

**A STUDY ON DYEING PERFORMANCE ON MULBERRY
SILK WASTE/WOOL BLENDED FABRIC**

Thesis

**Submitted to the Punjab Agricultural University
in partial fulfillment of the requirements
for the degree of**

**MASTER OF SCIENCE
in
CLOTHING AND TEXTILES
(Minor Subject: Home Science Extension Education)**

**By
Pooja Bhatt
(L-2009-HSc-256-M)**

**Department of Clothing and Textiles
College of Home Science
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CERTIFICATE I

This is to certify that the thesis entitled, “**A study on dyeing performance on mulberry silk waste/wool blended fabric**” submitted for the degree of **Master of Science** in the subject of **Clothing and Textiles** (Minor subject: Home Science Extension Education) of Punjab Agricultural University, Ludhiana, is a bonafide research work carried out by **Pooja Bhatt (Admn No. L-2009-HSc-256-M)** under my supervision and that no part of this thesis has been submitted any other degree.

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

Major Advisor
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CERTIFICATE II

This is to certify that the thesis entitled, “**A study on dyeing performance on mulberry silk waste/wool blended fabric**” submitted by **Pooja Bhatt (L-2009-HSc-256-M)** to the Punjab Agricultural University, Ludhiana in the partial fulfillment of the requirements for the degree of Master of Science in the subject of Clothing and Textiles (Minor Subject: Home Science Extension Education) has been approved by the Student’s advisory committee along with Head of the Department after an oral examination on the same.

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Abstract

The present study entitled “A study on dyeing performance on mulberry silk waste/wool blended fabric” was carried out to analyse the effect of dyeing on mulberry silk/wool blended fabric. Cold reactive, hot reactive, leveling acid and milling acid dyes were selected for the study. Optimum conditions for dyeing the mulberry silk waste/wool blended fabric with cold reactive dye was, pH 7 for 60 minutes at room temperature using 4 percent dye concentration. Whereas 5 pH for 70 minutes at 90°C with 3 percent dye concentration was considered optimum for dyeing the fabric with hot reactive dye. In case of levelling acid dyeing optimum dyeing conditions were, pH 5 for 80 minutes at 90°C using 4 percent dye concentration. In order to optimize the dyeing condition for milling acid dye best results were obtained at 7 pH with dyeing time 80 minutes, temperature 90°C and 4 percent dye concentration. Physical and mechanical properties like crease recovery angle and tensile strength increased after dyeing the fabric with cold reactive dye, this was followed by hot reactive dyed fabric. Whereas fabric dyed using milling acid dye showed better GSM, thickness, drapability and elongation. However no change was observed on the cover factor after dyeing. Light fastness grade was excellent for the fabric dyed using milling dye and good. Fabric dyed using hot reactive dye showed excellent grade for wash fastness in terms of colour change and no staining on both wool and silk fabrics. The fabric dyed with milling dye exhibited good to excellent grade for dry rubbing in terms of colour change and negligible staining on adjacent fabric, for wet rubbing good grade in terms of colour change and slight staining was observed on adjacent fabric. Hot reactive dyed fabric showed better results for perspiration fastness as compared to the other dyed fabrics. The perspiration grade of hot reactive dyed fabric was excellent for colour change in acidic medium and slight staining on both wool and silk fabrics. When the dyed fabric was kept in the alkaline medium the colour change was ranged between good to excellent, negligible staining was found on wool fabric and slight staining was observed on silk fabric.

Key words: Mulberry silk waste, optimized condition, reactive dye, acid dye, stiffness, flexural rigidity, drape coefficient, crease recovery, cover factor tensile strength, elongation.

Signature of the Major Advisor

Signature of the Student

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“ਮਲਬਰੀ ਸੂਤ ਰਹਿੰਦ-ਖੂੰਹਦ/ਉੱਨ ਬਲੈਂਡਿੰਗ ਧਾਗਾ ਤੇ ਡਾਈਇੰਗ ਕਾਰਗੁਜ਼ਾਰੀ ਦਾ ਅਧਿਐਨ” ਸਿਰਲੇਖ ਵਾਲਾ ਮੌਜੂਦਾ ਅਧਿਐਨ ਮਲਬਰੀ ਸੂਤ ਰਹਿੰਦ-ਖੂੰਹਦ/ਉੱਨ ਬਲੈਂਡਿੰਗ ਧਾਗਾ ਤੇ ਡਾਈਇੰਗ ਦੇ ਅਸਰ ਦਾ ਵਿਸ਼ਲੇਸ਼ਣ ਕਰਨ ਲਈ ਆਯੋਜਤ ਕੀਤਾ ਗਿਆ ਸੀ। ਅਧਿਐਨ ਲਈ ਠੰਢੇ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ, ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ, ਲੈਵਲਿੰਗ ਤੇਜ਼ਾਬੀ ਅਤੇ ਮਿਲਿੰਗ ਡਾਈਆ ਦੀ ਚੋਣ ਕੀਤੀ ਗਈ। ਠੰਢੇ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ ਨਾਲ ਮਲਬਰੀ ਸੂਤ ਰਹਿੰਦ-ਖੂੰਹਦ/ਉੱਨ ਬਲੈਂਡਿੰਗ ਧਾਗਾ ਨੂੰ ਡਾਈ ਕਰਨ ਲਈ ਸਰਵੋਤਮ ਹਾਲਾਤ, 4% ਡਾਈ ਸੰਘਣਤਾ ਵਰਤ ਕੇ ਕਮਰਾ ਤਾਪਮਾਨ ਤੇ 60 ਮਿੰਟਾਂ ਲਈ 7 ਪੀ.ਐਚ. ਸੀ, ਜਦੋਂ ਕਿ ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ ਨਾਲ ਧਾਗਾ ਨੂੰ ਡਾਈ ਕਰਨ ਲਈ ਸਰਵੋਤਮ ਹਾਲਾਤ, 3% ਡਾਈ ਸੰਘਣਤਾ ਵਰਤ ਕੇ 90°C ਤਾਪਮਾਨ ਤੇ 70 ਮਿੰਟਾਂ ਲਈ 5 ਪੀ.ਐਚ. ਸੀ। ਲੈਵਲਿੰਗ ਤੇਜ਼ਾਬੀ ਡਾਈਇੰਗ ਦੇ ਕੇਸ ਵਿਚ, ਸਰਵੋਤਮ ਡਾਈਇੰਗ ਹਾਲਾਤ 4% ਡਾਈ ਸੰਘਣਤਾ ਵਰਤ ਕੇ 90°C ਤਾਪਮਾਨ ਤੇ 80 ਮਿੰਟਾਂ ਲਈ 5 ਪੀ.ਐਚ. ਸੀ। ਤੇਜ਼ਾਬੀ ਡਾਈ ਦੀ ਮਿਲਿੰਗ ਲਈ, ਸਰਵੋਤਮ ਹਾਲਾਤ, 4% ਡਾਈ ਸੰਘਣਤਾ ਵਰਤ ਕੇ 90°C ਤਾਪਮਾਨ ਤੇ 70 ਮਿੰਟਾਂ ਲਈ 7 ਪੀ.ਐਚ. ਸੀ। ਧਾਗਾ ਨੂੰ ਠੰਢੇ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ ਨਾਲ ਡਾਈ ਕਰਨ ਬਾਅਦ ਕਰੀਜ਼ ਰਿਕਵਰੀ ਕੋਣ ਅਤੇ ਤਣਾਓ ਤਾਕਤ ਜਿਹੀਆਂ ਭੌਤਿਕ ਅਤੇ ਮਕੈਨੀਕਲ ਵਿਸ਼ੇਸ਼ਤਾਵਾਂ ਵਿਚ ਵਾਧਾ ਹੋਇਆ, ਅਤੇ ਇਸ ਬਾਅਦ ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ ਹੋਏ ਧਾਗਾ ਦੀ ਵਾਰੀ ਸੀ। ਮਿਲਿੰਗ ਤੇਜ਼ਾਬ ਡਾਈ ਵਰਤ ਕੇ ਡਾਈ ਕੀਤੇ ਧਾਗੇ ਨੇ ਵਧੀਆ ਜੀ ਸੀ ਐਮ, ਮੋਟਾਈ, ਲਪੇਟਣਯੋਗਤਾ ਅਤੇ ਲੰਬਾਈਯੋਗਤਾ ਵਿਖਾਈ। ਪਰ, ਡਾਈ ਕਰਨ ਦੇ ਬਾਅਦ ਕਵਰ ਫੈਕਟਰ ਤੇ ਕੋਈ ਪਰਿਵਰਤਨ ਦਰਜ ਨਾ ਕੀਤਾ ਗਿਆ। ਲੈਵਲਿੰਗ ਅਤੇ ਮਿਲਿੰਗ ਡਾਈਆਂ ਦੀ ਵਰਤੋਂ ਕਰਕੇ ਡਾਈ ਕੀਤੇ ਧਾਗਿਆਂ ਲਈ ਹਲਕੇ ਕਠੋਰਤਾ ਗਰੇਡ ਚੰਗੇ ਤੋਂ ਬਹੁਤ ਚੰਗੇ ਵਿਚਕਾਰ ਸਨ, ਅਤੇ ਠੰਢੇ ਅਤੇ ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈਡ ਧਾਗਿਆਂ ਲਈ ਚੰਗੇ ਹਲਕੇ ਕਠੋਰਤਾ ਗਰੇਡ ਪਾਏ ਗਏ। ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ ਵਰਤ ਕੇ ਡਾਈ ਕੀਤੇ ਧਾਗਿਆਂ ਨੇ ਰੰਗ ਪਰਿਵਰਤਨ ਦੇ ਰੂਪ ਵਿਚ ਧੋ ਕਠੋਰਤਾ ਲਈ ਬਹੁਤ ਵਧੀਆ ਗਰੇਡ ਦਿਖਾਏ ਅਤੇ ਉੱਨ ਅਤੇ ਸੂਤ ਧਾਗੇ ਦੋਹਾਂ ਤੇ ਕੋਈ ਨਿਸ਼ਾਨ ਨਹੀਂ ਪਾਇਆ ਗਿਆ। ਮਿਲਿੰਗ ਡਾਈ ਨਾਲ ਡਾਈ ਕੀਤੇ ਧਾਗੇ ਨੇ ਰੰਗ ਪਰਿਵਰਤਨ ਦੇ ਰੂਪ ਵਿਚ ਖੁਸ਼ਕ ਰੱਬਿੰਗ ਲਈ ਚੰਗੇ ਤੋਂ ਬਹੁਤ ਚੰਗੇ ਗਰੇਡ ਵਿਖਾਏ ਅਤੇ ਨਾਲ ਵਾਲੇ ਧਾਗੇ ਤੇ ਨਾਂਮਾਤਰ ਨਿਸ਼ਾਨ ਪਾਏ ਗਏ, ਅਤੇ ਗਿੱਲੇ ਰੱਬਿੰਗ ਕਠੋਰਤਾ ਲਈ ਨਤੀਜੇ ਰੰਗ ਪਰਿਵਰਤਨ ਦੇ ਲਈ ਚੰਗੇ ਪਾਏ ਗਏ ਅਤੇ ਨਾਲ ਵਾਲੇ ਧਾਗੇ ਤੇ ਨਾਂਮਾਤਰ ਨਿਸ਼ਾਨ ਪਾਏ ਗਏ। ਦੂਜੇ ਡਾਈ ਕੀਤੇ ਧਾਗਿਆਂ ਦੇ ਮੁਕਾਬਲੇ, ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈਡ ਧਾਗੇ ਨੇ ਪਸੀਨਾ ਸਖਤੀ ਲਈ ਵਧੇਰੇ ਚੰਗੇਰੇ ਨਤੀਜੇ ਵਿਖਾਏ। ਤੇਜ਼ਾਬੀ ਮਾਧਿਅਮ ਵਿਚ ਰੰਗ ਪਰਿਵਰਤਨ ਲਈ ਗਰਮ ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈਡ ਧਾਗੇ ਲਈ ਪਸੀਨਾ ਗਰੇਡ ਚੰਗਾ ਸੀ ਅਤੇ ਉੱਨ ਅਤੇ ਸੂਤ ਧਾਗੇ ਦੋਹਾਂ ਵਿਚ ਹਲਕੀ ਜਿਹੇ ਨਿਸ਼ਾਨ ਪਾਏ ਗਏ। ਜਦੋਂ ਡਾਈ ਕੀਤੇ ਧਾਗੇ ਨੂੰ ਖਾਰੇ ਮਾਧਿਅਮ ਵਿਚ ਰੱਖਿਆ ਗਿਆ, ਰੰਗ ਪਰਿਵਰਤਨ ਚੰਗੇ ਤੋਂ ਬਹੁਤ ਚੰਗੇ ਵਿਚਕਾਰ ਸੀ, ਅਤੇ ਉੱਨ ਧਾਗੇ ਤੇ ਨਾਂਮਾਤਰ ਧੱਬੇ/ਨਿਸ਼ਾਨ ਪਾਏ ਗਏ, ਅਤੇ ਸੂਤ ਧਾਗੇ ਤੇ ਹਲਕੇ ਜਿਹੇ ਨਿਸ਼ਾਨ ਪਾਏ ਗਏ।

ਮੁੱਖ ਸ਼ਬਦ: ਮਲਬਰੀ ਸੂਤ ਰਹਿੰਦ-ਖੂੰਹਦ/ਉੱਨ ਬਲੈਂਡਿੰਗ ਧਾਗਾ, ਔਪਟੀਮਾਈਜ਼ਡ ਹਾਲਾਤ, ਪ੍ਰਤੀਕ੍ਰਿਆਸ਼ੀਲ ਡਾਈ, ਤੇਜ਼ਾਬੀ ਡਾਈ, ਸਖਤੀ/ਕਠੋਰਤਾ, ਫਲੈਕਸਰਲ ਕਟੋਰਤਾ, ਲਪੇਟ ਅੰਕਣ, ਕਰੀਜ਼ ਰੀਕਵਰੀ, ਕਵਰ ਫੈਕਟਰ ਤਣਾਓ ਤਾਕਤ, ਲੰਬਾਈ ਯੋਗਤਾ

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CHAPTER I

INTRODUCTION

Colour has always fascinated man and has played a dominant role in adding beauty to the world since the dawn of civilization. It expresses emotions and ideas. Sensitivity to colour has been revealed in paintings, poetry, music and costumes. The importance of colour in textiles has been recognised for thousands of years which is evident in ancient writings and contains frequent reference to it. The art of applying colour through dyeing is usually a process that leads to adding aesthetic appeal to fabric (Paul *et al* 2003).

