed through the padding mangle and were dried at 80°C for 6 minutes and cured at 160°C for 3 minutes. Treated samples were dyed using one natural dye as per procedure given under section [3.5.1].

Optimum concentration of catalyst was decided on the basis of maximum dye absorption and wash fastness.

3.5.8 Optimization of drying temperature and time

i. Drying temperature: For optimization of the drying temperature, fabric samples were treated using optimum sericin concentration, treatment temperature, treatment time, pH, treatment stage, material to liquor ratio, concentration of cross linking agent and catalyst. The application was carried out by pad-dry-cure method in a laboratory padding mangle. The pressure of 2 kg/cm was maintained and the 70-75% expression was achieved. Drying of treated samples was carried out at five different temperatures i.e. 60, 70, 80, 90 and 100^{0}C for 6 minutes and cured at 160^{0}C for 3 minutes. Treated samples were dyed using selected natural dye as per standard procedure given under section [3.5.1]. Temperature exhibiting maximum dye absorption and wash fastness was taken for further research work.

ii. **Drying time:** For optimization of the drying time, fabric samples were immersed in five padding baths using optimized sericin concentration, treatment temperature, treatment time, pH, stage of treatment, material to liquor ratio, cross linking agent and catalyst concentration while other variables were kept constant. Afterwards the impregnated samples were passed between the squeezing rollers of the laboratory padding mangle. Drying of treated samples was carried out for five different time durations i.e. 2, 4, 6, 8 and 10 minutes at optimum drying temperature. The treated fabric samples were cured at 160^{0}C for 3 minutes. Treated samples were dyed using selected natural dye as per procedure given under section [3.5.1]. Drying time reflecting maximum dye absorption and wash fastness was taken for final treatment.

3.5.9 Optimization of curing temperature and time

i. **Curing temperature:** For optimization of the curing temperature, fabric samples were treated using optimized conditions. Curing was carried out at 5 temperatures i.e. 140, 150, 160, 170 and 180°C for 3 minutes of curing time. Treated samples were dyed using selected natural dye as per procedure given under section [3.5.1]. Temperature giving maximum dye absorption and wash fastness rating was taken for further research work.

ii. **Curing time:** Fabric samples were treated using optimized variables for optimization of the curing time. The application was carried out by pad-dry-cure method in a laboratory padding mangle. Padded samples were dried at optimum temperature for optimum time and cured at optimum temperature for five different time durations i.e. 1, 2, 3, 4 and 5 minutes. Treated samples were dyed using standardized natural dye as per procedure given under section [3.5.1]. The curing time exhibiting maximum percent dye absorption and wash fastness was selected as optimum curing time.

3.6 Application of Sericin with Optimized Variables and Dyeing

The sericin was applied on scoured cotton fabric after optimizing all variables. Fabric was immersed in the prepared padding bath using optimum sericin concentration, cross-linking agent and catalyst, optimum treatment temperature, treatment time, pH, treatment stage at optimum material to liquor ratio. The fabric and padding solution were placed in the trough of padding mangle. The fabric was passed between the rollers of the pneumatic padding mangle. The treated samples were dried at optimum temperature for optimum time and cured at optimum temperature for optimum time. Treated fabric was dyed with the selected natural dye using optimized conditions *i.e.* dye material concentration, dyeing temperature and dyeing time as mentioned under section [3.5.1].

3.7 Assessment of Colour Fastness Properties of Dyed Fabrics

Fastness is the Ability of dye to retain its colour after exposure to sun, perspiration, atmosphere, washing or other colour destroying agents (Wingate, 1988). Natural destructive agents like light, weather, oxygen and other atmospheric gases can fade and destroy certain dyes. In addition to natural agents, there are many chemicals and finishing treatments used in wet textile processing which may influence fastness of colours to some degree. Most dyes are organic compounds and are, therefore, vulnerable in varying degree to the action of destructive agents.

Several tests for the assessment of fastness of dyes are available. A number of tests are necessary to cover all the important properties of any dye because good fastness to one inference is not necessarily accompanied by equal fastness to other conditions. Tests may be conducted as per those of customer's significance such as light, washing, rubbing, perspiration etc. All the dyed samples were evaluated for colour fastness to washing, rubbing, light and perspiration using the methods prescribed by the Bureau of Indian Standards. The colourfastness grading was given by experts/judges having experience of working with natural dyes.

3.7.1 Fastness to washing: For the present study washing fastness test was carried out as per recommendations of IS: 3361-1979 method (BIS, 1979).

The dyed samples were placed between two pieces of bleached fabrics measuring 10cm x 4cm (in parallel lengths). One piece was of the same material as of the dyed sample i.e. cotton and other piece was of wool fabric. Two pieces of fabric were stitched together, taking the sample in between the stitches.

The composite specimen were weighed and the required quantity of soap solution at the rate of 5 g/1 of water was prepared keeping material liquor ratio 1:50. One composite specimen was placed in each of the eight containers of launder-o-meter and the soap solution was added to it. The composite specimen were treated for 45 minutes at 50±2^{o}C in the launder-o-meter. The specimen were removed and rinsed in cold water. The samples were squeezed and the stitching along the two long sides and one short side was removed. The samples were opened and dried in air with remaining line of stitching. The change in colour of dyed samples was assessed with Grey Scale No. 1 as per the recommendations of the ISO 105 method.

The nine-step scale consisting of the half fastness ratings was used. A piece of the original dyed sample and the test specimen was placed side by side in the same plane. The light was incident upon the surfaces at approximately angle of 45^{o} and the direction of viewing approximately perpendicular to the plane of surface. The visual difference was compared between the original and tested material with the difference represented by the Grey Scale. The fastness rating of the specimen is that number of Grey Scale which has a perceived colour difference nearest in magnitude to perceived colour difference between the original piece and the tested specimen. Also the degree of staining on the bleached cloth was assessed with the help of Grey Scale No.2 for staining.

3.7.2 Fastness to sunlight: Light fastness is defined as the resistance to fade or hue change of a coloured object when exposed to light source, most notably sunlight or the résistance of a material to change its colour characteristics as a result of exposure of the material to sunlight or to artificial light source. The effect of long-term ultra violet attack is to break the coloured compound down into by-products of a different shade, either to cause a hue shift such as darkening or to remove the colour altogether.

The apparatus used for testing of fastness to sunlight was the exposure rack. The fastness to sunlight was carried out according to IS: 686-1985 method (BIS, 1985). The test specimen was mounted in exposure rack in such a way that half of each specimen was covered and the other half was exposed to light. The samples were exposed to daylight everyday from sunrise to sunset, keeping the exposure rack at an angle of 45^{o} and the total exposure time being 48 hours. The change in colour of the exposed portion was compared with that of the unexposed portion using Grey Scale.

3.7.3 Fastness to perspiration: The fastness to perspiration was tested using the test IS: 971-1983 prescribed by the Bureau of Indian Standards (BIS, 1983). The acidic test liquor was prepared by dissolving 0.5 g of L-Histidine monohydrochloride monohydrate, 5 g of sodium chloride and 2.2 g of sodium dihydrogen orthophosphate dehydrate in one liter of water. The solution was brought to pH 5.5 with 0.1 N acetic acid solution.

The alkaline test liquor was prepared by dissolving 0.5 g of L-Histamine monohydrochloride monohydrate, 5 g of sodium chloride and 2.5 g of disodium hydrogen orthophosphate dehydrate in one liter of water. The solution was brought to pH 8 with 0.1 N-sodium hydroxide solution. The composite specimen were prepared in the same manner as for the wash fastness test. The test specimen were soaked in the acidic and alkaline test solutions separately with material to liquor ratio 1:50 for 40 minutes at room temperature. The treated samples were kept between two acrylic plates of a perspirometer under a force of 5 kg. The loaded apparatus was kept in hot air oven for four hours at $37\pm2^{o}C$. The test samples were removed from the oven and air-dried with temperature not exceeding $60^{o}C$. The numerical grading for colour change of the test pieces and for staining of two adjacent pieces was done using the Grey Scale.

3.7.4 Fastness to rubbing: Fastness to rubbing means the resistance of textile materials to every type of rubbing and staining from textiles in actual use. The fastness to rubbing was carried out according to IS: 766-1988 method (BIS, 1988). The samples were made for carrying out dry as well as wet rubbing tests. One test sample was taken and fixed to the rubbing device of a crock meter. The bleached cotton sample of size 2x2 cm was fixed to the finger of rubbing device of crock meter. The test specimen was rubbed to and fro with the bleached piece with a downward force of 900 g in a straight line, along a track of 10 cms for 10 times in 10 seconds. The test specimen was graded for the change in colour and staining using the Grey Scale. The test was carried out similarly for wet rubbing fastness using wet bleached white cotton fabric for fixing to the finger of rubbing device of crock meter.

3.8 Colour Measurements of Dyed Fabrics

The methods of measuring colour numerically were established by the Commission International del 'Eclairage (CIE) in 1931 and 1976. These methods provide uniform colour differences in relation to visual differences. Also the measurements of colour are precise and colour is specified by a set of numbers, which can be used to re-create the original colours at any other time and place i.e. reproduction. Measurement of colour increases the objectivity of the experimental result of dyeing (Giles, 1974 and Valia, 1987).

The colour of dyed samples was measured numerically through computerized colour matching machine. The reflectance spectra of dyed samples were observed by using spectrophotometer SS5100A and K/S values and the CIE LAB co-ordinates L*, a* and b* were noted down directly from the computer screen. This spectrophotometer uses CIE LAB (1976) colourspace, D65 illuminate matching and appraisal) and 420 nm wavelengths to measure the actual colour and change in colour. The Kubelka Munk theory makes it possible to predict the colour value.

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

Where,

K = a constant about the light absorption of the dyed fabric

S = a constant about the light scattering of the dyed fabric

R = reflectance of the dyed fabric, expressed in fractional form

The CIE LAB colour space uses L*, a*, b* scales to describe colour. L* is a measure of darkness/lightness of colour of an object and ranges from 0 (black) to 100 (white), a* is a measure of redness (+ve a*) or greenness (-ve a*), b* is a measure of yellowness (+ve b*) or blueness (-ve b*) and c* is a measure of dullness/brightness.

3.9 Testing of Physical and Performance Properties of Dyed Fabrics

The samples were conditioned for 24 hours prior to the determination of fabric properties under standard test conditions i.e. relative humidity: 65±2 percent, temperature: 27 ±2°C.

3.9.1 Physical Properties

Physical properties play an important role in the buying and selling of fabrics as well as in consumer use. Assessment of physical properties aims to predict behavior of fabric in use. It aids in defining the quality of a fabric or those associated with serviceability as there is not always, a clear dividing line between physical and performance properties.