The most notable contribution to the science of colour and constitution was due to Witt Whoin, who proposed that dyes contain two types of group namely chromophore and auxochrome which are responsible for their colour. Chromophore is a group of atoms principally responsible for the colour of the dye and this is capable of interacting with light and auxochromes are the 'salt forming' groups of atoms whose role is to provide an essential enhancement of the colour.

Dyes are substances that can be used to impart colour to materials such as textiles, food stuffs and paper (Chooiling 2009). A dye that does not fade when the material to which it is applied and exposed to the conditions associated with its intended use refers to fast dye, whereas, a dye that loses its colouring during proper usage is referred to as a fugitive dye. Some of the conditions that could cause such a change in the properties of a dye include exposure to acids, sunlight or excessive heat as well as various washing and cleaning procedures. Certain dyes may be considered both fast and fugitive, depending on the material on which these are used. A dye can generally be described as a coloured substance that has an affinity to the substrate to which it is being applied. It is usually used as an aqueous solution and may require a mordant to improve the fastness of its dye on the fiber.

Dyeing is an ancient art which predates written records. Primitive dyeing techniques included sticking plants to fabric or rubbing crushed pigments into cloth. Today, dyeing is a complex, specialized science. Nearly all the dyestuffs are now produced from synthetic compounds the costs of which have been greatly reduced and certain application and wear characteristics have been greatly enhanced (Bhat 2011).

Textile dyeing is concerned with organic (that is, carbon-based) compounds that can be dissolved in appropriate solvents, usually water. The dyes in solution are absorbed on the surface of the textile fibers and then passed into the interior of the material by a process called diffusion. The process of transferring the dye from solution to the fiber is called exhaustion, with 100 percent exhaustion meaning that there is no dye left in the dye bath solution.

Dyeing is a method for colouring a textile material in which a dye is applied to the substrate in a uniform manner, to obtain an even shade, with the performance and fastness appropriate to its final use. The process of dyeing is carried out in a variety of ways

depending on the specific dye utilized as well as the properties of the substrate. Textile dyeing involves the use of a number of different chemicals and auxiliaries to assist the dyeing process. Some of these are process-specific, while others are also used in other operations. Some auxiliaries (e.g. dispersing agents) are already contained in the dyestuff formulation, but more commonly, auxiliary agents are added at a later stage to the dye liquor. Since auxiliaries in general do not remain on the substrate after dyeing, they are ultimately found in the emissions (Kuehni and Henning 2011).

Effect of dyeing varies according to the type of fiber. Similarly, a dye responds differently on a blended fabric. This is because the affinity of fibers for dyes varies and a different fiber absorbs dyes at different rates (Bae *et al* 2006).

Blending of fibrous materials is a technique to achieve and satisfy the requirements of both, the manufacturers and consumers. It is an intimate mixture of fibers of different composition, length, diameter or colour spun together into one yarn in which the constituent fibers are present in the same yarn, in planned proportion (Kadolph and Langford 1993). Fiber types cannot be separated from the composite and lie next to each other throughout the yarn. Fiber blending is recognized as a highly complex operation with many controllable variables, the nature of the fibers both natural and man-made, the range of fiber dimensions length, thickness and degree of pigmentation are perhaps the most important factors influencing the ultimate appearance of fiber blends.

Modern blending is the combination of different fibers into a mass in which the components are proportionately distributed in pre-determined percentages, in any given cross section of the mass (Sadhna and Bhawani 1998). Thus, in view of their critical influence on the appearance, fault incidence, grade and the sale value of fabric intricacy of mixing as well as blend variations are important parameters to be controlled in blends (Balasubramanian 2003).

Blending of fibrous materials is carried out for achieving a more economical and fuller use of different kinds of raw materials and for obtaining yarn and fabric with predetermined properties. Each kind of raw material used in the blend is called a component. The blends are composed according to formulations. The formulation is a list of components with indication of the percentage content of component in the blend. The components comprising the blend have different properties, and moreover, the fibers which it contains also differ in properties. Blending is aimed at careful mixing of fiber component and their uniform distribution throughout the mass of the mixture.

In a country like India with extremes of temperature and humidity, garments made from natural fibers or their blends are certainly preferred to synthetics or blended synthetics either with natural or manmade for the reason of environment and health.

Natural fibers are self blended in order to improve the uniformity of the fiber. This improves spinning, weaving and finishing efficiency and results in a more uniform final product. Depending upon the type of blend, there is a decrease in cost, added qualities of each fiber, more effective usage of the materials and mixture of textures. The design values of such blended yarn can be explored to give an innovative look to the textile, and more innovative processes can be explored. Generally, natural fibers are blended with synthetic fiber, but now a day's blending of natural fibers is also done to achieve higher quality fabrics. Natural protein fibers, wool and silk, are also blended together in different proportions.

Natural protein fiber is made from animal sources through condensation of alpha amino acids to produce repeating poly amide units with various substituents on the alpha carbon atoms.

The sequence and type of amino acid making up with individual protein chains contribute to the overall properties of the resultant fiber. Two major classes of natural protein fibers are wool and silk (Feughelman 2002). Blending of silk and wool is the way to incorporate better appearance in the woollen fabric and increase the utility of silk fabric. The yarn can be used for the production of warmer varieties of fabrics. The combined effect of warmth and comfort of wool with high strength, luster and good hand properties of silk can be successfully achieved through blending (Papnai and Goel 2005).

Silk is the most cherished of all textile fibers. As the queen of the natural fibers, silk has been used for the production of luxurious textiles of the finest quality. Silk is the most luxurious fabric and has unrivalled qualities of softness, sheerness and lustrous brilliance. It is the most hypoallergenic of all the fibers because of its natural protein structure. It is relatively robust and strong, its smooth surface resists soil. It is wrinkle and tear resistant and dries quickly. It easily competes with steel yarn in tensile strength (Sengupta 2003).

There are four varieties of silk viz. mulberry, muga, eri and tassar and only India grows all the four varieties of silk in the world. Mulberry is the most lustrous, smoother and gives better drape properties than other silks. During the processing of mulberry silk reeling, different qualities of reeling waste are obtained as by products. The technology of blending of waste and left over fibers with other fibers results in improving the constituent fibers and cost effectiveness of the resultant yarn/ fabric has great potential of adoption and development (Papnai and Goel 2005).

Silk has good affinity for dyes. Being proteinous, it absorbs dyes at lower temperature. Silk is dyed with the same dyes as wool, excluding 1:1 metal complex dyes. In addition, direct dyes can be used. The pH of dye bath for dyeing silk is slightly higher as compared to wool. It has the virtue of being very amenable to dyes, soaking and displaying these with unequalled brilliance and lustre.

Due to slightly cationic character of silk with isoelectric point at above pH 5.0, it can be dyed with anionic dye such as acid, metal complexes, reactive and selected direct dyes. But the main objective of coloration of a textile fiber is that the permanency of the colour should not damage the fiber. This implies that it should not destroy its colour during processing and subsequent use (i.e. washing, light, rubbing, perspiration, and saliva). Permanency of dyestuff is most essential while using it for dyeing silk (Uddin and Hossain 2010).

Two properties of the silk that determine its colour behaviour are, firstly, very fine fiber fibrils and secondly high fiber orientation. These fine fibrils produce a large fiber surface. Such a large fiber surface results in low colour yield. Thus, twice the amount of dye is required for achieving a given dark shade. Further, the fastness is poor. A large fiber surface also leads to a high dye-strike rate even at very low temperature resulting in rapid saturation at the fiber surface and unlevel dyeing. The high orientation of the fiber, which is a barrier to diffusion, causes a very slow rate of further dye absorption, after the first rapid saturation. The high pH tends to soften silk and remove some of its lustre. Further, the surface of silk is very easily chafed by abrasion, particularly when the material is in a wet and swollen state.

Wool is a natural, highly crimped, protein hair fiber derived from sheep. Major varieties of wool come from merino, lincoln, sussex and other varieties of sheep. Wool fiber is composed of extremely complex, highly cross linked keratin protein made up of over nineteen different amino acids. It is a tough, durable and resilient animal fiber, known for its dye ability, softness, luxurious look and feel. The utility of the fiber itself is evident in cold-weather and high-performance applications offer superior breathability, temperature regulation and inherent antimicrobial properties.

Wool can be dyed with the acid (metal-free), chrome, reactive, 1:1 and 1:2 metal complex dyes. Absorption of water by wool is less immediate because of hydrophobic surface scales, but in a dye bath the fibers swell with water. Wool fiber has tiny pores, which open up and help in dye absorption, resulting in rich colour. The bond fragility to high temperature (over 90-100°C), alkaline pH, reduction or oxidation, must be taken into account while dyeing (Galafassi *et al* 2009).

Mulberry silk/wool blended fabrics are not new to the world but due to lack of awareness, these have not got commercialized. Therefore, research needs to be developed and intensified for appropriate utilization of silk waste and blending with other protein fibers like wool. Blending of silk and wool is a way to incorporate better appearance and strength in the woollen fabric and increase the utility of the silk fabric (Papnai and Goel 2005).

Natural silk is blended in an equal proportion with wool to make high class apparel goods, contributing lustre and strength; silk yarn is also used in wool piece goods as effect

threads. Mulberry silk/wool blended fabric improves its physical properties from their union with wool becoming smoother, shinier, better drape and easier to wash without fear of felting, while silk acquires resiliency and warmth. The resulting yarn has a firm twist and is easy to weave. Mulberry silk/wool blends are mainly used for apparel in woven or knitted form to give these a luxury character. A large variety of blend ratio can be found ranging from 5 to 50% of silk and wool. The wool gives soothing warmth and breathability and it is also super-absorptive & temperature-regulating. The silk adds even more softness, durability as well as its own temperature regulating properties (Choudhury 2006).

Textile structures are often blends of more than one fiber type. Blends containing similar fibers are easy to dye with dyes of similar structure and application characteristics. Care must be taken in dyeing blends containing fibers of highly different dye affinities, if the dyeing is carried out in the same dye bath this dyeing is referred to as "union dyeing," whereas dyeing fibers in a blend of different colours is referred to as "cross dyeing." In blends, interesting tone-on-tone, tone-on-white, and differential dyeing are possible by selection of appropriate dyes and dyeing conditions (Needles 1986).

Both silk and wool are protein fibers due to the presence of many amines, carboxylic acid, amide, and other polar groups, so these possess similar chemical properties. Silk and wool blends are dyed in piece form or as yarn on packages and hanks. Silk is chemically related to wool because the amino groups are integral components of the both fibers, thus both can be dyed with the same dyes. Silk and wool are hydrophilic in nature, wetted by water and are dyed with either acid or basic dyes through the formation of ionic bonds (salt linkages). These may also be dyed with reactive dyes that form covalent bonds with available amino groups. Dyeing system offers the advantages of a wide range of colours, including brilliant shades, one-bath dyeing method and excellent solid shades (Choudhury 2006).

Silk and wool have a slightly cationic character with an isoelectric point at about pH 5.0. Thus, in an acidic solution, silk fibroin or wool takes on a positive charge through absorption of hydrogen ions. The resulting electric charge can be counter balanced with negatively charged anions of the dye, while dyeing with anionic dyes such as acid, metal complex, direct and reactive dye. The quality and type of silk and wool fibers affects the distribution and shade depth on the fiber (Oswal 2010).

In all protein fiber, the reactive group is the positively charged amino group. These are a part of the peptide bond that strings the amino acids together. Protein fibers can have additional reactive groups that are unique to each fiber, depending on the specific amino acids in the protein. Wool has a lot of the sulphur-containing amino acid, cystine and consequently, forms disulfide bonds. Silk is formed from amino acids that have a few hydroxyl groups that can also bind a dye. These are the same reactive groups as found on cellulosic fiber and so silk will also colour with dyes designed for cellulose (Iqbal 2008).

Acid dyes are water-soluble anionic dyes that are applied to nitrogenous fibers such as wool, silk, nylon and modified acrylic. Attachment of dye to the fiber is attributed at least partly to salt formation between anionic groups in the dyes and cationic groups in the fiber. Acid dyes are not substantive to cellulosic fibers. The acid dyes were probably originally so named because of the presence of one or more sulphonic acid or other acidic groups in their molecules. The term applies to an application class rather than a chemical class since acidic groups are also present in many mordant, direct and reactive dyes but their presence is not a distinguishing feature. Dyes have found wide application in dyeing wool, polyamide fiber and blends of both these fiber but they have to meet very high requirements as regards their application and fastness (Patel *et al* 2010).

Acid dyes are commonly classified according to their dyeing behaviour, especially in relation to the dyeing pH, their migration ability during dyeing and their washing fastness. The molecular weight and the degree of sulphonation of the dye molecules determine these dyeing characteristics. Level dyeing, fast acid dyes, milling acid dyes, super-milling acid dyes are the main types of acid dyes (Uddin and Hossain 2010). Acid dyes are water-soluble anionic dyes that are applied to fibers such as silk, wool, nylon and modified acrylic fibers using neutral to acid dye baths. Attachment to the fiber is attributed, at least partly, to salt formation between anionic groups in the dyes and cationic groups in the fiber (Lokman 2006).

Wool dyed with acid dyes has low wash fastness because of the weak, secondary forces between the fiber and the dye molecule. The amount of covalently fixed dye increases when after treating the dyed wool samples with salts (Trezl *et al* 1998).

Silk has an affinity for acid dyes, but the colour tends to be less fast than on wool. Silk will exert its affinity for acid dyes at lower temperature than in the case of wool, and dyeing is usually commenced at 40°C and the temperature is not allowed to rise above 80°C (Tortman 1994).

Reactive dyes first appeared commercially in 1956 and were used to dye cellulosic fibers. These are so called because their molecules react chemically with the fiber polymer to form covalent bond, thus dyestuff becomes the part of fiber and much less likely to be removed by the washing, this is the reason that these dyes possess good to excellent fastness to light and washing cellulosic fibers (Uddin and Hossain 2010). The dyes contain a reactive group that, when applied to a fiber in a weekly alkaline dye bath, forms a chemical bond with the fiber (Lokman 2006). Reactive dyes can also be used to dye man-made and natural protein fibers. Cold reactive dyes and hot reactive dyes are two type of reactive dye (Lewis 1996). Cold reactive dyes are very easy to use because the dye can be applied at room temperature.

The reactive dyes give colours on protein fibers and require temperature between 70°C and 90°C applied at neutral pH (Tortman 1994).

Researches on dyeing of silk (Weibin *et al* 2007) and wool (Ookson *et al* 1995) are available but dyeing of mulberry silk waste/wool blend is a new concept. The mulberry silk waste/wool blend unites the best qualities of this natural fiber, creating a truly unique textile and further dyeing provides value addition to the blended fabric. Therefore, the present study was designed with the following objectives:

1. To optimize the dyeing conditions for dyeing mulberry waste silk waste/wool blended fabric with acid and chrome dyes.
2. To compare the selected physical properties of the undyed and dyed mulberry silk waste/wool blended fabric.
3. To compare the dye absorption by the mulberry silk waste/wool blended fabric with selected dyes.
4. To evaluate the colour fastness properties of dyed mulberry silk waste/wool blended fabric.

The study has following limitations

- The study was limited to the use of mulberry silk waste and wool fiber.
- The study was limited to the use of plain weave.
- The study was limited to the use of one acid and one reactive dyes only as chrome dyes are now banned.
- The study was limited to the only two shades of each dye.

CHAPTER II

REVIEW OF LITERATURE

The review of literature related to this study has been collected from libraries of Punjab Agricultural University Ludhiana, Gobind Ballabh Pant University of Agriculture and Technology Pantnagar and the internet.

Review of literature was divided into following categories

- 2.1 Dyeing of silk
- 2.2 Dyeing of wool
- 2.3 Dyeing of blends
- 2.4 Dyeing with reactive dyes
- 2.5 Dyeing with acid dyes
- 2.6 Effect of dyeing on physical and mechanical properties of the fabric

2.1 Dyeing of Silk

Ashkeanazi (1994) carried out a research on pre-treatment of silk with formic acid. Results showed that amount of dye absorption and the dyeing rate achieved during the dyeing of silk can be increased significantly by pre-treatment with 10% of formic acid without affecting the service.

Investigation was done by Freddi *et al* (1994) on the dyeability of silk fabrics with dibasic acid anhydrides. Silk fabrics were chemically modified with different kinds of dibasic acid anhydrides. Samples with different acyl contents, ranging from 24 to 107 mol/10⁵g, were obtained by reaction with succinic (S), glutanic (G), phthalic (PA), and o-sulfobenzoic (OSBA) anhydrides. The absorption of basic dye was significantly enhanced for all samples. The maximum dye uptake was attained by OSBA-treated silk fabrics, while samples treated with S, G and PA did not differ significantly.

The behaviour of heterobifunctional reactive dyes, containing a monochlorotriazine and a vinyl sulphone group, on silk was studied by Agarwal *et al* (1996). The results indicated that maximum exhaustion and fixation were obtained in a neutral medium at 90°C. The addition of sodium sulphate was found to promote the exhaustion of reactive dyes at pH values above the isoelectric point of silk. A reduction in the solubility of dyed silk indicated that cross links were formed with bifunctional reactive dyes.

The effect of bleaching on dyeing of silk fabric with acid green and basic red dyes using hydrogen peroxide was studied by Singh *et al* (2000). The silk fabric was kept in bleaching solution at 40°C and 8.0-8.4 pH. Results showed that bleaching made significant changes on the percent dye absorption values of acid and basic dyes. It not only improved the shade of the dye but also significantly improved the colour fastness to washing, light, rubbing and perspiration.

Gahlot and Singh (2002) optimized the dyeing parameters for batik dyeing on silk. Dyeing was performed at different temperatures- 30°C, 40°C and 50°C for different times i.e. 10, 60 and 80 minutes. Results for colourfastness to light, washing, rubbing and perspiration were in accordance with the results of percent dye absorption at 40°C for 80 minutes dyeing time.

According to Mousa and Youssef (2003) while dyeing nylon 6 and silk fabrics with a model disulphide bis(ethylsulphone) reactive disperse dye, optimum dye exhaustion and fixation were achieved at pH 8 and 130°C. The results of dyeing on both substrates indicated that the model disulphide bis(ethylsulphone) reactive disperse dye had a higher degree of exhaustion and fixation on silk than on nylon 6. The fastness and levelling properties on both fabrics were good.

Sen and Babu (2004) studied the effect of silk structure on dyeing behaviour of Indian silk. Noticeable differences in the dye uptake were observed among the different varieties of silk. Mulberry varieties showed higher dye uptake as compared to that of all the three non mulberry varieties. Among the non mulberry varieties, tasar showed higher dye uptake followed by eri and muga. Interestingly, dye uptake reduced significantly within a variety from the outer to the inner layers. The reduction within a variety was found to correlate well with the morphological parameters.

Shukla *et al* (2004) worked on low-temperature ultrasonic dyeing of silk. Silk was dyed with cationic acid and metal complex dyes at low temperature in the presence of ultrasonic energy for relative short duration. It was seen that the dyeing in the presence of ultrasound for only 15 minutes at a temperature as low as 45°C, showed increased dye uptake by 39 percent. Dyeing of silk for 15 minutes, at 50°C increased the dye uptake of about 71 percent over conventional dyeing. Further they reported an increase of 38 percent and 76 percent respectively in dye uptake over that conventional dyeing when dyeing of silk with metal complex dyes for 15 min at 45°C and 50°C in the ultrasonic bath.

In a study of water soluble cationic sulphur dye for the dyeing of silk, the solubilised dye gave excellent fixation on silk (Wang and Yang 2005). It was as high as 98.5 percent, representing a dramatic improvement on the parent sulphur dye when applied after reduction with sodium sulphide. The solubilisation of sulphur dyes using this technique was therefore promising for the dyeing of silk, without adverse effect on the substrate.

Prachayawarakorn and Kryratsamee (2006) explored the dyeing properties of *Bombyx mori* silk grafted with methyl methacrylate and methacrylamide. To improve its dyeing and colourfastness properties, degummed *Bombyx mori* silk was chemically modified by a grafting technique with either methyl methacrylate (MMA) monomer or methacrylamide (MAA) monomer. Acid and basic dyes were used. The colourfastness to washing of the MMA-grafted and MAA-grafted silks dyed with acid and basic were greatly improved.

Colourfastness to both acid and basic perspirations with acid and basic dyestuffs also improved slightly.

Wang and Ji (2006) worked on superfine pigment dyeing of silk fabric by exhaust process. The results showed that the conditions suitable for modification treatment are 10 g/l concentration of cationic reagent, pH 8 and liquor to goods ratio of 100:1 at 60°C for 30 minutes. The whiteness index decreased with the increase of alkali of cationic treatment. The crock fastness and wash fastness of silk dyed by pigment exhaust dyeing achieved 3-4 and 4 scales respectively. The treated silk fabrics retained a soft handle.

Silk was modified with aminated polyepichlorohydrin to improve dye ability with reactive dyes (Weibin *et al* 2007). Reactive dyes have almost complete exhaustion and ideal fixation on aminated polyepichlorohydrin pretreated silk without addition of salt or alkali. The effects of varying pre-treatments and dyeing conditions were studied. Dyeing of modified silk showed good wash fastness, dry and wet rub fastnesses as well as light fastness.

Feiz and Salimpour (2008) researched on dyeing of pile silk yarn with reactive and basic dyes along with the effect of after treatment on fastness properties. Effectiveness of after treatment which improved the fastness of reactive and basic dyes to repeated washing at 50°C was enhanced by the application of metal salts ($MnSO_4$, $CuSO_4$ and $K_2Cr_2O_7$) and enzymes (protease and savinase). This was due to formation of large molecular size, low water solubility complex, situated at the surface of dyed substrate which physically resist the diffusion of dye from the dyed substrate during washing.

El-Zawahry *et al* (2009) prepared the modified phospholipids from the commercial soyabean lecithin via acetylation of the acetone insoluble fraction phosphatidylethanolamine. *N*-Acetyl-phosphatidylethanolamine was used to prepare liposomes for encapsulating anionic dyes (acid and reactive dyes) for dyeing silk fabric. A comparison of efficiency of the micro-encapsulated dyes to dye silk fabric with the conventional dyeing process revealed that the acetylated acetone insoluble fraction liposome showed better encapsulation of the reactive dyes and achieved more dye uptake than the acid dyes. It was also found that fastness properties of dyed silk with micro-encapsulated anionic dyes did not change significantly than the conventional dyeing method. Reuse of the micro-encapsulated dye bath produced low water pollution as the effluent was virtually colourless. The process was also economic and eco-friendly.

2.2 Dyeing of Wool

According to Galek (1980) mohair could be easily dyed like wool, but rabbit hair was hard to wet and calls for levelling acid dyes. Channeling in mohair fiber leads to unlevelness and chrome dyes were avoided since these caused a harsh handle. It was simple and easy to dye mohair-nylon blends but the colours were in pale shades in hand knitting yarn.

In a study of on speciality hair blended products Sugumar (1988) reported that dyeing of rabbit hair and wool mixture with acid and chrome dyes produced a two tone effect. The rabbit hair was lighter in shade because of the medulla. The rate of dyeing and exhaustion in mohair was found to be greater than that of wool. In another study on dyeing mohair containing kemp, Hunter *et al* (1990) described that the dye uptake varied with class of dye. Kemp fibres could be disguised in fabrics dyed in yellow, but it was conspicuous when dyed in black, red and green shades.

Page and Dodds (1990), in their study on fastness to sunlight exposure of dyed wool yarns treated with a cationic after treatment, revealed that dyed wool yarn in pale shades can be produced with 1:2 metal complex dyes with or without cationic after treatment. The greatest changes were observed for range of shades, particularly with reactive dyes. Cationic after treatment showed little effect on colour change on exposure to sunlight. It was recommended that dyes should be selected carefully for pale shades, if satisfactory resistance to sunlight is to be achieved.

Patel *et al* (1991) worked on free radical grafting of basic and acid dyes on wool fibers. It was observed that the fixation of both acid and basic dyes on wool fiber was possible only in acidic medium. The amount of dye fixed on wool fiber was maximum at pH 5.5 in basic dye and pH 4.5 in case of acid dye. The free radical grafting of dyes on wool fiber was dependent on the presence of the free radical in the system that is why fixation strongly depends on the pH of dye liquor.

Exploring the importance of dye hydrophobicity in acid dyeing of wool and nylon (Needles and Weatherall 1992) concluded that the acid dye uptake on wool and nylon was highly dependent on the dye structure. It showed that dye absorption was dependent on the hydrophobicity of the dye on its polar or ionic character particularly at higher pH.

Gomes *et al* (1996) studied the hydrolysis of reactive dye for application on wool fibers. The synthesis of a reactive dye in the anhydride form was described and its kinetics of hydrolysis was investigated at various pH values. It was found that the experimental rate constant depended on pH, with the maximum between pH 5 and 6. The best wool dyeing conditions were obtained by dyeing for 60 min at 100°C and pH 6.

Patel *et al* (2002) researched on the influence of benzyl alcohol pre-treatment on physio-mechanical characteristic and dyeing behaviour of acid dyes on wool. Treatment of wool with aqueous benzyl alcohol modified its morphological structure and permanently removed the cuticle resulting in loss of tensile strength and also caused shrinkage and weight loss in wool fiber. Normally wool was dyed at boil for 90 minutes but on treatment with benzyl alcohol, the duration of dyeing was reduced to 45 minutes and dyeing may be performed at 70°C.

A study of the effect of dye bath acidity on dye absorption and mechanical properties of wool fibers revealed that the higher the strength of acid used as dye assistance, the greater the percent of dye absorption over progressive increase in temperature (Bello and Abdul 2008). The presence of the sulphate ions in the dye bath greatly promoted the levelness of the dyeing. Due to high acidity, hydrolysis of wool lead to increased dye absorption with corresponding decreased tensile strength of the wool fiber.

Jocic *et al* (2005) while researching on the effect of low-temperature plasma and chitosan treatment on wool dyeing with Acid Red 27 found that the dyeing kinetics was measured at two different pH (4.2 and 6.5) and three different temperatures (60, 85, and 100°C) to gain information about the contribution of the surface modification treatment to the dyeing mechanism. When treated with chitosan, the polymer sheath spread on the surface of the fibers and acted as a predominant dyeing site in very short dyeing times.

According to Juozas *et al* (2005) the use of a non-ionic auxiliary based alkylphenoethoxylate enhanced both the dyeing rate and the colour yield of acid dye on wool. Increased dye substantivity, dyeing rate and colour yield for alkylaminoethoxylate was most important at higher temperatures. Increasing dyeing temperature from 60°C to 85°C, or using auxiliaries, lead to a change in the colour yield of the wool fiber.

Suwanruji and Freeman (2006) reported that sumifix supra dye can be applied to wool with high degree of fixation and high fastness. The highest fixation on wool can be obtained between pH 4-5. Dyeing at 95-100°C for 50-60 minutes produced the highest wash fastness. A levelling agent was also used to prevent skitteriness on wool.