Physical properties tested in the preliminary stage i.e. fabric count, thickness and fabric weight were again tested as per procedure mentioned in sections (3.4.1), (3.4.2) and (3.4.3) respectively, to analyze the effect of processing on the physical properties of the treated fabric. In addition to these, some other physical properties were also tested as follows:

i. Bending length and flexural rigidity: Bending length is the length of fabric that will bend under its own weight to definite extent. It determines the drapability of fabric. Bending length is related to the quality of stiffness that is appreciated by visual examination of the draped material, in the sense that clothes having a high bending length tend to drape stiffly. The bending length of the fabrics was determined by the Paramount stiffness tester using IS: 6490-1971 test method. Samples of size 25x200 mm were cut from warp and weft directions with the aid of template and conditioned. Both template and sample was transferred to the platform with the fabric underneath, coinciding the zero mark of the scale and zero line engraved on the side of platform. The template was moved slowly over 41.5 degree sloped along with the strip till the top of the specimen viewed in the mirror cut both index lines. The bending length was read from the scale, which coincided with the front edge of the top plate. Each sample was tested at all the four edges.

Reading from the scale was noted in mm and an average of four readings was calculated for both the directions.

$$\text{Average Bending Length} = \frac{S}{4 \times n}$$

Where

S = Sum of total bending length of specimens (four readings per specimen in either warp or weft direction)

n = Total number of specimen tested in warp or weft direction.

Flexural rigidity in warp direction (G_1)

$G_1 = W \times C^2$ mg- cm

Flexural rigidity in weft direction (G_2)

$G_2 = W \times C^2$ mg- cm

Where**,**

W = Weight per unit area of the fabric in milligrams per square centimeter.

C = The mean bending length for the respective direction.

Overall flexural rigidity (G) = $\sqrt{G_1 \times G_2}$

In the same way, bending length and flexural rigidity of controlled and sericin treated dyed fabrics were measured.

ii. Tensile strength and elongation: Tensile strength is the ability of the fabric to withstand the load of force usually expressed as kilogram weight or pound weight. Elongation of fabric corresponding to tensile strength is the original length of the sample at breaking point. Tensile strength along with elongation of fabrics was determined on Paramount digital tensile strength tester (Analogue Model), using IS 4169 standard test method. The samples of size 6x 4±0.05 inches were cut from warp and weft direction of the fabric. The samples were mounted between the jaws approximately 1.5 inch of fabric protruding from each side of the jaws at a distance of 3 inches. The speed of upper jaw was adjusted at 300±10 mm/min. The instrument was started and the upper jaw moved in upward direction. The readings were noted from the digital display at sample break. The specimen were slightly tensioned for getting accurate elongation at break. Five readings of the specimen from both directions (warp and weft) were taken and the averages were calculated.

Elongation is the ability to be stretched, extended or lengthened. Elongation of the fabric measured along with tensile strength through the digital monitor screen fitted on the tensile strength tester which gives elongation reading along with tensile strength of the fabric. The same procedure was followed to calculate the percent elongation of the fabric as the standard test method used for tensile strength testing.

iii. Crease recovery: Crease recovery properties of fabric include the crease recovery angle and percent cloth crease recovery. By using these properties the crease recovery performance of the fabric was assessed.

Wrinkle recovery is the resistance to and recovery from creasing. Resistance to creasing depends on the rigidity while the recovery depends on the elasticity. The measure of crease recovery is the angle at which the sample recovers from creasing. The wrinkle recovery was determined on the Shirley crease recovery tester using BS3086:1972 test method. Samples were cut both in warp and weft directions from the fabrics with a template measuring 0.5×2.5 cm and tested after conditioning. The test specimen was carefully creased by folding in half and was placed between the two glass plates. The specimen was creased for 3 minutes under 2 kg weight, the specimen was removed and transferred to the fabric clamp in the instrument and allowed to recover from the crease for 3 minutes. As it recovers, the dial of the instrument was rotated to keep the free edge of the specimen in line with the knife edge. The recovery angle in degrees was noted from the engraved scale. Warp and weft way recovery was observed separately to the nearest degree from the mean values of the five readings in each direction. The untreated and sericin treated fabric samples were tested for fabric crease recovery by standard test method.

Fabric crease recovery was determined by using the following formula:

$$\text{Fabric crease recovery} = \sqrt{\text{warp} - \text{way angle}} \times \sqrt{\text{weft} - \text{way angle}}$$

iv. Air permeability: It is the property of the fabric to allow air to pass through under the effect of difference in pressure. The corresponding measurement is that of the quantity of air passing through a surface of defined magnitude, under defined pressure difference and within a determined period of time. Prolific Air Permeability Tester was used to find the air permeability of the fabrics using BS3321: 1960 test method at 10 mm water column. A sample size of 2x2 inches was taken and placed on the opening of the test cylinder under the clamping place. The clamps were tightened to the samples firmly. The motor of the vacuum pump was switched on and was opened on the biggest rotameter (fixed on the right side of the instrument) till the required pressure difference was obtained on the manometer. The flow rate on the scale fitted to rotameter tube against the top surface of the float was read. The experiment was repeated five times and mean was calculated. The experiment was conducted for the untreated dyed and sericin treated dyed fabrics at different places where warp and weft threads do not repeat. Air permeability was calculated with the help of following formula:

$$\text{Air permeability (AP)} = \frac{\text{Rate of air flow}}{\text{Exposed area}} \times 100$$

$$\text{AP} = \text{k} \times \text{Rotameter reading}$$

Where k is the conversion factor i.e. = 0.01667 (the 10cm^2 area of the fabric was exposed for checking the air permeability in cm^3/ second/cm^2).

v. **Wetting:** Wetting is defined as the time in seconds for a drop of distilled water to sink into the fabric. The extent of hydrophilicity is inversely proportional to the time taken by the water to sink into the fabric completely. If the time to soak water droplet exceeds 20 seconds, the fabric is considered unwettable. AATCC Test Method 79 for testing the absorbency of textiles was used to test the propensity of the fabrics to take up water. Samples were fixed in an embroidery hoop with all creases out of it over the top of a beaker. A drop of distilled water was dispensed from a burette onto the surface of the fabric from a distance of 9.5 mm. Time was recorded until the water drop absorbed completely. A total of five readings were taken for each fabric sample and mean was calculated.

3.9.2 Performance Property

i. **Ultraviolet protection property:** Ultraviolet Protection Factor (UPF) of untreated and treated dyed fabrics was determined using SDL UV penetration and protection measurement system (Compsec M 350 UV- Visible Spectrometer), according to test method UVR TRANSMISSION AATCC-183:2004. The UPF is computed as the ratio of erythemically weighted ultra violet radiation (UV-R) irradiance at the detector with no specimen to the erythemically weighted ultra violet radiation (UV-R) irradiance at the detector with specimen present. Four specimens from each sample were tested for dry testing. Specimens of 5 cm were cut from each sample. Conditioned specimens, under standard atmosphere were placed against the sample transmission port opening in the sphere. UV transmission was taken with the specimen oriented in one direction, a second measurement at 0.79 rad (4^{50}) to the first and third at 0.79 rad (4^{50}) to the second. Individual measurements were recorded. Average spectral transmittance was calculated for the three measurements per specimen with a total of five specimen representing each fabric sample. UPF was computed using mean percent transmission in the UVA range (320-400nm) and mean percent transmission in the UVB range (280-320nm).

The UPF of each specimen was calculated using following equation:

$$\text{UPF} = \frac{\sum E\lambda \times S\lambda \times \Delta\lambda}{\sum E\lambda \times S\lambda \times T\lambda \times \Delta\lambda}$$

Where,

$E\lambda$ = relative erythemal spectral effectiveness

$S\lambda$ = solar spectral irradiance

$T\lambda$ = average spectral transmittance of the specimen (measured)

$\Delta\lambda$= measured wavelength interval (nm)

The UV protection category was determined by the UPF values described by Australia/New Zealand Standards AS/NZS4399 (1996) given in table:

UPF classification system

UPF range	UVR Protection category	UPF ratings
15-24	Good protection	15, 20
25-39	Very good protection	25, 30,35
40-50, 50+	Excellent protection	40, 45, 50,50+

3.10 Statistical Analysis

The data were tabulated and appropriate statistical tools and tests were applied to draw meaningful inferences.

i. Percentage change: The percentage change was used for calculating change in physical properties. It was obtained according to the formula:

$$\text{Percentage change (\%)} = \frac{\text{Difference between value of untreated and treated dyed fabric}}{\text{Value of untreated dyed fabric}}$$

ii. Mean: Arithmetic mean is calculated by obtaining the sum of all the observations and then divided by total number of observations in the set. In the study, mean was calculated for the physical and performance properties of the fabric.

$$\text{Arithmetic Mean} = \Sigma X / n$$

Where,

X = sum of all the observations,

n = total number of observations

iii. Weighted mean score: WMS was calculated to quantify the data regarding the rating of colour fastness.

$$\text{WMS} = \frac{\text{Toatal weighted score}}{\text{No. of colourfastness ratings}}$$

iv. Standard Deviation: The standard deviation is the positive square root of the average of the squared deviations taken from arithmetic mean. Standard deviation was used to calculate the error in the mean of different fabrics.

$$\sigma = \sqrt{\sum} \text{X}i^2/\text{n}$$

v. Standard Error: The standard error of the mean (SE_m) is the standard deviation of the sample mean's estimate of a population mean. In the study, the standard error was used to calculate the error in the mean of physical properties of treated and untreated fabrics. SEM is usually estimated by the sample estimate of the population standard deviation (sample standard deviation) divided by the square root of the sample size (assuming statistical independence of the values in the sample):

$$\text{SE} = \frac{s}{\sqrt{n}}$$

Where,

s = standard deviation,

n = total number of observations

vi. Coefficient of Variance: For knowing the variability of two or more sets of data, Coefficient of Variance was used. It is expressed in percentage.

$$C.\ V. = \frac{\text{S.D.}}{\text{A.M.}} \times 100$$

Where,

S.D. = standard deviation,

A.M. = arithmetic mean

CHAPTER-IV

RESULTS

The present study was undertaken to study the effect of sericin treatment on dyeability of cotton fabric using natural dye. To achieve the objectives, sericin treatment was given using a crosslinking agent and catalyst to make the treatment more durable. Different conditions of sericin treatment were optimized on the basis of maximum percent dye absorption and better wash fastness when treated fabric was dyed with a natural dye *i.e. manjistha* using its standardized dyeing procedure. The colourfastness, physical and performance properties of the treated dyed cotton fabric were investigated. The results obtained are explained under the following subsections:

4.1 Preliminary Data of the Fabric

4.2 Selection of a Natural Dye

4.3 Standardization of Different Variables for Sericin Treatment

4.4 Application of Sericin using Optimized Variables and Dyeing

4.5 Assessment of Colourfastness Properties of Dyed Fabrics

4.6 Colour Measurements of Dyed Fabrics

4.7 Testing of Physical and Performance Properties of Dyed Fabrics

4.1 Preliminary Data of the Fabric: The cotton fabric was desized and scoured which was used for further treatment. The selected fabric was assessed for fabric count, fabric weight and thickness.