Kan and Yuen (2006) treated the wool fiber with low temperature oxygen plasma and dyed with three types of dyes i.e. acid dye, chrome dye and reactive dye. For acid dyeing, the dyeing rate of the LTP-treated wool fiber was greatly increased but the final dyeing exhaustion equilibrium did not show any significant change. For the chrome dyeing, the dyeing rate of the LTP-treated wool fiber also increased, but the final dyeing exhaustion equilibrium increased to a small extent. In addition, the rate of after chroming process was similar to the chrome dyeing process. For the reactive dyeing, the dyeing rate of the LTP-treated wool fiber increased greatly and the final dyeing exhaustion equilibrium also increased significantly. Thus, LTP treatments improved the dyeing behaviour of wool fibers in different dyeing systems.

Cai and Qiu (2008) examined the dyeing properties of wool fabrics treated with atmospheric pressure plasmas. The scanning electron microscopy analysis showed etching and crack marks on treated fiber surfaces. The plasma treatments greatly increased initial dyeing rate, shortened half-dyeing time and the time to reach dyeing equilibrium, although the final exhaustion did not change.

Results of dyeing of wool with an oil-in-water micro emulsion system showed that the dye exhaustion on the fabric took place mainly when the temperature of the dye bath promoted a change in the molecular organization of the micro emulsions with the liberation of the dye solubilized in the oil droplets of the micro emulsions. Although uniformly and evenly dyed fabrics were obtained, these showed very low wash fastness. Both the dye exhaustion and wash fastness improved considerably for the fabrics dyed at a high temperature (Paul *et al* 2008).

Naebe *et al* (2010) reviewed about the effects of levelling agent on the uptake of reactive dyes by untreated and plasma treated wool. Results showed that in the presence of levelling agent the dyeing temperature increased to the 90°C which resulted in penetration of dye to the fiber. It also promoted uniformity and levelling of the dyed fabric.

Rombaldoni *et al* (2010) in his study mentioned that the oxygen plasma treatment to reduce the dyeing temperature of wool fabrics. Fabrics were dyed at 80°C, 85°C, and 90°C. The plasma treatment modified the surface morphology greatly, increased the wettability, initial dyeing rate, shortened the half-dyeing time, improving the diffusion of the dyes into the fibers at lower dyeing temperatures. The pre-treatment allowed dyeing the wool fabrics at 85°C without affecting their dyeing performances in terms of final bath exhaustion and colour fastnesses.

Yuan *et al* (2010) investigated the dyeing behaviours of ionic liquid treated wool. A new class of “green” solvent- ionic liquid (IL) was employed to improve the dye-ability of wool. The water contact angle of the fabric treated with IL at 100°C decreased from 118.6°C to 106.4°C. The tensile strength of IL-treated wool fibers decreased slightly when the treating temperature was less than 100°C. Dyeing kinetics experiments revealed that the IL treatments greatly increased initial dyeing rate, shortened half-dyeing time, and time to reach dyeing equilibrium. The final exhaustion and colour depth of IL-treated wool were also increased accompanying with slightly decreased colour fastness.

2.3 Dyeing of Blends

Maged (1998) studied the effect of sandospace R on the dyeability of gamma-irradiated wool, wool/polyester and polyester fabrics with some disperse dyes containing amino groups. It was found that the pH of the dyeing bath at which the highest colour strength obtained was 3. Increasing the dye concentration up to 4% based on fabric weight, caused a significant enhancement in the colour strength, and whilst raising/the dyeing bath temperature from 60°C to 100°C appreciably accelerated the rate of dye uptake. Complete exhaustion absorption of the disperse dye occurred over a period of 2 hours.

Doran (1999) highlighted some problems associated with dyeing wool-rich blends of polyester. It had been found that whilst dyestuff selection is generally unchanged, the dyeing method must be changed completely in order to achieve good results. Blends containing 50

percent or less of wool may be dyed successfully by a one-bath method, but wool-rich blends need to be dyed by a novel two-bath method.

Salam *et al* (2001) in the paper on dyeing of bleached sulphonated jute/cotton fabrics highlighted that the dye absorption decreased with increase of dye concentration. Even and bright shades were obtained when fabric was dyed with 1.5-2.0 wt% mordant dyes. Dye absorption increased with dyeing time and reached to saturation within 50-60 minutes. Electrolyte concentration also increased the dye absorption which was maximum at 90°C-100°C.

Ghosh *et al* (2003) performed a study on the effect of dyes and finishes on UV protection of jute/cotton fabrics. It is observed from spectral analysis that the monochlorotriazinyl reactive dye with cyanuric chloride nucleus such as Cibacron Red FAL, was quite effective in UV protection. Simultaneous dyeing and finishing with Cibacron Red FAL and Cibatex UPF provides higher UV protection to the jute/cotton blend.

Kim *et al* (2006) researched on dyeing of cotton and polyester/cotton blend with disperse dyes using sodium 2-(2, 3-dibromopropionylamino)-5-(4, 6-dichloro-1,3,5-triazinylamino)-benzenesulfonate. The results indicated that it was possible to dye polyester/cotton blend at one-bath dyeing using one kind of disperse dye containing amino groups. Two kinds of dyeing methods such as two-bath process and one-bath process one-bath dyeing were investigated. The differences between these were negligible.

Kumar *et al* (2006) compared the single and two bath methods of dyeing of polyester and viscose blends and concluded that, when the polyester-viscose blend ratio of 60:40 was dyed with two bath technique, the water consumption was two times more as compared to single bath dyeing. In single bath technique two separate bath were used, where as in double bath technique four separate bath were used with M: L 1:20. Single bath technique was more eco friendly as the presence of total effluent in single bath was less than two bath technique.

Broadbent *et al* (1996) used water-soluble arylating agent to dye wool and wool blends with disperse dyes. The compound sodio-2, 4-dianilino-6-[4'- β -sulphatoethylsulphonylanilino]-s-triazine (FAA 200) was found to be promising. This compound could be applied in the same bath as a disperse dye at pH 5-6. Application with commercially available disperse dyes gave bright, level dyeing that exhibited promising wet fastness. FAA 200 was used as an auxiliary to dye disperse dyes on both fiber components of a wool-polyester blend fabric. It was found that addition of hydrogen peroxide or sodium thiocyanate was necessary to obviate reduction of certain disperse dyes when dyeing at 120°C.

Menezes (2008) reported that dyeing of wool acrylic blend wool acrylic blend can be dyed with chrome, reactive, acid and 1:1 metal complex dyes. Chrome and reactive dyes gave the best results and these can be used for wool in the isoelectric region at pH 4-5. Reactive dyes on wool/acrylic blend gave good fastness to perspiration.

In a study on one-bath union dyeing of wool/polytrimethylene terephthalate blends (Zheng *et al* 2008). A technique was developed which minimised the staining of disperse dyes on wool and produced dyed goods with high levels of wet colour fastness. Carriers were not required to enhance the dyeability of PTT at low temperatures. The wool component appeared to be protected against damage at 110°C by the reactive dyes. The results indicated the potential for blending PTT fiber and wool to produce fabrics that were easier to dye at lower temperatures than conventional wool/polyester blends.

Chollakup *et al* (2010) compared the physical characteristics and dyeing properties of eri silk fiber and eri silk/cotton blended yarn and found that the percentage of crystallinity of the cotton yarn tend to increase after bleaching, whereas the percentage of crystallinity of the eri yarn decreased marginally. Percentage exhaustion and the colour yield of the blends tend to decrease with the increasing silk content. Shade variation was observed on the yarns at different blend ratios. This was expected to be caused by the different physical nature of eri silk and cotton fibers. The dye uptake and visual shade of each dye on the two fibers were different.

El-Shishtawy *et al* (2011) performed the union dyeing of wool/acrylic fabrics with acid and reactive dyes, namely CI Acid Red 40, CI Acid Blue 25, CI Reactive Red 194 and CI Reactive Blue 25 using a one-bath dyeing process. Factors affected the dyeability of the blend fibre, such as dye bath pH, liquor ratio, temperature, time and dye concentration, were evaluated with respect to the dye exhaustion, fixation, colour strength, levelling and fastness properties. Excellent to good fastness was obtained for all samples, irrespective of the dye used. The result of the investigation offers a new viable method for union dyeing of wool/acrylic fibres in a one-dye bath process.

2.4 Dyeing with reactive dyes

Ball *et al* (1986) tested the cross linking effects in reactive dyeing of protein fibers. Dyes featuring two reactive groups or one bifunctional group were applied to wool and silk which reduced the solubilities of these fibers in appropriate solvent systems. This was consistent with the formation of crosslinks during dyeing. On wool, dyes with more closely located reactive sites were comparable in effect to formaldehyde. The reduced solubility of wool dyed with dyes containing one α -bromoacrylamide group indicated bifunctional reaction of this group. On silk, α -bromoacrylamide dyes underwent bifunctional reactions to a limited extent only. The inhibition of dye penetration after crosslink formation on silk in dyeing with a Difluorochlo Ropyrimidine dye at 40°C can be greatly alleviated by dyeing at 90°C and thereby distributing the cross links more evenly throughout the fiber. The effectiveness of the highly reactive dichlorotriazine system in cross linking depends on the method of dyeing used on wool and silk.

A study on loss of light fastness properties of wet textiles by Anonymous (1994) revealed that the most colours affected by exposure to light in a wet state included blue shades, specially certain reactive dyes. He further reported that especially blue components containing green and turquoise shades, as well as bluish red shades were more fugitive.

Imada (1993) conducted a study on fading of azo reactive dyes by perspiration and day light. The combined effect of perspiration and light fastness of reactive dyes was investigated and compared with results obtained in conventional light fastness test, for five different blue and black commercial reactive dyes. Reactive dyeing containing H or K azo chromospheres (hydrogen and potassium) performed better in the conventional light fastness test.

Jocic (1997) applied chitosan/nonionic surfactant mixture to wool and assessed its dyeing using reactive dye. A weak time interaction was observed between chitosan and the surfactant over time, resulting in changes in the conformation of chitosan in solution, which was confirmed by viscosity measurements. The presence of chitosan/surfactant association improved the application to wool, which was assessed by dyeing the treated wool with a reactive dye at 60°C. Since this technique offered the possibility of producing deeper and more vivid colours without increasing the concentration of dye used.

Ahmad *et al* (2001) compared the wash fastness of reactive and vat dyed samples. The findings of this study showed that reactive dyes might be better substitute for the vat dyes. The grey scale rating for both the reactive and vat dyes was 5 for colour change, where as vat dyes showed 5 for staining on wool as well as on silk and grey scale rating for reactive dyes was 5 for silk and 4-5 for wool.

The influence of salt, alkaline and dye was studied by Iftikhar *et al* (2001). The study revealed that the varying concentrations of alkali, dye and salt influenced the dyeing of cotton fabrics with reactive dyes. Excellent results for rubbing, ironing and dry cleaning fastness were recorded at 30 percent salt concentration, 10 percent alkali and 1 percent dye concentration. The gray scale rating for sample was 4 for dry rubbing and 3-4 for wet rubbing, the gray scale rating was 4-5 for dry and 4 for wet ironing. Overall gray scale rating for both change of shade and staining ranged from 4 to 4-5.

Richard and Stephen (2002) investigated a greener approach to cotton dyeing with excellent wash fastness. Attempts were made to find a more environmentally friendly method of dyeing cotton. It was intended that the new method would not compromise the excellent wash fastness levels typical of reactive dyed cotton. By employing a pre-treatment method, salt and alkali could be completely eliminated from the dyeing process and in comparison with standard reactive dyeing processes, the time taken for the dyeing process to be completed could be significantly reduced and the volume of water required could be halved. The dyeing secured using the pre-treatment method had wash fastness values equal to those observed for the standard reactive dyeing.

In another study on Matyjas and Rybicki (2003) concluded that reactive red showed dyes high water solubility due to the presence of many sulphonic groups and their symmetric arrangement in the dye molecule. Their excellent solubility in water made it possible to carry out the application process with the low liquor ratio of 1:10. These dyes showed substantivity to cellulose fibers due to an increased molecule size of the dyes. The kind of active compound used in chromophoric grouping had a great influence on exhaustion and fixation values. The bi-functional reactive dyes (the derivatives of anthranilic acid (R-6 - R-11) showed highest secondary exhaustion degree from the dye bath. The derivatives of 4-(β -ethylsulphate) sulphonylaniline dyes showed the lowest secondary exhaustion degree values due to the presence of vinylsulphone reactive groups in the dye molecule.

Kasahara *et al* (2004) analysed the effect of processing and reactive dyeing on the swelling and pore structure of lyocell fibers. Lyocell yarns were treated with NaOH, liquid ammonia, high pressure steam and polycarboxylic acids, and then dyed with five reactive dyes. The water content of the samples was also measured by centrifugal and chromatographic techniques. A good correlation was found between the values from the different techniques for all samples, except for the samples dyed with bi-functional reactive dyes. The total pore volume substantially increases with NaOH treatment and decreases with high pressure steaming. The pore size distributions for the dyed samples affected by the dyes used.

Chattopadhyay *et al* (2005) observed the reuse of reactive dyes for dyeing of jute fabric. It was found that the two-step two-bath method of reactive dyeing, where exhaustion and fixation step was separated, was most ideal for reuse of dye bath. Separate original samples produced K/S value same as that of original sample and the K/S value of separate reuse sample varied from 50 percent to 80 percent of the original sample depending on the class of dye. In case of same bath method, colour yield of original reuse samples varied from only 10 percent to maximum 30 percent of the original samples depending on the class of dyes. Reuse of reactive dyes following separate bath method was particularly suitable for higher depth of shade (4 percent and above). This process not only utilised costly reactive dyes to the maximum extent but it also produced low water pollution as the effluent contain minimum amount of dye.

The influence of cationization on cotton reactive dyeing was studied by Subramanain *et al* (2006). The results concluded that 20 percent concentration of cation agent at 70°C with 10g/l soda ash gave better colour strength and maximum dye utilization. Influence of temperature and concentration on K/S value was predominant than the pH of dye bath. Washing and rubbing fastness grade of cationized cotton are similar to normal dyeing.

Findings of the research entitled, the influence of pH on reactive dyes showed that cold brand dyes are highly reactive requiring comparatively milder conditions in dye fixation.

These were primarily dyed at normal room temperature (about 25°C-30°C) using soda ash or Sodium Bicarbonate. Due to the very high reactivity of this class of dyestuff, they require lesser amount of alkali for fixation. A pH of 9.3 to 9.8 was sufficient for proper fixation of the dye to cellulose (Anonymous 2007).

Klemola *et al* (2007) evaluated the toxicity of reactive dyes and dyed fabrics with the HaCaT cytotoxicity test. The results showed that reactive dyes react with cellulose under alkaline conditions but the reactive dyes themselves have pH values between 4.5-6.5. Results also revealed that the dyes were toxic, but the dyed fabrics were not. This was explained by the fact that the pure reactive dye in powder form was very active, but after the dyeing processes many of the reactive sites on the dye molecules take part in the formation of covalent bonds with fiber molecules. These bonds were very stable and thus the dyed fabric material is not toxic.

Hussain and Ali (2009) compared the properties of cotton fabric dyed with pigment and reactive dye. Study concluded that in terms of fabric stiffness, tear strength and rubbing fastness, pigment dyeing was inferior to reactive dyeing. In terms of light fastness and fabric pilling, pigment dyeing was superior to reactive dyeing, while in terms of tensile strength and washing fastness, there was no significant difference between the two types of dyeing. While pigment dyeing could be a good alternative to reactive dyeing for light shades.

Salt-free continuous pad-steam dyeing process with reactive dyes was conducted by Yong *et al* (2009) and revealed that the dyed fabrics exhibited good levelling property, dyeing penetration and higher K/S value. Compared with the conventional reactive dyeing process, the dyed fabric using the salt-free dyeing process can obtained the similar colour fastness and higher K/S value, thus solving the pollution problem.

In another study on cold pad-batch dyeing method for cotton fabric and dyeing with reactive dyes using ultrasonic energy by Khatri *et al* (2011) concluded that the reactive dyes were vastly used in dyeing and printing of cotton fiber. These dyes had a distinctive reactive nature due to active groups which form covalent bonds with –OH groups of cotton through substitution and/or addition mechanism. Among many methods used for dyeing cotton with reactive dyes, the cold pad batch (CPB) method was relatively more environment friendly due to high dye fixation and non requirement of thermal energy. The dyed fabric production rate was low due to requirement of at least twelve hours batching time for dye fixation. The proposed CPB method for dyeing cotton involves ultrasonic energy resulted into a one third decrease in batching time. The study showed that the use of ultrasonic energy not only shortened the batching time but the alkalis concentrations can be reduced considerably. In this case, the colour strength (K/S) and dye fixation (% F) also enhanced without any adverse effect on colour fastness of the dyed fabric. The appearance of dyed fiber surface using

scanning electron microscope (SEM) showed relative straightening of fiber convolutions and significant swelling of the fiber upon ultrasonic application.

2.5 Dyeing with acid dyes

The influence of arylamide groups on the properties of acid dyes was examined by (Blus and Kraska 1993) results showed that the dyes, which had a very high degree of exhaustion from a dye bath at pH 5.0-6.0, can be used for dyeing polyamide, wool and natural silk fibers. They confirmed that introducing the amide groups (carbamide and sulphonamide) into the dye molecule significantly improved the fastness of dyeing to wet agencies. It also slightly improved the light fastness, compared to the dyes lacking the amide groups.

Burkinshaw and Maseka (1996) improved the wash fastness of non-metallised acid dyes on conventional and microfiber nylon 6,6 using syntan (cation agent). The uptake of the on both types of fiber increased with decreasing liquor ratio, possibly as a result of syntan aggregation and secondly, it also increased with increasing application temperature, this being attributable to the higher kinetic energy of the syntan molecules and the greater extent of fiber swelling operative at the higher temperatures. The presence of 1 percent (owf) dye on the two types of fiber reduced the extent of syntan uptake, the mechanism of syntan adsorption on to both substrates was unaffected. The finding that syntan uptake was greater on microfibers than conventional fibers was attributed to the greater surface area of microfibers. Despite the greater uptake of the syntan on to dyed microfiber, the wash fastness of syntan dyed microfiber was lower than that of its syntan dyed conventional decitex counterpart. The effectiveness of the syntan in improving the wash fastness of several non-metallised acid dyes on microfiber was enhanced by the subsequent application, to the syntan, dyed substrate, of certain cationic agents. The level of wash fastness achieved using this syntan/cationic agent system was considerably higher than that obtained using an after treatment with either 4 percent syntan (owf).

Zhang *et al* (1997) prepared six violet acid dyes from bromaminic acid and their structures were determined. The colour, fastness and dyeing rate of these dyes were also measured and compared. It was found that the dyes, with one exception, could be used to dye wool, silk and polyamide fibers with excellent fastness to light, wet treatment, rubbing and high dye uptake, with intense brilliant bluish violet hues.

Yang (1998) examined the effect of salts on physical interactions in wool dyeing with acid dyes. The modified donnan model, which considers the dye distribution coefficient as a function of salt concentration and property in the dye bath, was used to describe the dyeing behaviour of wool with acid dyes and different kinds of salts. They affected the free energy of the dye bath through their influence on water structures. Adding salt to the dye bath decreased the dye sorption due to its reduction of ionic attraction between negatively charged acid dyes

and positively charged wool fibers. However, adding some salts such as sulphate and dihydrogen phosphate could increase the dye absorption if the salt concentration was high.

Blackburn and Burkinshaw (2000) observed the effect of after treatment of acid dyes on conventional nylon 6.6 with a commercial syntan/cation system. Conventional nylon 6.6 fabric was dyed using two commercial non metallised acid dyes. After treatment using a commercial syntan alone improved the wash fastness after five washes, as compared to the non-after treated sample. The sequential application of a cationic compound to the syntan dyeing imparted a further improvement in wash fastness. Further improvements to the system could be achieved by varying the cationic agent, the amount of cationic agent applied and the pH of the application system.

Neelima and Mahale (2000) in their study on colourfastness of acid dye UAS sheep breed wool to crocking revealed that acid dyes exhibited good fastness to dry crocking at fiber stage and excellent fastness at yarn stage. The colour staining on undyed cotton samples showed slight change at fiber stage and noticeable change at yarn stage. Fastness to wet crocking on acid dyes was ranged between poor to fair at fiber stage and fair at yarn stage. The colour staining ranged from much change to considerable change at fiber and yarn stage respectively. It was also concluded that acid dyed animal fiber (UAS sheep breed wool) exhibited very good fastness to wash, poor to excellent fastness to sunlight and excellent fastness to hot pressing.

Burkinshaw *et al* (2001) dyed nylon 6, 6 knitted fabrics using three, levelling dyes and three milling dyes. Later the fabric was after treated with a commercial syntan as well as a newly developed full backtan. When the dyed sample was subjected to five, consecutive ISO 105:C06/C2 wash tests, it was found that the new back tanning process imparted greatest wash fastness improvement. The concentration of acid dyes were a major influence on the direction and degree of the structural transformation of polyamide substrate.

Research findings of Nedkova *et al* (2005) on the influence of acid dyes upon some structural and physico-mechanical indices of polyamide fibers stated that the dependencies of the indices on the dye concentration were of a complex character, which reflected the gradual penetration of the dye molecules into the inter-fibrillar and intra-fibrillar amorphous areas of the structure which were different in solidity. The fluctuations in the course of the dependencies in the area of the lowest concentrations were the result of the combined influence of the processes of improvement of the crystal phase and the relaxation phenomena occurred in the most accessible amorphous areas of the fibrous structure. With the increased dye concentration, the cross-linking effect started to appear. As a result of the formation of intermolecular links of the polymer-dye-polymer type, which influenced the properties under investigation in a characteristic way. The increased dye concentration in the substrate provoked the repeated alternation of the above-mentioned phenomena in the thicker

amorphous areas of the samples, with a pronounced influence of the cross-linking effect in the highest concentrations.

A study was conducted by Motafa *et al* (2005) on aggregation behaviour of four sulphonated monoazo dye by means of visible absorption spectroscopy. The results showed that the aggregation numbers (n) were decreased as the percentage of solvents increased. Furthermore, the effect of different electrolytes was used to clarify the influence of co-solutes on the aggregation behaviour. The aggregation number was found to obey the following order: monosulphonic > disulphonic > trisulphonic acid dyes.

Dar *et al* (2006) worked on dyeing and fastness properties of acid dyeable polypropylene nanocomposite. PP nanocomposite films with various clay concentrations were dyed with four acid dyes. The effect of clay add-on, ultrasonication time and dye fixation time on the amount of dye absorbed by the PP nanocomposites were investigated in relation to dye structures. It was found that increasing the clay content from 0 percent to 8 percent had a significant effect on the dye uptake. Ultrasonication time and dye fixation time had no immediate effect on the colour yield of acid dyes although comparatively more uniform dyeing was obtained with increased ultrasonication. Fairly reasonable wash fastness results were achieved with best ratings.

Feiz and Zeinab (2006) observed the improvement in the wash fastness of direct and acid dyed silk when it was treated with syntan, syntan/cation, and full backtan processes. Results showed that after treatment of the acid and direct dyes on silk with full backtan improved their wash fastness. Back tanning system showed greatest wash fastness improvement toward wash testing. After treatment of the dyes with a commercial syntan improved the wash fastness and sequential application of a cationic compound to the syntan dyeing fibers caused further improvement in wash fastness.

The effect of the interaction between acid dye and nonionic surfactants on the adsorption of dye in wool fiber was observed by Musnickas *et al* (2008). The results revealed that the extent of dye/surfactant interaction was significant when the molar ratio was 1:1, and the concentration of alkoxyethoxylates used was lower than the critical micellar concentration. The alkylaminethoxylates used in this study mostly manifested a higher ability to form complexes with the dye compared with the alkoxyethoxylates. With an increased temperature, significant changes in the extent of dye/surfactant interaction occurred in terms of the conductance ratio measurement.

Research was conducted on synthesis and application of acid dyes based on 3-(4-Aminophenyl)-5-benzylidene-2-substituted phenyl-3, 5-dihydroimidazol-4-one by (Wadia and Patel 2008). The results showed that the exhaustion (%E) of the dyes on wool fibers increased with decreasing pH of application and that fixation (%F) of the dyes on wool fibers increased with

increasing pH of application. Results displayed that the highest total fixation efficiency was achieved at pH 5. Light and wash fastness grades were between 4/5-5.

Effect of water hardness on the dyeing of silk with acid dye under acid, alkaline and isoelectric point dyeing conditions was studied using the zeta potential method. Under acidic conditions and in the presence of calcium ion, the positive zeta potential of silk was found to decrease with a reduction in dye adsorption. Such a phenomenon might be due to the presence of cation which increased the dyeing potential barrier at the interface between fiber and dye solution. This would result in a higher resistance of dye anions passing through the interface. Under alkaline conditions the zeta potential of silk was negative and resulted in a strong potential barrier against the dye anions. The presence of calcium ions would result in a decrease in the absolute value of the zeta potential of silk fiber, with an overall increase in dye absorption. At the isoelectric point, the zeta potential of the silk fiber was found to be near zero, and the dye adsorption was not influenced by the cations. These results showed that calcium ions could have a strong electrolytic effect on dyeing, even at very low concentrations (Wai 2008).

Patel and Bhattacharya (2009) concluded that polyurethane fiber can be successfully dyed with acid dyes with the light fastness ranged between 4/5 to 5, the washing fastness ranged between 3/4 to 4. Langmuir isotherms were found for the acid dyestuffs, which indicated ionic bonding. This was further supported by the infrared spectra for polyurethane dyed with acid dye. A characteristic absorption peak was observed for sulphonamides at 1311.5 cm⁻¹.

A study on the synthesis of acid dyes and their dyeing performance on various fibers were performed by Patel and Patel (2010) and he analysed that the acid dyes gave yellow, orange, red and purple shades with good levelling properties. Alternation in the shade depended on the coupling component used. All the dyes show moderate to very good light fastness and good to excellent washing and rubbing fastness properties. This indicated good substantivity of these dyes for wool, silk and nylon fabrics.

2.6 Effect of dyeing on physical and mechanical properties of the fabric

Dye induced changes in the mechanical and physical properties of dyed nylon 6 fibers were reported by Gulrajani *et al* (1980). He revealed that the presence of acid, disperse, and reactive dyes brought about perceptible changes in the stress-strain characteristics of the dyed fibers. From the results it was also concluded that the physical hindrance by the dye molecules was independent of the forces binding the dye to its site in the fiber phase. The dye participated in the structural rearrangements of the fiber during dyeing.

In another study on dyeing and mechanical properties of nylon 6 filaments Subramanian *et al* (1982) revealed that the percentage elongation increased as a result of

swelling treatment under slack condition in spite of increased lateral order. This was due to the slippage of chain-folded crystallites.

Todorova *et al* (1991) observed the effect of structural changes on dye diffusion in acrylic fibers. Acrylic fiber has a higher degree of orientation which proved better composed macromolecules in the polymer chain. The degree of orientation was in close correlation with the mechanical properties of the fiber and it was proportional to young's modulus, tensile strength and density but vice versa with the elongation of the fiber. The dye uptake values were enhanced by the fiber which had higher diffusion coefficients, lower orientation and lower activation energy for dyeing. Dye uptake also showed lower young's modulus and tensile strength, but higher elongation values.

Research findings of Garcia *et al* (1994) mentioned the effects of dyeing and finishing on hygral expansion and other crimp-dependent physical properties of wool fabrics. The results concluded that dyeing and finishing altered the mechanical and physical properties of a fabric by changing the crimp of its yarns, it was due to stress-relaxation forces of interyarn during the processing. Acid and chrome dyes did not affect the resulting fabric properties, but reactive dyes often had a dramatic effect on changes that took place during piece dyeing. Reactive dye and fiber interactions altered fabric crimp as a result of dyeing. In turn, crimp differences affected the resulting crimp-dependent fabric properties like hygral expansion and low-stress tensile properties.