Table: 1 Preliminary data of the cotton fabric

Fabrics	Fabric Properties			
	Fabric count (threads/inch)		Fabric weight (g/sq.m)	Thickness (mm)
	Warp	Weft		
Unscoured Cotton	99	55	121.78	0.32
Scoured Cotton	101	57	114.43	0.31

The data in Table 1 indicates that fabric count of selected unscoured cotton fabric used for the study was having 99x55 ends and picks per square inch, weighing 121.78 gm/m^2 and 0.32 mm thickness. It is evident from the table that fabric count of scoured fabric was 101 and 57 threads per inch in warp and weft direction, respectively, weight per unit area was 114.43gm per square meter and thickness of fabric was 0.31 mm.

4.2 Selection of a Natural Dye

Four natural dyes i.e. *kachnar* bark, *manjistha* roots, *neem* leaves and safflower flowers were used to dye sericin treated cotton fabric using their standardized dyeing procedure. Out of four dyes, one dye was selected on the basis of effect of sericin treatment on percent dye absorption and wash fastness property of the dyed fabric. The effect of sericin treatment on percent dye absorption and wash fastness grades of all the four dyes are presented in Table 2.

Table-2 Dye absorption and wash fastness of fabrics

Dye source	Botanical name	Family	Plant part	Percent dye absorption		Wash fastness			
				Untreated dyed fabric	Sericin treated dyed fabric	Untreated dyed fabric		Sericin treated dyed fabric	
						CC	CS	CC	CS
Neem	Azadirachta indica	Meliaceae	Leaves	13.37	14.12	2/3	3	3	3/4
Manjistha	***Rubia cordifolia***	***Rubiaceae***	**Root**	**19.68**	**22.63**	**3**	**3/4**	**3/4**	**4**
Safflower	*Carthamus tinctorius L.*	*Asteraceae*	Flowers	14.19	15.93	3	3/4	3/4	4
Kachnar	*Bauhinia variegate*	*Cesalpiniaceae*	Bark	15.72	17.60	3	3/4	3	3/4

CC: Colour Change, CS: Colour Staining

The data in Table 1 indicates that sericin treated '*manjistha*' dyed fabric exhibited maximum percent dye absorption (22.63) as compared to sericin treated fabric and dyed with other plant sources i.e. *neem,* safflower and *kachnar.* Hence taking into consideration maximum percent dye absorption and wash fastness, *manjistha* was selected for further study.

4.3 Standardization of Different Variables for Sericin Treatment

Different variables of sericin treatment were optimized to enhance the dyeability of cotton fabric with better fastness properties. The sericin concentration, treatment temperature, treatment time, pH, M:L ratio, treatment stage, auxiliaries' combination, concentration of cross linking agent and catalyst, drying temperature and time, curing temperature and time were optimized for treatment of cotton fabric on the basis of maximum percent dye absorption and better wash fastness ratings.

4.3.1 Optimization of sericin concentration: To optimize concentration of sericin, scoured fabric samples were treated with 0.25, 0.50, 0.75, 1.00 and 1.25% sericin concentrations (o.w.f.) keeping other variables constant as given under section [3.5.1]. The treated samples were dyed with standardized dye i.e. '*manjistha*' employing 5% dye material, 45 minutes dyeing time and 90°C dyeing temperature (Teli *et al*., 2004) as per procedure given in [3.5.1].

Table-3 Optimization of sericin concentration

Concentration of sericin (%)	Percent dye absorption	Wash fastness	
		CC	CS
0.25	22.70	3/4	4
0.50	**26.55**	**4**	**4**
0.75	25.63	4	4
1.00	24.37	3/4	4
1.25	24.29	3/4	4

CC: Colour Change, CS: Colour Staining

It is observed from Table 3 that dye absorption was maximum at 0.50 percent sericin concentration which is 26.55 percent but percent dye absorption further decreased i.e. 25.63, 24.37, 24.29 with progressive increase in concentration of sericin at 0.75%, 1.00%, and 1.25% respectively. Wash fastness rating (4) was maximum at both 0.50 and 0.75% for both colour change and colour staining but 0.50 percent concentration of sericin was selected on the basis of maximum percent dye absorption for further investigation.

4.3.2 Optimization of treatment temperature: Treatment temperature is the temperature that is suitable for textile material for fixing of sericin. For optimizing sericin treatment temperature, treatment was carried out at 40, 50, 60, 70 and 80°C.

Percent dye absorption and wash fastness of treated dyed samples at different temperatures are given in the following table:

Table-4 Optimization of treatment temperature

Temperature (°C)	Percent dye absorption	Wash fastness	
		CC	CS
40	22.64	4	4
50	**26.61**	**4**	**4/5**
60	24.02	4	4
70	23.07	4	4
80	23.67	4	4

CC: Colour Change, CS: Colour Staining

It is evident from Table 4 that the percent dye absorption at different treatment temperature i.e. 40, 50, 60, 70 and 80°C was 22.64, 26.61, 24.02, 23.07, 23.67% respectively.

It is clear from the table that the percent dye absorption increased with increase in temperature upto 50°C but further decreased with progressive increase in temperature. Wash fastness was similar at all temperatures except at 50°C *i.e.* grade 4 for colour change and grade 4/5 for colour staining which was comparatively better. Thus, 50°C was selected as the optimum sericin treatment temperature.

4.3.3 Optimization of treatment time: Treatment time is the time, which is required to get the sericin fixed on fabric. For the optimization of sericin treatment time, treatments were given for five different time durations i.e. 15, 30, 45, 60 and 75 minutes.

Table-5 Optimization of treatment time

Time (minutes)	Percent dye absorption	Wash fastness	
		CC	CS
15	22.05	4	4
30	26.50	4	4
45	**28.72**	**4**	**4/5**
60	27.50	4	4
75	25.26	4	4

CC: Colour Change, CS: Colour Staining

It is learnt from the table that the percent dye absorption at 15, 30, 45, 60 and 75 minutes time duration is 22.05, 26.50, 28.72, 27.50 and 25.26 respectively. It is evident from the table that the percent dye absorption increased with increase in dyeing time up to 45 minutes which decreased later with further increase in time. Therefore 45 minutes of sericin treatment time was selected for further study as it exhibited better wash fastness property as compared to the other samples (Table-5).

4.3.4 Optimization of treatment pH: The role of pH is evident from its influence on colour yield and percent dye absorption. Sericin treatment was given at different pH ranging from acidic to alkaline i.e. 5 to 9**.**

Table-6 Optimization of treatment pH

pH	Percent dye absorption	Wash fastness	
		CC	CS
5	23.06	4	4
6	25.51	4	4/5
7	28.67	4	4/5
8	**30.02**	4	4/5
9	30.83	4	4

CC: Colour Change, CS: Colour Staining

Treated fabrics were dyed and dye absorption was calculated for all samples which were 23.06, 25.51, 28.67, 30.02 and 30.83 at pH 5, 6, 7, 8 and 9 respectively. Wash fastness was same at pH 6, 7, and 8 which was very good for colour change and very good to excellent for colour staining. Although dye absorption was highest at pH 9 but due to undesirable hue obtained, pH 8 was selected for research work as the optimum sericin treatment pH (Table 6).

4.3.5 Optimization of treatment stage: Sericin was applied at three stages namely pre, simultaneous and post treatment.

Table-7 Optimization of treatment stage

Stage of treatment	Percent dye absorption	Wash fastness	
		CC	CS
Pre	**30.75**	**4**	**4/5**
Simultaneous	27.62	4	4/5
Post	30.29	4	4/5

CC: Colour Change, CS: Colour Staining

The percent dye absorption was found to be maximum at pre-treatment stage i.e. 30.75 followed by post and simultaneous stage i.e. 30.29 percent and 27.62 percent respectively with equal wash fastness. Hence pre-treatment stage was selected for further investigation (Table-7).

4.3.6 Optimization of material to liquor ratio: For application of sericin, five treatment baths were prepared with different material to liquor ratios i.e. 1:20, 1:30, 1:40, 1:50 and 1:60. It is clear from Table 8 that at different material to liquor ratios i.e. 1:20, 1:30, 1:40, 1:50 and 1:60, the dye absorption obtained was 29.02, 30.68, 30.31, 29.40 and 28.42 percent respectively. It indicated that percent dye absorption increased with increase in M:L ratio till 1:30 but further decreased from 1:40. It can be noticed from the table that there is equal wash fastness at all MLRs except at MLR 1:20 which is lowest. So material to liquor ratio 1: 30 was selected for final application.

Table-8 Optimization of material to liquor ratio

Material to liquor ratio (M:L)	Percent dye absorption	Wash fastness	
		CC	CS
1:20	29.02	4	4
1:30	**30.68**	4	4/5
1:40	30.31	4	4/5
1:50	29.40	4	4/5
1:60	28.42	4	4/5

CC: Colour Change, CS: Colour Staining

4.3.7 Optimization of auxiliaries' combination and concentration: After optimizing M:L ratio, three combinations of chemicals were tried on the basis of reviews i.e. BTCA and sodium hypophosphite, citric acid and sodium hypophosphite and gluteraldehyde and magnesium chloride.

Table-9 Optimization of auxiliaries' combination

Auxiliaries' Combination	Percent dye absorption	Wash fastness	
		CC	CS
BTCA and sodium hypophosphite	31.16	4	4/5
Citric acid and sodium hypophosphite	30.79	4	4/5
Gluteraldehyde and magnesium chloride	29.24	4	4/5

CC: Colour Change, CS: Colour Staining

The data in Table 9 envisaged that all three combinations had shown almost similar wash fastness and percent dye absorption with minor variations i.e. 31.16, 30.79 and 29.24 percent for BTCA and sodium hypophosphite, citric acid and sodium hypophosphite, gluteraldehyde and magnesium chloride respectively. Though percent dye absorption was noticed slightly better with BTCA and sodium hypophosphite but citric acid and sodium hypophosphite were selected because both these chemicals are easily available, are cheap and if used in less quantity are considered eco-friendly.

4.3.8 Optimization of cross-linking agent concentration: To optimize cross- linking agent concentration, 1, 2, 3, 4 and 5 percent concentrations of citric acid were taken.

Table-10 Optimization of citric acid concentration

Concentration of citric acid (%)	Percent dye absorption	Wash fastness	
		CC	CS
1	26.02	4/5	4
2	26.79	4	4
3	27.81	4	4
4	**31.87**	**4**	**4/5**
5	28.29	4	4

CC: Colour Change, CS: Colour Staining

It is revealed from Table 10 that the percent dye absorption increased with the increase in citric acid concentration up to 4 percent but with further increase the dye absorption reduced. The concentration of citric acid which gave better colour and colour fastness properties was selected as the optimum citric acid concentration.

Maximum dye absorption (31.87) was obtained with 4 percent citric acid concentration. Hence, 4 percent was selected as the optimum citric acid concentration for further work.