Tsukada *et al* (2002) produced the antimicrobial active silk proteins by the use of metal-containing dyestuffs. A new technique for preparing silk fibers and films with persistent antimicrobial activity the dyeing process was discussed. In the experiment the length of the silk fibers contracted when the fibers were immersed in concentrated neutral salt solutions, such as calcium or potassium nitrate, at elevated temperature levels. The birefringence and molecular orientation of the silk fibroin molecules became less ordered by the action of the neutral salt solutions, resulting in increased dyestuff absorption. Subsequently, contracted silk fibers were dyed with metallic dyestuffs containing Cr or Cu for the purpose of obtaining silk fibers with antimicrobial activity. Silk fibers dyed with metallic dyestuffs showed significant antimicrobial activity against the plant pathogen *cornibacterium* and the human pathogen *coli bacillus*. Tensile strength of the silk fibers after the salt shrinking and dyeing processes did not show a significant change, whereas the elongation at break increased slightly.

A study on dyeing and mechanical properties of wool fiber treated with ammonia gas was carried out by Muncheu *et al* (2003). The fiber was dyed with two acid dyes and the results obtained revealed that dyeing transition temperature for both acid dyes decreased by the treatments. Results also showed that the acid dyes penetrated by the intercellular diffusion through the interscale cell membrane complex (CMC) of wool. The shearing and bending

modulus decreased a little by the treatment. The treatment was not always defective to improve the soft hand of the wool fabric.

Leksophee *et al* (2004) analysed the effects of cross linking agents, dyeing temperature and pH on mechanical performance and whiteness of silk fabric. The results indicated that improvements in the mechanical property by sodium citrate dyed silk fabric were attributed to a chemical reaction between –COOH groups and amino acid side chains in silk fabric. Increased dyeing temperature decreases the mechanical properties and whiteness. Fabric treated with sodium citrate evidently conferred a remarkable improvement in fabric abrasion resistance because sodium citrate introduced as a additional cross linking agent which repaired some degraded chemical bonds in the silk fibers. The tearing strength also showed very similar behaviour to that of abrasion resistance. It was concluded that the suitable bleaching and dyeing conditions with sodium citrate improved the mechanical properties of silk.

A research on the effect of enzyme treatment together with the dyeing process on the low-stress mechanical properties of the linen fabric was conducted by Kan *et al* (2009). The low-stress mechanical properties were assessed quantitatively, including the tensile, shearing, bending, compression and surface properties of the enzyme-treated and dyed linen fabrics. The results revealed that the enzyme treatment with subsequent dyeing altered these properties to a greater extent, depending predominantly on the concentration of enzyme used.

Choudhury and Mahajan (2008) mentioned the effect of dyeing of wool fiber and correlated it with yarn quality and performance. The dyeing had direct effect on single fiber tenacity and elongation. The weaker the fiber after dyeing, the more will be fiber breakage at the combing stage leading to the percentage noil at comber. There was deterioration in the fiber strength due to high fiber rupture after dyeing but it did not play any significant role in yarn strength.

The effect of tannic acid on the dyeing process of nylon 6 fabric with cationic dye was observed by El-Gabry and El-Zawahry (2008). The obtained results showed that the tensile strength increased as the amount of tannic acid increases from 5-30 percent (owf). This was due to the higher physical interactions between the phenolic hydroxy groups of tannic acid and carboxyl end groups of the nylon 6 fabrics. Elongation at break increased from 137% for untreated and 162 percent for pretreated fiber with 15 percent (owf) tannic acid concentration which makes the surface of the fiber more hydrophilic. The pretreated fabric with 20 percent and 30 percent (owf) tannic acid showed less elongation at break than the untreated sample. The results of both flexural rigidity and bending length of the treated fabric showed that the higher concentration of tannic acid 20-30 percent (owf) offered increase in the bending stiffness compared to those with a lower concentration of tannic acid 10-15 percent (owf). This may be due to the adsorption of large molecular weight of tannic acid,

resulting in the formation of a multilayer of tannic molecules situated at the periphery of nylon 6.

An optimized dyeing system for resistance to the physical strength loss of the PLA/cotton blended fabric was prepared by Huang *et al* (2011). The optimal dyeing systems were established according to the fixation rate of the dyes, tear/tensile strength loss, and SEM micrographs of the fabric. To avoid the strength loss during the disperse/reactive dyeing process, the recommended disperse dyeing conditions were 110°C, pH 5 for 20 min, whereas the reactive dyeing conditions were $\leq 60^\circ\text{C}$ with alkali concentration ≤ 3 g/L. In this regard, reactive dyes containing monofluorotriazine and monofluorotriazine/sulphatoethylsulphone groups were especially suitable for the reactive dyeing systems.

Periyasamy *et al* (2011) worked on the salt-free dyeing of lyocell/cotton blended fabrics using reactive dyes. Lyocell/cotton fabrics were pre-treated with PVAmHCl. This pre-treatment increased the reactivity of reactive dyes on fiber. Results concluded that wash fastness and rubbing fastness of pre-treated sample were better than that for the conventionally dyed sample. Fabric crease recovery and flexural rigidity increased as a result of pretreatment on other hand there was no change in the tensile strength of the fabric as a result of pre-treatment.

From the literature reviewed in this chapter, it is evident that lot of work has been done on dyeing of silk and wool. Similarly, research has also been conducted on dyeing of blends. However, no work has done on the dyeing of silk/wool blended fabric with synthetic dyes. Therefore a study on dyeing performance on mulberry silk waste/ wool blended fabric has been under taken.

CHAPTER III

MATERIAL AND METHODS

The present investigation was carried out to optimize the conditions for dyeing mulberry silk waste/wool blended fabric with synthetic dyes (reactive and acid dyes). To facilitate the presentation this chapter has been divided into following categories.

- 3.1 Material used for study
- 3.2 Optimization of dyeing variables
- 3.3 Dyeing of mulberry silk waste/wool blended fabric
- 3.4 Determination of fabric properties
- 3.5 Colour measurement of dyed fabric
- 3.6 Evaluation of colour fastness of the fabric
- 3.7 Statistical analysis

3.1 Materials used for study

The materials used in the study included mulberry silk waste/wool blended fabric, reactive dyes, acid dyes and various chemicals. The investigator made use of various instruments during the experiment. The details of the materials and methods are discussed below:-

3.3.1 Textile material

Hand woven blend of mulberry silk waste/wool in the ratio of 65:35 was used for the research work. For this purpose mulberry silk waste was procured from Srinagar (Jammu and Kashmir) and wool fiber was procured from the local market of the Ludhiana city. Blending and spinning was done at Kullu (Himanchal Pradesh) and weaving was done in Ludhiana. Preliminary data of fabric was collected using standard test methods, details of which have been mentioned later in this chapter.

3.3.2 Selection of Dye

Four dyes were selected for the study. Details of these have been given in table 3.1 and chemical structure of the dyes has been illustrated in figure 3.1. These dyes were obtained from local market of Ludhiana. Acid and reactive dyes were selected in red and green colour. These two colours were selected to include both the dark and light shades.

Table 3.1 Dye material used in the study

Name of the dye	C.I NO.	Molecular structure	Rs/kg
Cold Reactive dye	Reactive Red 2	$C_{19}H_{10}Cl_2N_6O_7S_2Na$	260
Hot Reactive dye	Reactive Green 19	$C_{40}H_{23}Cl_2N_{15}O_{19}S_6Na$	180
Levelling acid dye	Acid Green 16	$C_{37}H_{34}ClN_2O_6S_2Na$	300
Milling acid dye	Acid Red 131	Not available	260

Figure 3.1 Chemical structures of dyes

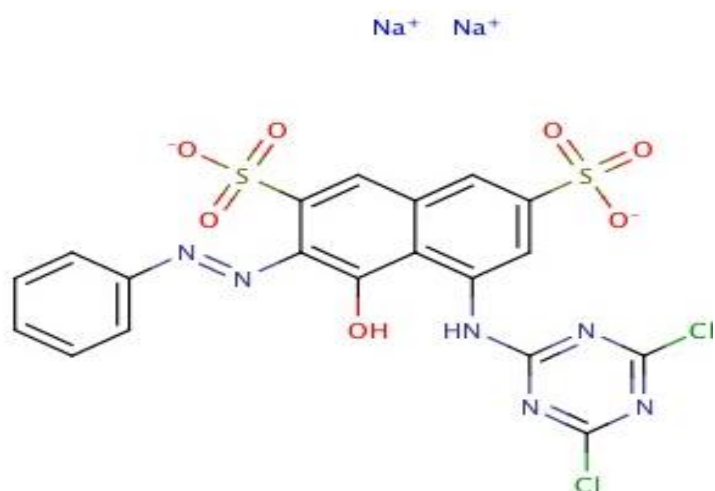


Figure 3.1 (a) Chemical structure of Reactive Red 2

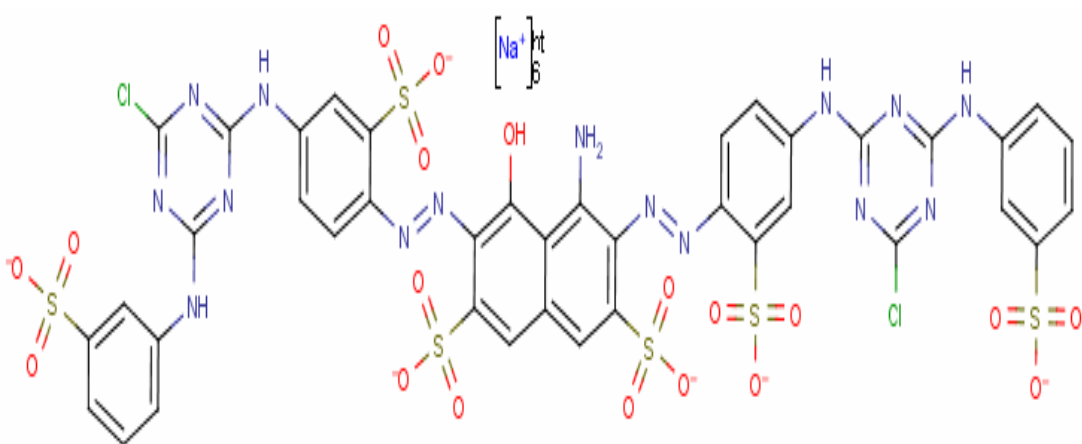


Figure 3.1 (b) Chemical structure of Reactive Green 19

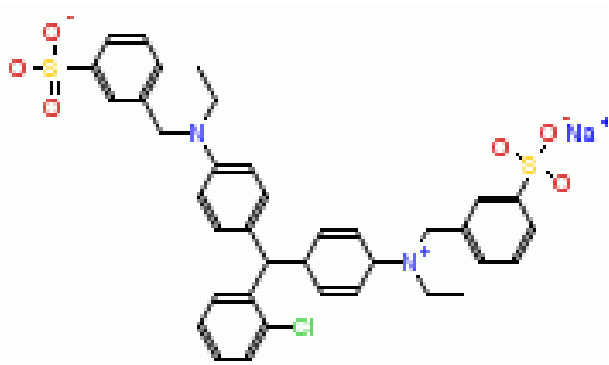


Figure 3.1 (c) Chemical structure of Acid Green 16

3.1.3 Chemicals used

The chemicals used in this study have been given in table 3.2. These chemicals have been supplied by Thames chemicals. All the chemicals used were of laboratory grade.

Table 3.2 Chemicals used in the study

Name of the Chemical	Molecular formula	Purpose
Acetic acid	CH ₃ COOH	pH regulator
Ammonium sulphate	(NH ₄) ₂ SO ₄	Dye fixation
Hydrogen sulphite	H ₂ SO ₄	Dye fixation
Caustic soda	NaOH	pH regulator
Sodium carbonate	Na ₂ CO ₃	Dye fixation
Sodium sulphate	NaSO ₄ .10H ₂ O	Colour depth and exhausting agent
Sodium chloride	NaCl	Perspiration fastness
Sodium bicarbonate	NaHCO ₃	Perspiration fastness

3.1.4 Instruments used

The details of instruments used in the study have been furnished in table 3.3.

Table 3.3 Instruments used in the study

Name of equipment	Make	Purpose	Test method
Colour flex spectrophotometer	Hunter Lab Colorimeter	CIE Lab Values and K/S values	Equipment Manual
Crockometer	Paramount Instruments Pvt. Ltd., New Delhi	Rubbing fastness	IS: 766-1988
Electronic balance 200B	Adair Dutt Instruments Pvt. Ltd. Calcutta	Weight per unit area of fabric (GSM)	IS: 1964: 1970
Tensile strength tester	Prolific Engineers Noida UP	Tensile Strength	IS: 1969-1985
Thickness tester	Paramount Instruments Pvt. Ltd., New Delhi	Fabric thickness	BS:2544:954
Fabric stiffness tester	-do-	Fabric stiffness and bending length	ASTM-D1388-64
Drape meter	-do-	Fabric drape	Equipment Manual
Crease recovery tester	Texlab Industries Ahmedabad	Wrinkle recovery	ASTM 1295-67
Hot air oven	Labmaster Instrument pvt. Ltd.,Ambala	Drying the material	Equipment Manual
Digital pH meter	-do-	Measuring the ph of dye solution	Equipment Manual
Launderometer	Paramount Instruments Pvt. Ltd., New Delhi	Washing fastness	IS: 3361- 1979
Digital light fastness tester	-do-	Light fastness	IS: 2454- 1985
Perspirometer	-do-	Perspiration fastness	IS: 971- 1985

3.1.5 Identification of textile material

Chemical tests (Bernard 1983) were conducted to check the composition of textile material and it was confirmed that the percentage of blended material was 65 percent silk and 35 percent wool.

3.1.6 Preparation of samples

Wetting/soaking - The sample was soaked in water to remove air and soften the fabric to facilitate the absorption of dye.

3.1.7 Preparation of stock solution

Solutions with accurately known concentrations are referred to as standard (stock) solutions. Stock solutions are frequently diluted to solutions of lesser concentration for experimental use in the laboratory. During the experiment 1 percent dye stock solution was prepared, by weighing 1 gm dye on weighing balance and then transferring it to the beaker containing 100 ml distilled water. The solution was stirred continuously until the dye was dissolved completely.

3.2 Optimization of variables for dyeing

Experiments were conducted to determine optimum values of four variables, viz pH of dyeing solution, dyeing temperature, dyeing time and dye concentration. These variables were optimized for both the groups of dyes on the basis of CIE Lab, K/S values and wash fastness grades. The procedure for optimization of each variable has been discussed below-

3.2.1 Optimization of dyeing variables for reactive dyes

3.2.1.1 Optimization of pH for reactive dyes

The pH optimization for cold reactive dyes was done at room temperature (about 25°C - 30°C) using soda ash (Anonymous 2007). The required amount of stock solution was pipited out in three beakers and their pH was adjusted to 5, 6 and 7 with a digital pH meter. Dyeing was carried out at room temperature for 1 hour in high temperature high pressure dyeing machine (HTHP dyeing machine). This machine was used for even dyeing and for continuous stirring. Cold reactive dyes are highly reactive and require comparatively milder conditions for dye fixation.

The pH optimization for hot reactive dye was done similarly as for cold reactive dye.

The experiment was replicated for both the types of dyes. After the completion of dyeing, all the samples were dried and CIE Lab values and K/S values and wash fastness of all the samples were determined. The pH value at which the samples gave the best results was considered as the optimized pH value.

3.2.1.2 Optimization of temperature for reactive dyes

Temperature was not optimized for cold reactive dye as these are dyed at room temperature.

After the optimization of dyeing pH for hot reactive dye, temperature was optimized. Stock solution was prepared using the optimized pH value. This stock solution was poured into three beakers, maintaining MLR 1:30. The treatment was carried out at three different temperatures i.e. 70°C, 80°C and 90°C for 60 minutes in HTHP dyeing machine.

The experiment was repeated two more times and the temperature which gave the best results on the basis of CIE Lab values, K/S values and wash fastness grades, was selected for further experiment.

3.2.1.3 Optimization of dyeing time for reactive dye

After the optimization of pH and temperature, dyeing time was optimized. For dyeing with cold reactive dye, stock solution was prepared having optimized pH. The stock solution was pipetted out and poured into four beakers, maintaining MLR 1:30. The dyeing was carried out for three different time durations, i.e. 60, 70 and 80 minutes in HTHP dyeing machine at 30°C.

While using hot reactive dye, dyeing was carried out for 60, 70 and 80 minutes at the earlier optimized temperature. The procedure of dyeing was same for both the dyes.

The experiment was repeated two more times for each of the dyes. The time at which best results were obtained for CIE Lab values, K/S values and wash fastness, was selected for the final treatment.

3.2.1.4 Optimization of dye concentration for reactive dyes

Upon the subsequent optimization of dyeing pH, temperature, time, the dye concentration was optimized. Stock solution of different dye concentrations 1, 2, 3, 4, 5 and 6 percent were prepared for cold reactive dyes (owf). These were separately poured into six beakers. Six samples of same weight were entered in to different dye concentration solutions, maintaining the MLR 1:30. The samples were dyed in HTHP dyeing machine using optimum pH, time and temperature.

The same procedure was followed to optimize the dye concentration for the hot reactive dyes.

This experiment was repeated two more times to get three replications for both the dyes. All the samples were then tested for CIE Lab, K/S values and wash fastness grades. The concentration at which the samples gave best results was the considered the optimum value of dye concentration.

3.2.2 Optimization of dyeing variables for acid dyes

3.2.2.1 Optimization of pH for acid dyes

For optimization of pH for levelling acid dye, the required amount of stock solution was pipetted out in three different beakers using the method mentioned in section 3.1.7. The pH of each beaker was adjusted to 5, 6 and 7 values. The samples were dyed in three pH separately, using HTHP dyeing machine at 70°C for 60 minutes.

The same procedure was followed for optimization of pH for milling acid dyes.

The experiment was replicated twice for both the dyes. After dyeing all the samples were dried. Later CIE Lab, K/S value and wash fastness grades of the samples were determined. The pH value at which the samples gave the best result was considered optimum.

3.2.2.2 Optimization of temperature for acid dyes

Following the optimization of pH, temperature was optimized for levelling acid dye. Stock solution having optimized pH was prepared. This stock solution was poured into three beakers, maintaining MLR 1:30. The treatment was carried out at three different temperatures i.e. 70°C, 80°C and 90°C for 60 minutes in high temperature high pressure dyeing machine.

The same course of action was followed to optimize the temperature for milling acid dye.

The experiment was repeated two more times for better results. The temperature which gave the best results with regard to CIE Lab values, K/S values and wash fastness grades was selected for final treatment.

3.2.2.3 Optimization of dyeing time for acid dyes

After the optimization of pH and temperature, dyeing time was optimized for levelling acid dye. Dye solution having optimized pH was prepared. This stock solution was poured into three beakers, maintaining MLR 1:30. The treatment was carried out for three different time durations i.e. 60, 70 and 80 minutes in HTHP dyeing machine.

Optimization of dyeing time for milling acid dyes was also done in a similar manner. The time duration which gave the best results for dyeing time on the basis of CIE Lab, K/S values and wash fastness was selected for further treatment.

3.2.2.4 Optimization of dye concentration for acid dye

For optimization of dye concentration for both levelling and milling acid dyes, the required amount of stock solution of optimum pH was taken in six beakers so as to make the dye solution of 1, 2, 3, 4, 5 and 6 percent (owf). Six samples, approximately of the same weight, were put in different dye solutions, maintaining the MLR 1:30. Sample was dyed in HTHP dyeing machine using optimum temperature and time.

This experiment was repeated two more times to get three replications for both the dyes. All the samples were then tested for CIE Lab, K/S values and wash fastness. The dye concentration at which the samples gave best result on the basis of CIE Lab, K/S values and wash fastness grades was selected for final dyeing.

3.3 Dyeing of mulberry silk waste/wool blended fabric

To study the dyeing performance on mulberry silk waste/wool blended fabric and assess the fastness properties, the blended fabric was dyed with reactive and acid dyes separately. The dyeing was carried out using the parameters which were optimized earlier.

The fabric of mulberry silk waste /wool blend was dyed with reactive and acid dyes and the effect of dyeing on fabric appearance, physical and mechanical properties were studied.

3.3.1 Dyeing of silk/wool blended fabric using reactive dyes

Dyeing procedure for dyeing mulberry silk waste/wool blended fabric using reactive dyes was followed as reported Anonymous (2002).

3.3.1.1 Recipe for cold reactive dyeing

Dye	:	x % (owf)
Liquor ratio	:	1:30
Dyeing pH	:	5-7
Sodium sulphate:		40g/l
Soda ash	:	3g/l
Temperature	:	Room temperature
Time	:	60-80 minutes

3.3.1.2 Procedure for dyeing silk/wool blended fabric with cold reactive dyes

The dye bath was set up with required quantity of dye solution having MLR 1:30. The wet test sample was entered into the dye bath at room temperature and worked for 10 minutes. After 10 minutes salt solution was added at two stages with a gap of 10 minutes between each. After 30 minutes, soda ash was added to the dye bath for fixing the colour. The material was then worked for 40 minutes.

The sample was then worked in 1:30 MLR with 6 per cent soap solution at boiling temperature for 30 minutes. Later it was washed thoroughly and shade dried. Soaping was a necessary procedure to remove remaining particles of acid, alkalis and other chemical reagents after dyeing.

3.3.1.3 Recipe for hot reactive dyeing

Dye	:	x % (owf)
Liquor ratio	:	1:30
Dyeing pH	:	5-7
Sodium sulphate:		30g/l
Soda ash	:	2g/l
Temperature	:	70°C-90°C
Time	:	60-80 minutes

3.3.1.4 Procedure for dyeing silk/wool blended fabric with hot reactive dyes

The dye bath was setup with required amount of dye. The wet test sample was entered into the dye bath and the temperature was raised to 90°C. After 10 minutes half portion of sodium sulphate (Glauber's salt) was added, to increase the colour depth and for better exhaustion, then the sample was worked for 10 minutes. Later the remaining portion of

sodium sulphate was added and the sample was worked for half an hour. Soda ash was added to the dye bath for fixing the colour and dyeing was carried out for another 40 minutes. After dyeing, the sample was taken for washing and soaping to remove the excess dye.

3.3.2. Dyeing of silk/wool blended fabric using acid dyes

Dyeing of mulberry silk waste/wool blended fabric was carried out using recipe given by Ketey and Shambu (2010) and Anonymous (2002).

3.3.2.1 Recipe for levelling acid dyeing

Dye	:	x % (owf)
Liquor ratio	:	1:30
Dyeing pH	:	5-7
Sulphuric acid	:	4%
Sodium sulphate:		20g/l
Temperature	:	70°C-90°C
Time	:	60-80 minute

3.3.2.2 Procedure for dyeing silk/wool blended fabric with levelling acid dyes

Optimized amount of stock solution was taken to prepare the dye bath to which required amount of sulphuric acid and sodium sulphate was added. The fabric was entered at 40°C and the temperature of the dye bath was raised to 90°C. Fabric was kept at 90°C for 30 minutes. Again after 20 minutes remaining sulphuric acid was added to the dye bath. Dyeing was carried out for another 10 minutes. After dyeing soaping of fabric was done and then the fabric was thoroughly rinsed in luke warm water followed by rinsing in cold water and were dried in shade.

3.3.2.3 Recipe for milling acid dyeing

Dye	:	x % (owf)
Liquor ratio	:	1:30
Dyeing pH	:	5-7
Acetic acid (96%):		3%
Sodium sulphate:		20g/l
Temperature	:	70°C-90°C
Time	:	60-80 minute.

3.3.2.4 Procedure for dyeing silk/wool blended fabric with milling acid dyes

The milling acid dye was mixed with required amount of hot water to form a paste without lumps and then made up to MLR 1:30 by adding remaining amount of cold water. The dye bath was set with required quantity of water, acetic acid and sodium sulphate. Then the dye paste was added to dye bath. The wet test sample was entered into dye bath at 45°C. The temperature was raised to 90°C within 30 minutes and the dyeing was continued at this temperature for 30 minutes.

After dyeing, soaping was done to remove particles of acid, alkalis and other chemical reagents after dyeing. It followed same procedure as discussed under section 3.3.1.2. The sample was rinsed thoroughly in cold water and shade dried.

3.4 Determination of fabric properties

Dyed and undyed both fabrics were studied for physical and mechanical properties. The physical properties studied were fabric weight, fabric thickness, bending length, flexural rigidity, drapability, crease recovery and cover factor. The mechanical properties studied were tensile strength and elongation.

3.4.1 Physical properties

3.4.1.1 GSM (IS: 1964: 1970)

The weight (GSM) is defined as the weight of specimen in one square meter of length. The sample was conditioned to maintain moisture equilibrium in standard atmosphere for 24 hours and then weighed. The principal of weighing grams per square meter of specimen is expressed in Test Method IS: 1964: 1970. The fabric was layed on a smooth surface. Square swatches of 25 x 25 cm were marked with the help of template. Swatches were then cut and conditioned. Weights of standard swatches were taken to an accuracy of 0.5 g (Nadiger and Subramanium 2001).

The GSM was calculated by using the following formula:-

$$\begin{aligned} 25\text{cm} \times 25\text{cm} &= \text{'a' grams} \\ \text{Weight/square meter} &= 16 \times \text{'a' grams/meter square} \end{aligned}$$

3.4.1.2 Fabric Thickness (B.S. 2544:954)

The thickness of a textile fabric is determined by a precise measurement of the distance between two plane parallel plates when these are separated by the cloth, a known arbitrary pressure between the plates being applied and maintained. It is convenient to regard one of the plates as the pressure foot and the other as the anvil. The principle of the measurement of the fabric thickness is expressed in test method B.S. 2544:954.

Using this method, the thickness of the fabric was measured with a Thickness Tester consisting of a graduated dial gauge of 10 mm capacity with a least count of 0.01 mm. The pressure foot is 5 cm² in area and a pressure from 20-100g/cm² i.e. a load of 100 g to 500 g can be applied. It has a base and a frame which is heavy enough to be stable, supporting a stationary circular plate or anvil, to fix it. The sample rests on this plate. A vertical sliding circular foot, with accurate surface rests on the top of the specimen. The later turns a dial pointer thus, giving the thickness of the sample under measurement i.e. the distance between the foot and the anvil. The measurements must be made at five different portions of the fabric is calculated by taking the mean of the five measurements.

3.4.1.3 Bending Length and Flexural Rigidity (ASTM- D1388- 64)

Bending length is the length of the fabric that will bend under its own weight to a definite extent. It is the measure of the stiffness that determines draping quality. The bending length of the fabric was determined by the Stiffness Tester using ASTM- D1388- 64 test method.