4.3.9 Optimization of catalyst concentration: Five concentrations of catalyst i.e. sodium hypophosphite were tried. It is clear from Table-11 that the percent dye absorption using 0.5, 1.0, 1.5, 2.0, and 2.5 percent catalyst were 28.94, 31.21, 28.52, 26.65 and 26.08 respectively with better wash fastness at 1 and 1.5 percent i.e. very good for colour change and excellent grading for colour staining. For further investigation 1 percent concentration of catalyst was selected on the basis of maximum dye absorption and wash fastness.

Table-11 Optimization of sodium hypophosphite concentration

Concentration of sodium hypophosphite (%)	Percent dye absorption	Wash fastness	
		CC	CS
0.5	28.94	4	4/5
1	**31.21**	4/5	5
1.5	28.52	4/5	5
2	26.65	4/5	4/5
2.5	26.08	4	4/5

CC: Colour Change, CS: Colour Staining

4.3.10 Optimization of drying temperature: Drying was carried out at 5 temperatures i.e. 60, 70, 80, 90 and 100°C.

Table-12 Optimization of drying temperature

Drying temperature (°C)	Percent dye absorption	Wash fastness	
		CC	CS
60	28.27	4	4/5
70	**30.67**	**4/5**	**5**
80	27.64	4	4/5
90	24.50	4	4/5
100	24.44	4	4/5

CC: Colour Change, CS: Colour Staining

At 60°C drying temperature, percent dye absorption was 28.27, when temperature increased to 70°C, it was 30.67 percent. With further increase from 80°C to 100°C, there was progressive decrease in dye absorption i.e. 27.64, 24.50 and 24.44 percent respectively. Therefore 70°C drying temperature was selected for the study as it gave maximum dye absorption (Table 12).

4.3.11 Optimization of drying time: For optimizing drying time, samples were dried for 2, 4, 6, 8 and 10 minutes.

Table-13 Optimization of drying time

Drying time (minutes)	Percent dye absorption	Wash fastness	
		CC	CS
2	28.70	4	4/5
4	**30.21**	**4/5**	**5**
6	27.95	4/5	5
8	26.21	4	4/5
10	24.07	4	4/5

CC: Colour Change, CS: Colour Staining

It is evident from Table 13 that at 2 minutes of drying time, dye absorption was noted as 28.70% and when duration was increased to 4 minutes dye absorption was 30.21%. With further increase in drying time i.e. 6, 8 and 10 minutes, dye absorption decreased progressively i.e. 27.95%, 26.21%, 24.07% respectively. So at 4 minutes of drying time, there was maximum dye absorption i.e. 30.21%, hence used for further work.

4.3.12 Optimization of curing temperature: For finding optimum curing temperature, the experiment was carried out at 5 curing temperatures i.e. 140, 150, 160, 170 and 180°C.

Table-14 Optimization of curing temperature

Curing temperature (°C)	Percent dye absorption	Wash Fastness	
		CC	CS
140	26.01	4	4
150	28.52	4	4/5
160	**31.53**	**4/5**	**5**
170	26.37	4	4/5
180	23.70	4	4

CC: Colour Change, CS: Colour Staining

It is clear from Table 14 that when curing temperature reached 160°C from 140°C, there was progressive change in dye absorption from 26.01 to 31.53%. But with further increase in curing temperature i.e. 170°C and 180°C, a progressive decline in dye absorption was observed i.e. 26.37% and 23.70% respectively. Wash fastness rating was also comparatively higher at 160°C. Hence 160°C was taken as optimum curing temperature.

4.3.13 Optimization of curing time: For optimizing curing time, treated fabric samples were cured for 5 curing time durations. It is observed from the Table 15 that when sericin is applied with optimized concentrations and conditions of treatment and cured for 1 minute, 28.53 percent dye absorption was obtained and with increase in curing time to 2 minutes there was increase in dye absorption of treated dyed fabric i.e. 31.67%. It can be noticed from table that further increase in time of curing for 3, 4 and 5 minutes resulted in progressive decrease in dye absorption i.e. 30.53, 26.79 and 24.96 percent. Thus 2 minutes curing time was chosen for treatment considering the better wash fastness as well.

Table-15 Optimization of curing time

Curing time (minutes)	Percent dye absorption	Wash fastness	
		CC	CS
1	28.53	4/5	5
2	**31.67**	4/5	5
3	30. 53	4	5
4	26. 79	4	4/5
5	24.96	4	4/5

CC: Colour Change, CS: Colour Staining

4.4 Application of Sericin using Optimized Variables and Dyeing

The sericin treatment was given to scoured cotton fabric using pad-dry-cure method after optimizing concentration of sericin, auxiliaries and treatment conditions. Each sample was pre-treated in a solution containing 0.50 percent sericin, 4% citric acid and 1% catalyst at 50°C for 45 minutes maintaining pH 8 keeping material to liquor ratio 1:30. Afterwards samples were dried in hot air oven at 70°C for 4 minutes and cured at 160°C for 2 minutes. The optimum conditions obtained are given in Table 16.

Table-16 Optimized concentrations and conditions for sericin treatment

Treatment Variables	Optimum conditions
Sericin	0.50 %
Auxiliaries' combinations	citric acid and sodium hypophosphite
Crosslinking agent (citric acid)	4 %
Catalyst (sodium hypophosphite)	1 %
Treatment temperature	50°C
Treatment Time	45 minutes
pH	8
Stage of treatment	Pre-treatment
M:L ratio	1:30
Drying temperature	70°C
Drying time	4 minutes
Curing temperature	160°C
Curing time	2 minutes

After sericin treatment, cotton fabric was wetted out to make it ready for dyeing. *Manjistha* dye was applied on sericin treated fabric using standardized dyeing procedure. (Teli *et al*., 2004).

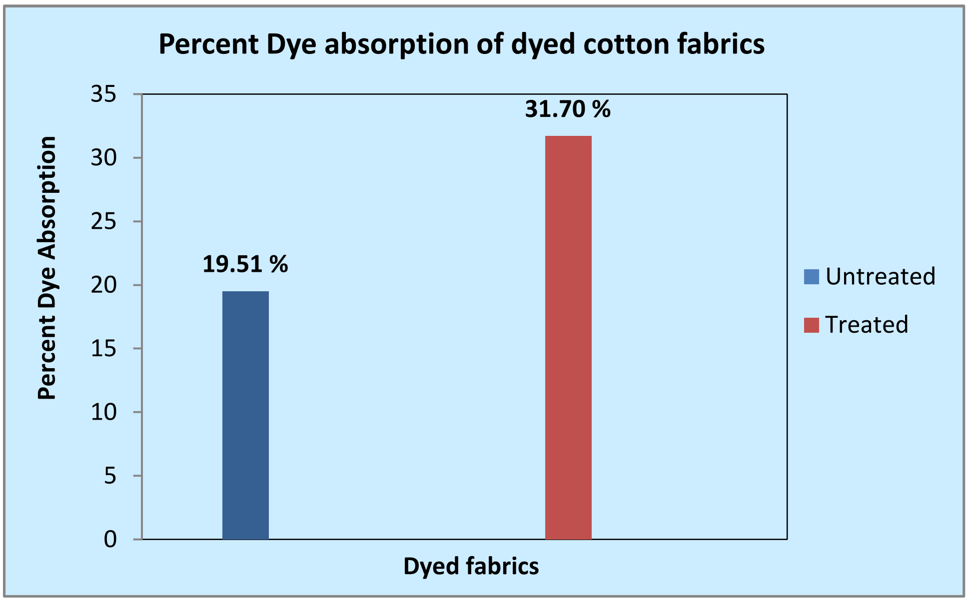


Figure-1 Percent dye absorption of dyed cotton fabrics

It is evident from Figure 1 that percent dye absorption of untreated dyed fabric was 19.51 and for treated dyed fabric it was 31.70 indicating there was a significant increase in dye absorption after sericin treatment.

4.6 Assessment of Colourfastness Properties of Dyed Fabrics

Colourfastness of textile material is of considerable importance to consumers. The fastness depends not only upon the nature and depth of shade of the dyestuff used but also upon the nature of fibre and the treatment given prior to dyeing. The ability of a dye in association with a given substrate to withstand the various agencies such as washing, sunlight, perspiration, crocking etc. in processing or use is called its fastness properties. The findings of colour fastness tests of treated dyed fabric and untreated dyed fabric to washing, light, perspiration and rubbing fastness are given in Table 17. The results being expressed in terms of change in colour and staining of adjacent cotton material.

4.6.1 Wash fastness: It is evident from the Table 17 that the wash fastness grades for colour change was very good to excellent for treated fabric whereas good for untreated fabric. While studying the staining of colour after washing it was observed from the table that colour staining grades for treated and untreated dyed fabric were excellent (5) and fairly good (3/4) respectively on adjacent cotton fabric, which indicated significant positive change in washing fastness grades of sericin treated fabric.

4.6.2 Light fastness: It is evident from Table 17 that light fastness rating for untreated dyed fabric was fair (3) and for treated dyed fabric it was very good (4/5).

<u>COTTON SAMPLES</u>

SCOURED FABRIC

SERICIN TREATED FABRIC

UNTREATED DYED FABRIC

TREATED DYED FABRIC

Table-17 Colourfastness Properties of dyed fabrics

Fabrics	Fastness properties											
	Washing fastness		Light fastness	Perspiration fastness				Rubbing fastness				WMS
	CC	CS	CC	Alkali		Acid		Dry		Wet		
				CC	CS	CC	CS	CC	CS	CC	CS	
Untreated dyed	3	3/4	3	3	3/4	2/3	3	3/4	4/5	3/4	¾	3.32
Treated dyed	4/5	5	4/5	4	4/5	3/4	4	4/5	5	4	4	4.32

CC: Colour Change, CS: Colour Staining, WMS: Weighted mean score

4.6.3 Perspiration fastness: The Table 17 shows the perspiration fastness grades for colour change under acidic and alkaline conditions. Colour change was less in alkaline condition as compared to acidic condition. Under alkaline condition colour change grade for treated fabric was 4 i.e. good and for untreated fabric grading was 3 (fair), however under acidic condition color change was 3/4 (fairly good) and 2/3 for treated and untreated fabrics respectively.

It was learnt that under alkaline condition colour staining was very good (4/5) and fairly good (3/4) for treated and untreated fabrics respectively whereas under acidic condition grades were good (4) and fair (3) for treated and untreated samples respectively.

4.6.4 Rubbing fastness: The Table 17 reveals that the dry rubbing fastness grade for colour change was very good (4/5) in comparison to control fabric i.e. fairly good (3/4) and colour staining were very good (4/5) and excellent (5). The wet rubbing colour change and colour staining grades were same i.e. fairly good (3/4) for untreated fabric and good (4) for treated dyed fabric.

4.7 Colour Measurements of Dyed Fabrics

The CIE LAB values for the sericin treated and dyed cotton fabric under optimum conditions had been compared with untreated dyed cotton sample presented in Table 18.

Table- 18 Colour measurements of dyed fabrics

Fabrics	L	a*	b*	C	E	K/S
Untreated dyed	65.024	24.476	1.281	24.509	-	3.2
Treated dyed	58.518	27.008	4.561	27.390	+7.54	5.7

The Table 18 depicts that there was decrease in L* value of the dyed sample from 65.024 to 58.518 which indicates the treated fabric is darker as compared to untreated fabric. The a* value increased from 24.476 to 27.008 which indicated redness in the sample. The yellowness of the colour increased (indicated by higher b* values) on treatment. Samples treated with sericin were brighter in colour than the control sample (as indicated by higher C* values). Total colour difference was 7.54 in treated dyed sample which is indicated by E. The increased K/S values depicted that the dye uptake by the samples increased on treatment i.e. 5.7 as compared to untreated dyed i.e. 3.2.

4.9 Testing of Physical and Performance Properties of Dyed Fabrics

Physical properties of untreated and treated dyed fabrics were measured to assess the changes in properties using various instruments.

Preliminary properties: Data in the Table 19 is given regarding the preliminary properties i.e. fabric count, weight and thickness.

The results revealed that the fabric count of the untreated dyed fabric was 103 for warp and 58 for weft direction respectively. For treated dyed, it was noticed that the fabric count increased to 104 and 60 for warp and weft direction respectively. When the mean fabric count of treated dyed fabric was compared with that of untreated fabric it was found that there was marginal increase (1.86 percent).

Table-19 Preliminary properties of dyed fabrics

Preliminary Properties		Untreated dyed fabric		Treated dyed fabric		Percent change
		Mean ± S. E	C.V.	Mean ± S. E.	C.V.	
Fabric count (threads/inch)	**Warp**	103±0.51	1.80	104±0.50	2.10	+1.86
	Weft	58±0.50	1.40	60±0.50	1.20	
Fabric weight (gm/sq.m)		121.8±0.79	1.46	123.2±.058	1.05	+1.14
Thickness (mm)		0.33± 0.01	1.63	0.34± 0.01	1.58	+3.03

+ = increase; - = decrease; S.E.= Standard error; C.V.= Coefficient of Variance

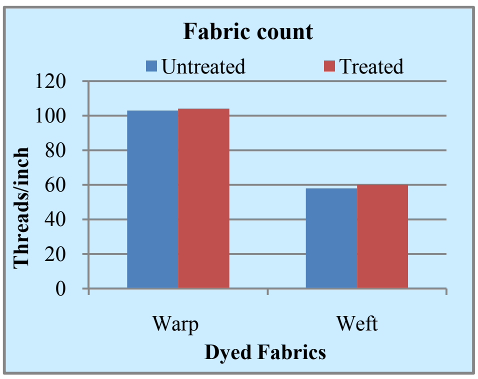


Figure- 2. Count of dyed cotton fabrics

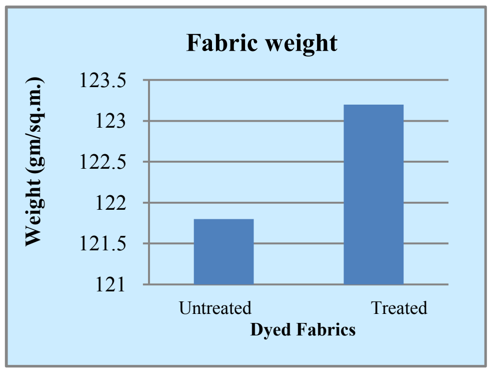


Figure- 3. Weight of dyed cotton fabrics

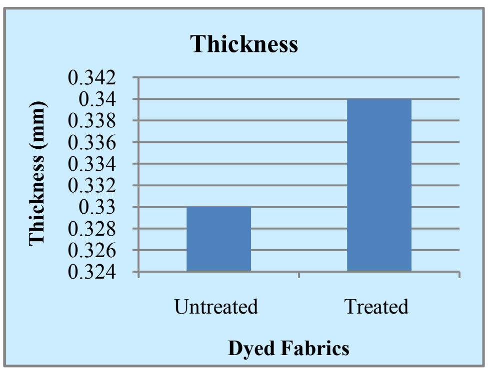


Figure -3. Thickness of dyed cotton fabrics

The results in the Table 19 show that the fabric weight also increased after treatment with sericin, which was 121.8±.79 g/sq.m. to 123.2±.58 g/sq.m. Percent increase in fabric weight of treated dyed fabric was only 1.14 as compared to untreated dyed fabric.

Table 19 narrates that the thickness of untreated dyed fabric was 0.33±0.01 and after application of sericin and dyeing it was 0.34±0.01. The thickness also increased to 3.03 percent only.

Physical Properties: From Table 20 it is learnt that the treated dyed test samples on sericin treatment showed increase in bending path *i.e.* from 3.54 cm to 3.65 cm, which means that there was slight increase in stiffness on application of sericin. Further, it is noticed that the warp-way bending length increased from 3.93 ±0.20 cm to 3.99± 0.17 cm when treated with sericin. A trend of increase in bending length was also observed in weft-way samples *i.e.* 3.15 ±0.17 cm for untreated dyed and 3.32±0.12 cm for treated dyed samples. There was only 3.01 percent change in bending length of treated dyed fabrics. Flexural rigidity of the untreated fabric was found to be 152.6 mg-cm and 164.1 mg-cm for treated dyed fabric with 7.5 percent increase.

Table-20 Physical properties of dyed fabrics

Physical Properties		Untreated dyed fabric			Treated dyed Fabric			Percent change
		Mean ±S.E.	C.V.	Mean	Mean ±S.E.	C.V.	Mean	
Bending length (cm)	Warp	3.93 ±0.20	11.2	3.54	3.99± 0.17	9.3	3.65	+3.01
	Weft	3.15 ±0.17	12.00		3.32±0.12	8.10		
Flexural Rigidity (mg-cm)		-	-	152.6	-	-	164.1	+7.5
Tensile strength (kg)	Warp	20.5 ±0.71	7.7	18.26	24.12±0.32	4.0	21.70	+18.83
	Weft	16.01± 0.80	11.2		19.28±0.38	5.6		
Elongation (%)	Warp	24.5± 0.60	5.4	27.68	23.46±0.70	6.6	25.38	-8.3
	Weft	30.87± 0.52	3.5		27.30±0.32	2.8		
Crease recovery degree	Warp	93.8 ±0.58	1.38	92.9	104.2±0.58	1.24	102.5	+9.7
	Weft	92 ±0.70	1.71		100.8±0.66	1.46		
Wettability (Sec)	-	5.65± 0.05	1.56	-	2.88±.025	1.34	-	+49.02
Air permeability ($cc/sec/cm^2$)	-	24.62±.005	-	-	22.07±.075	-	-	-10.35

+ = increase; - = decrease; S.E.= Standard error; C.V.= Coefficient of Variance

Table 20 also reflects tensile strength of untreated dyed fabric i.e. 18.26 kg and sericin treated dyed fabric i.e. 21.70 kg. Increase of 18.83 percent in tensile strength was observed when treated with sericin as compared to untreated fabric. However, warp-way tensile strength was greater in both cases of untreated and sericin treated test sample as compared to the weft-way fabrics.

Table 20 depicts the elongation of test samples. In general the weft-wise elongation was greater than warp-wise. The percent warp-way elongation decreased with sericin treatment i.e. 23.46 ±0.70 percent compared to control 24.5± 0.60 percent. Similar trend was noticed in weft elongation *i.e.* 27.30± 0.32 percent for treated dyed and 30.87± 0.52 percent for untreated dyed fabric. Untreated dyed samples exhibited greater elongation compared to sericin treated dyed samples. The mean elongation of treated and untreated dyed fabrics was 25.38 percent and 27.68 percent respectively. It is evident from table that when elongation of untreated dyed samples was compared to treated dyed sample a reduction of 8.3 percent was noticed.

Table 20 also narrates the crease recovery of the untreated and sericin treated dyed samples. In general the warp-way recovery was found to be higher than weft-way and the cloth crease recovery was the resultant of warp and weft recovery angle. The warp recovery was slightly higher for treated dyed fabric (104.2 ±0.58 degree) compared to untreated sample dyed with *manjistha* (93.8 ±0.58 degree). Similarly there was increase in crease recovery degree in weft way for treated dyed fabric i.e. 100.8± 0.66 degree with respect to untreated dyed fabric i.e. 92 ±0.70 degree. There was 9.7 percent increase in the crease recovery angle of treated dyed fabric i.e. 92.9° to 102.5° for untreated dyed fabric and treated dyed fabric respectively.

Wettability of untreated dyed fabric and treated dyed fabric was found to be 5.65± 0.05 and 2.88±.025 seconds respectively with 49.02 percent increase in wettability upon sericin treatment.

The air permeability of the treated dyed fabric was observed to be 22.07±.075 $cc/sec/cm^2$ with a decrease of 10.35 percent from untreated dyed fabric with air permeability of $24.62cc/sec/cm^2$. (Table 20).

3.9.2 Performance property: Sericin treated, *manjistha* dyed and sericin treated *manjistha* dyed fabrics were tested for UV protection property. The data in the Table 21 shows that Ultraviolet Protection Factor (UPF) of scoured cotton is good which increased with sericin treatment from 14.25 to 22.39 which is also falling in good UV protection class. *Manjistha* dyed fabric has very good UV protection and UPF value is 32.6. When sericin treated fabric was subsequently dyed with *manjistha* (treated dyed) the UP protection rating increased upto 48.4 which means fabric offered excellent UV protection.

Table -21 Ultraviolet Protection property of dyed fabrics

Fabric	**UV Protection Property**		
	UPF	**UPF rating**	**UV Protection class**
Scoured cotton	14.25	15	Good
Sericin treated cotton	22.39	25	Good
Untreated dyed fabric	32.6	30	Very good
Treated dyed fabric	48.4	50	Excellent

15-24: Good protection, 25-39: Very good protection and 40-50, 50+: Excellent protection.

CHAPTER-V

DISCUSSION

Due to non-biodegradable, noxious and dangerous nature of synthetic dyes for body contact, its use in different fields has been banned by many countries. As a result, reapplication of natural dyes on textiles has increased considerably on account of their high compatibility with environment, relatively low toxicity and allergic effects, as well as availability of various natural colouring sources. Almost all natural dyes need to be fixed with mordants. Although natural mordants exist but the toxic and environmental-unfriendly metallic mordants are still the major mordants used nowadays due to low cost and easy availability. Unexhausted metallic mordant remains in the residual dye-bath causing serious effluent disposal problem. Due to toxicity and environmental unfriendliness of metallic mordant, several natural compounds were studied to substitute metallic mordant recently.

One of the natural components is sericin which forms a film and increases the number of reaction sites on the substrates for dyes. In the textile industries, sericin is exclusively released as the unutilized by-product from the process of making sericin-free fibroin fibers. Sericin consists of polar side chain made of hydroxyl, carboxyl and amino groups that enable easy cross-linking, copolymerization and blending with other polymers. If sericin can be utilized in dyeing of textiles from the degumming waste liquor, it will represent a significant source of profit for silk cultivators, not to mention the beneficial effect of waste reduction for pollution prevention. It was therefore thought to be of interest to investigate the roles and effects of sericin fraction of silk containing amino acid, when used on cotton for the improvement of its dyeability.

To achieve the objectives proposed in the research plan, bleached cotton fabric and one natural dye material was selected on the basis of maximum dye absorption and better wash fastness. For application of sericin on fabric, different parameters were optimized. After the application of the sericin using optimized variables, the treated fabric was dyed. The untreated and treated dyed fabrics were tested for colourfastness, physical and performance properties of the. The chapter presents the discussion regarding the findings of the study under following sub-heads.

5.1 Selection of a Natural Dye

5.2 Standardization of Different Variables for Sericin Treatment

5.3 Application of Sericin using Optimized Variables and Dyeing

5.4 Assessment of Colorfastness Properties of the Dyed Fabrics

5.5 Colour Measurements of the Dyed Fabrics

5.6 Physical and Performance Properties of the Dyed Fabrics

5.1 Selection of a Natural Dye: Natural dyes have aromatic hydroxyl groups in their molecular structures which enable them to ionize in water giving dye anions. Thus, the number of positive charges on cotton and the degree of ionization of dye is a decisive factor in determining the magnitude of ionic attraction between dye anions and fibre cations and subsequently the obtained colour strength. *Manjistha* dye was selected from four standardized natural dyes as it gave the maximum dye absorption and better wash fastness upon treatment with sericin, which may be due to acidic nature of the dye which means there might be higher ionic attraction between dye anions and the positively charged cotton fibres. Zhou *et al.* (2012) found that the sericin largely developed the positive charge on cotton fibers. The results revealed that the uptake of annatto dye on modified cotton fibers occurred via electrostatic attractions between the anion of the dye and the cationic segments on the modified cotton fibers.

5.2 Standardization of Different Variables for Sericin Treatment: For application of sericin, treatments were given to optimize sericin concentration, treatment temperature, treatment time, pH, M:L ratio, treatment stage, auxiliaries' combination, concentration of cross linking agent and catalyst, drying temperature and time, curing temperature and time for Pad-Dry-Cure method on the basis of maximum percent dye absorption and better wash fastness property.

5.2.1 Optimization of sericin concentration: Five different concentrations of sericin i.e. 0.25, 0.50, 0.75, 1.00 and 1.25 percent were applied on cotton fabric. The results showed that as the concentration of sericin increased from 0.25 to 0.50 percent there was increase in dye absorption from 22.70 to 26.55 percent but further decreased progressively upon increasing concentration in the padding baths and wash fastness was maximum at 0.50 and 0.75 percent sericin concentration. So 0.50 percent concentration was selected for final application. The reason might be that sericin acted as glue at concentrations greater than 0.50%, hindering the dye uptake thus negatively affecting the dyeing performance. The findings of study done by Zhou *et al*. (2012) supported the results that when the sericin concentration was less than 0.5-0.6 % (w/v) (1gm per 100 ml), the amount of natural dye adsorbed on modified cotton increased due to the reason that when sericin concentration was more than 0.5-0.6 %, too much sericin might have blocked the gaps between cotton fibers, which may have made it difficult for the dye to spread into the cotton fibers and decreased the dye uptake. Sundrarajan *et al*. (2012) found that 1.0 % concentration of chitosan produced better dye uptake.

5.2.2 Optimization of treatment temperature: The optimum treatment temperature was found by varying the temperature *i.e*. 40, 50, 60, 70 and 80°C. The percent increase in dye absorption was noticed from 22.64 to 26.61 with increase in treatment temperature upto 50°C. The treatment temperature of 50°C was chosen because at this temperature there was better wash fastness along with maximum percent dye absorption. The reason might be that under higher temperature some of the sericin might have desorbed into the treatment bath leading to less modification of cotton fabric which in-turn affected dyeability of the fabric. The results are in line with Marie *et al*. (2011) that above 60°C, the stability of the cationizing agent on cotton fabric was negatively influenced. In case of acid safflower extract, bright yellow colour was obtained at lower than 60°C temperature whereas at higher temperature, the obtained yellow colour was dull and reddish.

5.2.3 Optimization of treatment time: The optimization was done by observing the dye absorption and wash fastness at five treatment times i.e. 15, 30, 45, 60 and 75 minutes. It was observed that the highest dye absorption and wash fastness of treated fabric was observed at 45 minutes treatment time. This might be due to the reason that the saturation point for absorption of sericin was at 45 minutes and no further increase in the uptake of the sericin was observed after 45 minutes of treatment time. It was revealed by Zhou *et al*. (2012) that amount of natural dye i.e. annatto adsorbed on cotton greatly increased when modification time was less than 60 minutes. Hence the results of the present study are in accordance to these findings.

5.2.4 Optimization of treatment pH: Treatment was given at pH 5, 6, 7, 8 and 9 and dye absorption along with wash fastness was noticed for all samples. Maximum dye absorption was observed at pH 9 followed by pH 8. However at pH 9 undesired tone of the colour was developed so pH 8 was selected for maximum dye absorption. The reason might be that sericin treatment at pH 8 has improved alkaline nature of cotton fabric and helped better uptake of *manjistha* dye which is acidic in nature. Wash fastness was same at pH 6, 7 and 8 which was good for colour change and very good for colour staining. Bains and Grewal (2007) reported optimum pH 7 at which the enzymatically treated cotton samples dyed with *bansa* dye were darker than untreated samples. The darker shade on enzyme pretreated fabric was attributed to the hydrolysis of cellulosic fibres during enzymatic treatment and removal of projecting fibres that help in better dye absorption.

5.2.5 Optimization of treatment stage: Sericin treatment was given at three stages i.e. pre, simultaneous and post treatment stage. The maximum dye absorption was found at pre treatment stage i.e. 30.75 percent followed by post and simultaneous i.e. 30.29 percent and 27.62 percent respectively with same wash fastness grades. Hence pre-treatment stage was selected for further investigation. The might be due to the reason that drying and curing of treated samples was carried out after dyeing during simultaneous and post treatment stage which made sericin treatment less durable resulting in lower dye uptake of treated samples. The results of the study were in agreement to the findings of Wang *et al*. (2006), Cheng *et al*. (2011) and Zhou *et al*. (2012) in which they have reported that pretreatment given to wool, jute and cotton fabric using sericin exhibited better dye uptake.

5.2.6 Optimization of material to liquor ratio: To optimize the material to liquor ratio (MLR) for application of sericin through Pad-Dry-Cure method, five ML ratios i.e. 1:20, 1:30, 1:40, 1:50 and 1:60 were taken. It revealed that the dye absorption is maximum at 1:30 ML ratio. The reason for the highest dye absorption observed at material to liquor ratio 1:30 might be that at MLR 1:30, the quantity of liquor was optimum for the absorption of contents of the liquor to the fabric and MLR less than that did not result in uniform absorption of the contents from the treatment bath. With increase in material to liquor ratio the dilution of contents increased resulting in less deposition of sericin on the fabrics. Zhou *et al*. (2012) reported that sericin treatment given to cotton fabric at m:l ratio of 1:40 had desirable effect on dye absorption and wash fastness.

5.2.7 Optimization of auxiliaries' combination and concentrations: There is a shortage of those groups that can result in covalent crosslinking with cellulose in the sericin protein molecular structure, which will lead to relatively low fixation rate of sericin. In order to improve the modification effect, cotton was treated with crosslinking agent and catalyst. Although BTCA and sodium hypophosphite gave maximum dye absorption but citric acid and sodium hypophosphite were selected for final treatment because BTCA is very expensive and it resulted in excess weight gain of treated fabric whereas citric acid was cheap and easily available. Xing *et al*. (2011) reported that polycarboxylic acid is a kind of medium strong acid, especially the acidity of BTCA is stronger than citric acid. Consequently, solution containing polycarboxylic acid could lead to serious damage after curing at high temperature. Wang *et al*. (2005) reported that sodium hypophosphite was the most effective catalyst for polycarboxylic acid finish.

5.2.8 Optimization of drying temperature and time: To determine the optimum drying temperature for the final application of sericin, optimum concentration of sericin, citric acid and sodium hypophosphite at optimum MLR, were applied with five drying temperature variations i.e. 60, 70, 80, 90 and 100^0C. For cotton fabric the optimum drying temperature was observed as 70^0C for 4 minutes optimum time duration for drying. Drying at higher temperature for longer duration might cause hydrolysis of sericin which makes less sericin available for modification leading to the less dye absorption and poor wash fastness of cotton fabric. Das *et al.* (2014) reported that the padded and squeezed cotton fabrics were dried at a temperature of 95°C for 5 min and subsequently cured at 140°C for 5 min.

5.2.9 Optimization of curing temperature and time: Temperature and time of curing are important variables to control and obtain good crosslinking in treated cotton fabrics. Curing was done to diffuse the sericin solution into the fibre polymer system for better affinity. For maximum dye absorption and wash fastness property, the treated fabrics were cured for 1 to 5 minutes. The maximum dye absorption was observed when cured for 2 minutes at 160^0C. Sericin being aromatic in nature tends to cause fabric yellowing at high temperature for longer duration which in turn makes the obtained colour dull. Xing *et al*. (2011) reported that when the curing temperature was enough, the yellowing of the fabric was serious which could cause severe damage for fabrics.

5.3 Application of Sericin using Optimized Variables and Dyeing

Sericin application was given to scoured cotton fabric by Pad-Dry-Cure method after the optimization of the variables. Cotton fabric was first dipped in the solution having optimum ML ratio 1:30, pH 8, and sericin concentration (0.50 percent) with optimum concentration of citric acid (4 percent) and sodium hypophosphite (1 percent). The fabric was impregnated into the liquor for 45 minutes at 50°C in oven. Afterwards the fabric was pressed between the squeezing rollers of the padding mangle machine. The fabric was dried at 70°C for 4 minutes and cured at 160°C for 2 minutes. After curing, fabric was dyed using 5 percent dye concentration of *manjistha* as per standardized dyeing procedure given by Teli *et.al*. (2004). Wang (2006) reported that the dyeing temperature decreased, dye-uptake increased and washing fastness improved when sericin treated wool fibers were dyed with reactive dye.

5.4 Assessment of Colourfastness Properties of the Dyed Fabrics

Colourfastness is one of the significant customer requirements for the modern textile industry. Customers generally expect textile products that will not change in colour when subjected to different usage conditions. It is known that the colour fastness of the dyes is affected by the photochemical interaction of the dye-material complex or to the pronounced tendency of the dye molecules to aggregate inside the fiber and this in turn depends on the type of the fabric and the dye used and also the pretreatment.

Colourfastness of treated dyed fabric improved for all fastness i.e. washing, light, perspiration and rubbing as compared to the untreated dyed fabric**.** The results were supported by findings of Zhou *et al*. (2012) that both wash and rubbing fastness of sericin treated cotton fabrics dyed with annatto dye were higher than those of untreated cotton fabrics. Similarly Das *et al.* (2014) reported that prior modification of cotton with sericin followed by dyeing of sericin modified cotton with reactive dyes led to balanced improvements in the properties of the dyed substrates with respect to fixation of reactive dyes, retention of tenacity, colour fastness to light, wash and rubbing. Haggag *et al*. (2009) also found significant improvement in fastness properties of the treated fabrics with hydrogen peroxide/sodium sulphite/sericin after being printed with acidic, reactive and basic dyes. They also reported that the dyes used were chemically bonded or cross-linked to wool by sericin in the presence of epichlorohydrin. The results of the study conducted by Wang (2006) indicated that the dyeing ability of reactive dyed sericin cross-linked wool fiber improved obviously.

5.5 Colour Measurements of the Dyed Fabrics

Upon sericin treatment the dyed fabric was darker in colour indicated by decrease in L* value. The higher a* values indicated the higher content of red in the treated sample and sample was brighter in colour than the control sample as indicated by higher C* values. The higher K/S value indicated the higher colour strength of sericin treated dyed sample. A study conducted by Kittinaovarat (2004) revealed that the chitosan treated cotton fabric showed improved depth of shade on dyed fabrics. This may be because the chitosan treated cotton fabric provided more dye sites to attach more dye than that of the untreated fabric. Increasing dye solubility means higher ionic attraction between dye anions and the positively charged cotton fibres leading to higher colour strength. Moses and Ramasamy (2004) also reported that the pre-treatment of jute and jute-cotton blend fabric with cellulase enzyme helped to improve the brightness of colour, dye uptake and also increased wash and light fastness properties.

5.6 Physical and Performance Properties of the Dyed Fabrics

Fabric count

The fabric count of the treated cotton fabric slightly increased. Which may be due to the presence of the crosslinking agent in the treatment that binds the cellulose molecules by the crosslinking bonds bringing the molecules close together and resist its movement here and there, as a result yarns come closer to each other, hence the fabric count increased. Mulasavalagi (2005) found that density of softener finished samples was greater in both warp and weft direction than the control because the treated fabric might have turned the yarns finer and due to wet treatment the yarn might have densely consolidated in the fabric. This increase was higher in samples treated with cationic softener and may be because the cationic softeners are known to impart high degree and an exceptional soft handle by their positively charged molecules which are deposited on the negatively charged fibre surface. Moreover the softness increased the yarn count that in-turn lead to the compactness of weave.

Fabric Thickness

The thickness of the treated dyed fabric increased slightly in comparison to the untreated dyed fabric. The thickness of the cotton fabric treated with sericin and dyed with *manjistha* increased by 3.03 percent. It may due to the absorption of the sericin and dye within the fibre. Thus the thickness of the fabric increased. Mulasavalagi (2005) also reported that cationic treatment increased the cloth thickness.

Fabric weight

The weight of the cotton fabric increased from 121.8 to 123.2 g/m^2 after sericin treatment and dyeing. The increase in weight may be due to the absorbance of the sericin and auxiliaries into the fabric. This may be due to that the presence of sericin and citric acid molecules in the treatment increased the weight per unit area of the fabric. They bind with the fabric by crooslinking reaction by the hydrogen bonds. The result of the study was supported by Chattopadhyay *et al*. (1998) who found that weight gain increased by increasing the citric acid concentration in presence of any of the catalyst used. This is attributed to higher amount of attachment of citric acid with cotton through crosslinking.

Bending Length and flexural rigidity

The results revealed that there was 3.01 percent increase in bending length and 7.5 percent increase in flexural rigidity of the sericin treated dyed fabric. This might be due to the crosslinking reaction which makes the fabric stiff. Due to the stiffness of the fabric there was increase in the bending length of the fabric. Chattopadhyay *et al*. (1998) supported the results with the fact that the crosslinking improved the dimensional stability and affected the softness of the material negatively, which ultimately increased bending length and bending modulus of treated fabric, irrespective of catalyst used. The findings were also supported by Ali *et.al.* (2011), bending length increased after chitosan application using Pad-Dry-Cure method as compared to the untreated fabric.

Tensile strength

The results of the study showed that the tensile strength for sericin treated dyed fabric increased to 21.70 kg from 18.26 kg of the untreated dyed fabric. Possible reason is that the compactness of the weave and bonding in the fibres after sericin treatment might have resulted in increase in tensile strength of treated fabric. The results of the study were in accordance with the Jassim *et al.* (2010) indicating that the tensile strength increased after treatment with 2% sericin. Whereas contradicting the results of Xing *et al*. (2011) who reported that all treated samples yield lower breaking strength compared with those of untreated samples. Because cotton fabric is alkali fast but not resistant to acid, strength losses were due to acid hydrolysis reactions of cellulose macromolecules.

Elongation

The results of study expressed that the elongation of the treated dyed fabric decreased after application of sericin from 27.68 to 25.38 i.e. 8.3 percent. It might be due to the stiffness of the fabric which was obtained by crosslinking after application of sericin. When the fabric becomes stiff the elongation of the fabric decreased due to the restricted movement of the cellulose molecules within the fiber. As a result of it, instead of stretching, the fabric breaks off. Mulasavalagi (2005) also concluded that there was reduction in elongation (percentage) when fabric was treated with enzymes.

Crease recovery

The crease recovery angle was 92.9 degree for untreated dyed fabric whereas it increased to 102.5 degree for sericin treated dyed fabric. Increased crease recovery might be due to the use of crosslinking agents, which can covalently crosslink with the adjacent cellulose chains within cotton fibers. The new crosslinking bonds formed in the treatment process are stronger than the former hydrogen bonds. The new crosslinks held when the fabric under distortion and moist conditions; the bonds pull the cellulose chains back into position after removal of a distorting force so that the fabric resisted wrinkling. Polycarboxylic acids react with hydroxyl groups of cellulose to form ester crosslinks connecting adjacent cellulose chains in a three dimensional network inside the cellulosic fibers. The researchers have reported a high degree of wrinkle resistance as well as smooth drying properties in cotton fabrics treated with polycarboxylic acids having three or more carboxyl groups, in the presence of alkali metal salts of a phosphorus-containing acid such as sodium hypophosphite, disodium phosphate or monosodium phosphate. The results are supported by the findings of Xing *et al*. (2011) that the wrinkle recovery angle increased from 163 to 195° and wet wrinkle recovery angle increased from 135 to 195 degree. Treated sample showed a dramatic improvement in wrinkle recovery angle (warp and weft), especially in the wet state. There were hydroxyl groups on the surface of cellulose, hence easy to form hydrogen bond. When samples were wrinkled, cellulose molecules chains slipped, which was the reason that the wrinkle recovery angle of cellulose fabric was low. Hydroxyl groups of the fabric treated with polycarboxylic acid cross-linked with the carboxyl of citric acid and form reticulation structure. Therefore the number of hydroxyl groups to form hydrogen bond decreased which restricted the relative slippage among the cellulose molecules. Consequently, the wrinkle recovery angles increased.

Wettability

The wetting property of sericin pretreated dyed fabric increased 49.02 percent as compared to untreated dyed fabric. It may due to the reason stated by Kongdee *et al*. (2005) that sericin has been found to improve moisture regain, water retention and reduce electrical resistivity of cotton fibers. The results of the study are in line with the findings of Kurioka *et al*. (2011) upon treatment with sericin (1.2%) solution containing citric acid (9%) as a cross-linking agent, the hydrophilic and thermal properties of cotton fabrics improved. The treated fabrics absorbed more water than the non-treated fabrics and the water diffusion time decreased.

Air permeability

The air permeability of treated sample decreased after the application of the sericin in comparison to the untreated dyed sample from 24.62 to 22.07cc/sec/cm^2 with 11.55 percent decrease. The reason of decreasing air permeability might be higher fabric thickness and more number of fibres per unit area resisting air flow which led to lower air permeability. Another possible reason is that cotton fibres are hydrophilic, hence absorbed a greater amount of sericin. The absorbed content within fibers could block the air spaces between fibers or yarns, resulting in decrease in air permeability. The results are in agreement to the findings of Ali *et.al.* (2011), they reported significant decrease in the air permeability of the chitosan treated fabric samples as compared to untreated ones. Significant loss in air permeability was also observed by Hooda (2012) in all the neem and *aloe vera* treated cotton fabrics as compared to the untreated samples.

Ultraviolet Protection property

Dyed fabrics can absorb significant amount of UV radiation and have protective effect. The effect of *manjistha* dye on ultraviolet protection of cotton fabric was investigated. It was observed that UPF value of fabric increased upto very good UV protection class on dyeing with *manjistha*. The increase in UPF value after dyeing is credited to the ability of dyes to selectively absorb visible light. Most dyes absorb light in the region between 400 and 700 nm and the absorption band extends to UV radiation spectrum i.e. 280 nm-400 nm for all dyes. Fabrics, especially when dyed, can absorb significant amount of UV radiation and have protective effect. UV protection capability turned from good to excellent when sericin treated fabric was dyed with *manjistha*, which can be attributed to the inherent UV resistant property of sericin and darker shade of treated dyed fabric. Pailthorpe (1998) observed that generally for the same fabric construction and dyestuff, the darker the shade, the greater is the fabric UPF. Gulrajani *et al.* (2008) and Xu *et al.* (2011) found improvement in the anti-ultraviolet property of sericin-treated fabrics.

CHAPTER-VI

SUMMARY AND CONCLUSIONS

Dyeing of textiles with natural dyes is a craft practiced by ancient people. The recent green movement has created awareness in sustainable practices in the apparel and textile industries and brought a renewed interest in natural dyes as an eco-friendly co-alternative to the toxic synthetic dyes currently being used in the textile and craft industries. The art of dyeing with natural dyes has gained momentum, not only for safety of health and environment but also for their beauty and novelty.

Cotton is the most widely used textile material because of its properties such as relatively low cost, fine cross section, high strength, durability, thermal stability, good mechanical properties, moisture absorbency and comfortable to wear. Cotton can be modified by a number of techniques to improve their performance characteristics such as dye fixation, soil repellency, crease resistance and flame retardancy. Chemical modification of cotton will improve their dye ability, but at the same time it will pollute the environment to greater level. A good dyer puts efforts to prevent dye from being hydrolyzed and try to obtain dyed goods with minimum loss of dye. Therefore, dyer always attempts to improve colour yield and higher dye fixation during cellulosic dyeing but this has remained a challenge. Over recent years, cationizing cellulose by the cationic agent has been increasing and shows significant advantages in terms of improved dyeing properties.

Recently, sericin has been utilized for its incorporation into cotton fabric prior to the application of dyes. About 50000 tons of sericin is produced annually during degumming of silk. It was therefore thought to be of interest to investigate the roles and effects of sericin fraction of silk containing amino acid, when used as a natural modifying agent under the influence of an esterification catalyst to modify cotton for the improvement of its dyeability. Thus, sericin could be the investigative focus of interest to be used as a biodegradable product with significant finishing effects, because of its available properties and reactivity. The present study ‘Effect of sericin treatment on dyeability of cotton fabric using natural dye’ was carried out to see the effect of sericin on dyeability of cotton fabric which can make the dyeing process ecofriendly. Moreover, utilization of sericin could be profitable to the farmers engaged in sericulture. Therefore the present research was undertaken with the following objectives:

1. To optimize the conditions for sericin treatment and apply on cotton fabric.
2. To dye the treated fabric with natural dye and to assess the colour fastness properties.
3. To study the physical properties of dyed fabric.

Methodology:

The bleached cotton fabric suitable for apparel purpose was desized and scoured which resulted in a loss of approximately 5 percent of fabric weight due to removal of small portion of starch, hemicelluloses and lignin. However, the treatments improved the softness, handle and appearance of cotton material. The scoured fabric was used for further treatment. To achieve the objectives proposed in the research plan, cotton fabric and one dye was selected out of four already standardized natural dyes on the basis of maximum dye absorption and better wash fastness.

The parameters of sericin treatment were optimized on the basis on maximum dye absorption and better wash fastness. The variables viz. sericin concentration, treatment temperature, treatment time, pH, MLR, concentration of crosslinking agent and catalyst, drying temperature and time, curing temperature and time, exhibiting maximum dye absorption and better wash fastness were optimized by varying the trial proportions. After optimization of different variables, the selected fabric was treated with sericin using optimized variables. Treated and untreated fabrics were dyed using natural dye *manjistha* (*Rubia cordifolia*). Colourfastness properties regarding washing, sunlight, perspiration and rubbing were evaluated employing BIS standard tests. Effect of sericin treatment on hue, value and intensity of the colour of dye was investigated by calculating CIE LAB values. Physical and performance properties of dyed fabric regarding fabric count, weight, thickness, flexural rigidity, tensile strength, elongation, wetting, air permeability and ultra-violet property were studied for treated and untreated dyed fabrics to analyze the effect of sericin on the fabric in relation to its dyeability. The important findings of the study are summarized as follows:

Major Findings:

Optimization of Variables for Application of Sericin on the Cotton Fabric

- Sericin treatment was given on scoured cotton fabric using optimized variables i.e. 0.50% sericin, 4 % crosslinking agent (citric acid), 1% catalyst (sodium hypophosphite) on the basis of weight of fabric at 50°C of treatment temperature for 45 minutes at pretreatment stage keeping M:L ratio 1:30 maintaining pH 8, on the basis of maximum percent dye absorption and wash fastness.
- Sericin treated fabric when dried at 70°C for 4 minutes and cured at 160°C for 2 minutes exhibited maximum percent dye absorption and wash fastness.
- Percent dye absorption of sericin treated dyed fabric significantly increased from 19.5 to 31.7 percent after application of sericin using all the optimized variables.

Colourfastness Properties of Dyed Fabrics

- The washing fastness grades for colour change and colour staining were 4/5 i.e. very good and 5 i.e. excellent for treated dyed fabric whereas 3 i.e. fair for colour change and 3/4 i.e.

fairly good for colour staining for untreated dyed fabric, indicating significant positive change in washing fastness grades of sericin treated dyed fabric.

- The light fastness grade for untreated dyed fabric was fair (3) and for treated dyed fabric it was very good (4/5).
- The perspiration fastness grades for colour change and colour staining were 4 and 4/5 respectively for treated dyed fabrics and 3 and 3/4 respectively for untreated dyed fabrics under alkaline perspiration. However under acidic condition color change grades were 3/4 and 2/3 and colour staining grades were 4 and 3 for treated and untreated dyed fabrics respectively.
- The rubbing fastness grade for colour change was very good (4/5) in comparison to control fabric i.e. fairly good (3/4) and colour staining were very good (4/5) and excellent (5) for untreated dyed and treated dyed fabric. The wet rubbing fastness grade for colour change and colour staining grades were fairly good (3/4) for untreated fabric and good (4) for treated dyed fabric.

Colour Measurements of Dyed Fabrics

- All the sericin treated dyed samples were darker than the untreated dyed samples indicated by lower L* values and increase in a* and b* values indicated that the sample became redder and yellower after sericin treatment.
- Fabric treated with sericin was brighter in colour than the untreated fabric as indicated by higher C* values.
- Total colour difference of 7.54 was observed in treated dyed fabric from untreated dyed fabric.
- The colour strength of cotton fabric increased on treatment as compared to untreated dyed fabric as indicated by higher K/S value.

Physical and Performance Properties of Dyed Fabrics

- The count, weight and thickness of sericin treated dyed fabric increased.
- Tensile strength increased by 8.5 percent whereas elongation decreased by 8.3 percent.
- Bending length, crease recovery and wettability increased by 3.1, 9.3 and 49.02 percent respectively while air permeability decreased by 10.35 percent.
- Ultraviolet protection factor of sericin treated cotton fabric increased to 22.39 from 14.25 of scoured cotton fabric. *Manjistha* dyed fabric exhibited very good (32.6) UPF whereas sericin treated dyed fabric offered excellent (48.4) ultraviolet protection.

Conclusions:

On the basis of maximum dye absorption and wash fastness, one dye was selected out of four standardized natural dyes. Pure cotton fabric was treated with sericin and the treatment conditions were optimized on the basis of effect of treatment on dyeing properties. These included 0.50% sericin concentration, 50°C treatment temperature, treatment time of 45 minutes, pH 8, pretreatment stage, drying temperature of 70°C, 4 minutes of drying time, 160°C curing temperature and 2 minutes of curing time. After optimizing the treatment conditions, untreated and treated samples were dyed with selected natural dye '*manjistha*'. The effect of sericin treatment on dyeing properties of cotton fabric using natural dye was studied by comparing the CIE LAB values and the colour fastness properties. It was observed that the sericin treated dyed samples had better dye absorption and colour fastness properties than the untreated fabric. Fastness properties increased after sericin treatment. The treated dyed samples were found to be darker than the untreated dyed samples. Not much change was observed in the preliminary properties of the fabrics after sericin treatment. A considerable gain in tensile strength, crease recovery, wetting and ultraviolet protection property was observed after treatment of the fabric with sericin. Sericin; a component of silk, is highly hydrophilic protein and is supposed to increase the dyeability of cotton using acidic natural dyes. Utilization of sericin in textile processing will benefits the farmers who are engaged in silk rearing. The information generated from the study is very useful for the silk cultivators who can utilize the sericin instead of discarding it. The results of the research has important role in environment protection, sericin application, adding value to the textiles and it will have a large spreading foreground of industrialization and can bring high economic and social benefits.

Recommendations:

- It is recommended that 0.50 percent sericin concentration can be taken for maximum percent dye absorption using *manjistha* dye without considerable changes in the physical properties of the cotton fabric.
- Sericin treatment can be given to cotton fabric using natural dyes to improve dye uptake without changing hue of the dyed fabric as in case of mordants.
- Fabrics treated with sericin and dyed with *manjistha* have higher grey scale ratings than fabrics without sericin treatment on tests of colourfastness to washing, light, perspiration and rubbing as compared to untreated dyed fabric.
- Use of sericin treatment is recommended for improving crease resistance, wettability and UV resistance properties.

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ABSTRACT

a)	Title of the thesis	:	**Effect of sericin treatment on dyeability of cotton fabric using natural dye**
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c)	Title of degree	:	**Master of Science in Textile and Apparel Designing**
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f)	Year of award of degree	:	2014
g)	Major subject	:	**Textile and Apparel Designing**
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The present research was planned to study the effect of sericin treatment on dyeability of cotton fabric using a natural dye. To achieve the objectives proposed in the research plan, cotton fabric suitable for apparel use during summer season was selected, desized and scoured. Four easily available dyes *i.e. Kachnar* bark*, Manjistha* root, *Neem* leaves and Safflower flowers were tried for the study. Sericin treated fabrics were dyed with all four dyes using their standardized dyeing procedure which were taken from secondary sources. One natural dye was selected on the basis of effect of sericin treatment on maximum percent dye absorption and wash fastness. Different variables *i.e.* sericin concentration, treatment temperature, treatment time, pH, material to liquor ratio (MLR), treatment stage, combination and concentrations of auxiliaries, drying temperature and time, curing temperature and time were optimized on the basis of maximum percent dye absorption and wash fastness for standardizing sericin application process. Sericin treated and untreated dyed fabrics were tested for colourfastness properties. L, a*, b* and K/S values of dyed fabrics were also compared. Effect of sericin treatment on change in physical properties regarding fabric count, thickness, weight, flexural rigidity, tensile strength, elongation, crease recovery, wettabiliy and air permeability were examined. Effect of sericin treatment and dyeing on ultraviolet protection property of cotton fabric was also investigated.

Out of tried dyes *manjistha* was found to have maximum dye absorption after sericin treatment. Different variables *i.e.* 0.50 % sericin, 4 % crosslinking agent (citric acid), 1% catalyst (sodium hypophosphite), 45 minutes treatment time, 50°C treatment temperature, M:L ratio 1:30, pH 8, drying temperature 70°C, drying time 4 minutes, curing temperature 160°C and curing time 2 minutes were selected for sericin treatment on the basis of maximum percent dye absorption and wash fastness. It was found that the dye absorption of the treated fabric increased from 19.5 to 31.7 percent using optimized variables. Sericin treated dyed fabric also exhibited better colourfastness ratings as compared to untreated dyed fabric. Fabric treated with sericin was redder, yellower and brighter indicated by lower L* value and higher a*, b* and C* values.

Fabric count, weight and thickness did not show any significant change however tensile strength, bending length, crease recovery and wettability increased by 8.5 3.1, 9.3 and 49.02 percent. Moreover, elongation and air permeability decreased by 8.3 and 10.35 percent upon sericin treatment. Ultraviolet protection factor of sericin treated cotton fabric increased to 22.39 from 14.25 of scoured cotton fabric. *Manjistha* dyed fabric exhibited very good UPF (32.6) whereas sericin treated dyed fabric offered excellent ultraviolet protection (48.4). Thus sericin treatment can be given to cotton fabric using natural dyes to improve dye uptake without affecting the hue and physical properties of the dyed fabric.

Major advisor **HOD** **Signature of the student**

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Full paper

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I also undertake that patent, if any, arising out of the research work conducted during the programme shall be filed by me only with due permission of the competent authority of Chaudhary Charan Singh Haryana Agricultural University, Hisar.

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