Flexural rigidity is a measure of stiffness associated with the handle. A rectangular strip of fabric 6 inch x 1 inch is mounted on a horizontal platform. It is supported by two side pieces made of plastic. A mirror is attached to the instrument which enables to view both index lines from a convenient position. The scale of the instrument is graduated in centimeters of bending length and it also serves as the template for cutting the specimens to size. The specimen is put on a platform in such a way that it overhangs like a cantilever and bends downward. The length of overhanging portions (l) when it is depressed under its own weight and the angle between the lines joining the tip of the edges of the platform θ is measured (Booth 1996). Three specimens warp way and three wefts way are usually tested. Each specimen is tested four times, at each end and again with the strip turned over. Mean values for the bending length in warp and weft directions can be calculated for the values of flexural rigidity.

$$\text{Bending length } C = Lf_1(\theta)$$

$$\text{where } f_1(\theta) = 4l^{1/2}$$

$$\text{also, } 4l^{1/2} = 1/2$$

$$\text{therefore, } C = L/2$$

L = Mean length of the overhanging portions.

$$W = \frac{\text{Average weight of samples}}{\text{Average area of samples}} \text{ g/cm}^2$$

Flexural rigidity in warp direction (G_1)

$$(G_1) = W_1 \times C^3 \times 10^3 \text{ mg/cm}$$

Flexural rigidity in weft direction (G_2)

$$(G_2) = W_2 \times C^3 \times 10^3 \text{ mg/cm}$$

Overall flexural rigidity (G_0)

$$(G_0) = \sqrt{G_1 \times G_2}$$

3.4.1.4 Crease Recovery (ASTM 1295-67)

Wrinkle recovery is the resistance to and recovery from creasing depends on the rigidity while 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 ASTM 1295-67 test method. Sample was cut both in warp and weft directions from all the fabrics with a template measuring 5 cm x 2.5 cm and were 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 on the instrument and allowed to recover from the crease for 3 minutes. As it recovered 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. The mean values of 50 readings were calculated.

3.4.1.5 Drapability

Drape is the ability of a fabric to assume a graceful appearance in use. It is an important property of textile materials which allows fabric to orient itself into graceful folds or pleats as a result of force of gravity. Drapability of a fabric can be determined by using the instrument drapameter and is expressed in terms of drape co-efficient. A circular specimen of diameter 10 inch was taken which was supported on a circular disc of diameter 5 inches. When doing so, the unsupported area of the fabric drapes over the edge of the supporting disc, assumed the folded configuration due to gravity. Measuring the following areas, the drape co-efficient F can be calculated

- a) The area of the specimen, A_D
- b) The area of the supporting disc, A_d
- c) The actual projected area of the specimen, A_s

The drape co-efficient, F is the ratio between the projected area of the draped specimen and its undraped area, after the deduction of the area of the supporting disc.

$$\text{Thus, } F = \frac{A_s - A_d}{A_D - A_d}$$

3.4.1.6 Cover factor

Cover factor indicates the extent to which the area of a fabric is covered by one set of threads. The warp and weft cover was calculated by using the following formula recommended by Pierce in 1937 (Haque 2009).

Warp cover factor, $k_1 = n_1 / \sqrt{N_1}$

Where, n_1 = Ends per inch and N_1 = Cotton count

Weft cover factor, $k_2 = n_2 / \sqrt{N_2}$

Where, n_2 = Picks per inch and N_2 = Cotton count

3.4.2 Mechanical properties

3.4.2.1 Tensile Strength (IS -1969 -1985)

Tensile strength is the ability of the fabric to withstand the load of force usually expressed as kilogram weight or pound weight. The tensile strength of the fabric was determined on computerized; Universal Strength Tester using IS -1969 -1985 test method.

Samples of size 325 x 60 mm each were cut from warp as well as weft direction of the fabrics. The yarns were unravelled from both sides to obtain sample of uniform width. This was done to get uniform results. Prior to the test, the specimens were conditioned for moisture equilibrium from dry side and test was carried out in standard atmosphere of 65 ± 2 per cent relative humidity at $27 \pm 2^\circ\text{C}$ temperature.

For testing a specimen was held between two clamps of the tensile strength testing machine in such a manner that the same set of yarns was gripped by both the clamps and a continual increasing load was applied longitudinally to the specimen by moving one of the clamps until the specimen ruptured. Value of tensile strength of the test specimen was read from the indicators on the machine.

In this manner, the tensile strength of all the samples were tested.

3.5 Colour measurement of the dyed samples

The method of measuring colour numerically were established by the commission internationals Del' Eclairage (CIE) in 1931 and 1976. These methods provide uniform colour difference in relation to visual difference. The measurement is precise and the colour is specified by a set of numbers, which can be used to re-create the original colours at any time and place, i.e. reproduction is possible. This method extends the use of the computer in colour matching prediction, i.e. to correct the colour of a recipe obtained by the use of the computer. The use of computer in measurement of colour thus, increases the objectivity of experimental results of dyeing.

The colour of the dyed samples were measured numerically through computerized colour matching system. This system uses CIE Lab (76) colour Space, D65 illuminant (International standard for day light published by CIE for the purpose of colour matching and appraisal) and 420 nm wavelengths to measure the actual colour and in colours. The CIE Lab colour space uses L^* , a^* , b^* scales to describe colour. The L^* is a measure of lightness/ darkness of colour of an object and ranges from 0 (black) 100 to (white), a^* is the measure of redness (+ve a^*) or greenness (-ve a^*), b^* is the measure of yellowness (+ve b^*) or blueness (-ve b^*).

The colour strength is expressed as K/S value and was assessed directly by the Spectrophotometer according to the Kubelka-Munk equation.

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

Where K and S are constants associated with the light absorption and scattering of the fabric respectively. R is the reflectance of the dyed fabric measured at the wavelength of

maximum light absorption expressed in fractional form. According to the Kubelka-Munk theory, K/S is directly proportional to the colour strength (content) of a solid surface.

The CIE Lab and K/S for all dyed sample was recorded.

3.6 Evaluation of colour fastness of the dyed sample

All the dyed sample was evaluated for colourfastness to washing, light, rubbing and perspiration by the methods prescribed by the Bureau of Indian Standards.

3.6.1 Evaluation of colourfastness to washing

For determining the washing fastness of dyed samples. Test-2 described in the Indian Standard IS: 3361-1979 was used.

For preparing a composite specimen, two fabric pieces, each measuring 10 cm x 4 cm, were taken. One of the fabric pieces taken was silk and the other wool. The dyed fabric to be tested was placed between these two pieces. The sample was sewn on all the four sides.

All the composite specimens were prepared as above were weighed and the required quantity of soap solution of 5 g/lit of water was prepared for keeping the material to liquor ratio of 1:50. One composite specimen was placed in each of the eight containers of a launderometer along with 10 steel balls and soap solution previously heated to $50 \pm 2^\circ\text{C}$ was added to it. The composite specimen was treated for 45 minutes at $50 \pm 2^\circ\text{C}$ was added to it. The composite specimen was squeezed and the stitching along two long sides and one short side was removed. The composite sample was then opened and dried in air with three pieces (one test specimen and two adjacent pieces) in contact along the remaining line of stitching.

The sample was evaluated for colour change. A piece of original dyed fabric and the test specimen were placed side on the same plane. Care was taken to keep the surrounding field neutral grey in colour. The visual difference between the original and test specimen was compared and fastness rating was given with the help of a grey scale for assessing change in colour. The fastness grade given was that number of grey scale which corresponded to the contrast between original and washed specimen. If the contrast was judged to be nearer to the non-existing half step, the approximate half step rating was given.

The different grades of grey scale indicated the following level of fastness.

Grey scale rating	Colour change	Colour staining
5	Excellent	Negligible or no staining
4	Good	Slight staining
3	Fair	Noticeable staining
2	Poor	Considerable staining
1	Very poor	Heavy staining

3.6.2 Evaluation of colourfastness to light

For determining the colourfastness of the dyed samples to light, the test prescribed by the BIS in the IS: 2454-1985 was employed.

The test sample was exposed to the light of MBTL fading lamp for standard time period (at least 5 hours). The exposure was continued until specimen faded to a correspond equal to grade 4 on the grey scale. The approximate number of hours required to fade each blue wool standard to a contrast equal to grade 4 of the grey scale on such exposure are given below:

Blue wool standard no.	No. of hours	Fastness level
1	5	Very poor
2	10	Poor
3	20	Fair
4	40	Fairly good
5	80	Good
6	160	Very good
7	320	Excellent
8	640	Outstanding

3.6.3 Evaluation of colourfastness to rubbing

Colour fastness of all the dyed samples against dry and wet crocking was determined using the test prescribed by the BIS in the IS: 766-1988.

For testing fastness to dry rubbing a test specimen was prepared, in the form of a layer the dyed fabric attached lengthwise on rectangular cardboard of size 14 cm 5 cm. the undyed cotton samples of size 5 cm x 5 cm was fixed at the base of the rubbing device. The test specimen was rubbed to and fro with undyed pieces, with a downward force of 900 grams in a straight line along a track of 10 cms for 10 times in 10 seconds. Similarly, fastness to wet rubbing was tested by rubbing wet undyed cotton sample of the test specimen. The dry and wet crocked sample was assessed against standard grey scales for colour change and colour staining. The different grades in the grey scale for change in colour and staining of colour were given as described in section 3.6.1.

3.6.4 Evaluation of colour fastness to perspiration

The fastness to perspiration was tested by the test IS: 971-1983 prescribed by the BIS. The acidic test liquor was prepared by dissolving 2.65 g of sodium chloride and 0.75 g of urea/liter and adjusting the pH of the solution to 5.6 with addition of acetic acid. The alkaline test liquor was prepared by dissolving 3 g of sodium chloride/litter and adjusting the pH of the solution to 7.2 with addition of sodium bicarbonate.

A composite specimen was prepared by placing the test specimen of 5 cm x 4cm between the two adjacent fabrics of 5 cm x 5 cm size, one being silk and other wool. The test specimen was stitched along the four sides. The test specimen were soaked in the acidic and alkaline test solutions as prepared above separately with material to liquor ratio 1:50 for 30 minutes at room temperature. Then the treated sample was kept between two glass plates of perspirometer under a force of 5 kg. The apparatus was kept in hot air oven for four hours at $37 \pm 2^\circ\text{C}$. Then the test sample was removed from the oven and air dried with temperature not exceeding 60°C . The numerical grading for colour change of test pieces and for staining of two adjacent pieces was done using a grey scale.

The acidic and alkaline perspiration sample was assessed against standard grey scales for colour change and colour staining. Different grades in the grey scale for change in colour and staining of colour were given as described in section 3.6.1.

3.7 Statistical analysis

The data for different dyed fabric tests were analyzed on the basis of statistical analysis. The mean, standard error, coefficient of variation, kurtosis, skewness, critical difference and their statistical significance were ascertained with the help of softwares CPCS1 (Cheema and Singh 1990) and GSTAT by (Cheema and Sidhu 2004).

CHAPTER IV

RESULTS AND DISCUSSION

The present study was conducted to optimize the dyeing conditions for mulberry silk waste/wool blended fabric. After dyeing the blended fabric at optimized dyeing conditions, the physical, mechanical properties and colour fastness properties were studied. These physical and mechanical properties were compared with undyed fabric. The results obtained have been discussed under the following subheads.

4.1 Optimization of the dyeing conditions for dyeing

4.2 Final dyeing of mulberry silk waste/wool blended fabric

4.3 Analysis of physical and mechanical properties of undyed and dyed fabric

4.4 Analysis of fastness properties of dyed fabric

4.1 Optimization of the dyeing conditions

Conditions for dyeing the mulberry silk waste/wool blended fabric were optimized. The parameters included dyeing pH, temperature, time and dye concentration, which were optimized on the basis of CIE Lab, K/S values and wash fastness grades. Optimization of each parameter has been discussed as under.

4.1.1 Optimization of the dyeing condition for cold reactive dye

4.1.1.1 Optimization of dyeing pH for cold reactive dye

The results pertaining to the effect of pH on dyeing, using cold reactive dye have been furnished in table 4.1. The CIE Lab values were recorded at pH 5, 6 and 7. It is evident from the table 4.1 that the brightness increased with an increase in the pH so the sample dyed at pH 7 showed highest value of L (53.120). The brightness of the fabric lesser when the samples were dyed in pH 5 and 6 and the observed value of L was found to be 49.910 and 50.830, respectively. This was due to the fact that the dye absorption on wool and silk increased with increasing pH. The highest total fixation efficiency was achieved at pH 7 which was also reported by Wadia and Patel (2008). As far as the value a was concerned, the highest (43.271) value was found at pH 7 which indicated that the sample dyed at this pH was more red as compared with the samples dyed at pH 5 and 6 (41.309 and 41.730). Looking into the b value of the sample dyed at pH 7, it was found to be -0.200, indicating that the hue at this pH was very less blue as compared with the samples dyed at pH 5 and 6. Showing a similar trend, the K/S values at pH 5, 6 and 7 were 5.330, 5.516 and 5.930 respectively. Thus the value of K/S was found to be highest at pH 7.

Table 4.1 Optimization of dyeing pH for cold reactive dye on the basis of CIE Lab and K/S values

pH	Wavelength (nm)	L	a	b	K/S values
5	520	49.910	41.309	-0.260	5.330
6	520	50.830	41.730	-0.230	5.510
7	520	53.120	43.271	-0.200	5.930

Table 4.2 revealed the wash fastness grades for the samples dyed at pH 5, 6 and 7. It is evident from the table 4.2 that the samples dyed at pH 5 and 6 possessed good grades in terms of colour change and negligible staining was found on wool fabric and slight staining was observed on silk fabric. When the wash fastness grade was evaluated for the sample dyed at pH 7 it was found that the sample had good grade in terms of colour change and the dyed sample showed slight staining on both the adjacent fabrics.

Although the samples dyed at pH 5 and 6 showed better results for wash fastness but pH 7 was considered to be optimum because there was no any marked difference among the wash fastness grades of the sample dyed at pH 5, 6 and 7. Secondly, the samples dyed at pH 7 gave best results for CIE Lab values. Thus it was considered optimum pH for dyeing.

Table 4.2 Optimization of dyeing pH for cold reactive dye on the basis of wash fastness

pH	CC	C S	
		W	S
5	4	4/5	4
6	4	4/5	4
7	4	4	4

4.1.1.2 Optimization of dyeing time for cold reactive dye

In order to optimize the dyeing time for cold reactive dye, dyeing was carried out for 60, 70 and 80 minutes, the results of which are furnished in table 4.3. The CIE Lab values at 520 nm wave length was recorded and it was found that the values remained stable in all three time durations. Cold reactive dyes are very reactive and require milder conditions for fixation (Anonymous 2007). Observed value of L was highest (48.833) for the sample dyed for 60 minutes. L value for the samples dyed for 70 and 80 minutes were found to be 48.240 and 48.000, respectively. Observing the trend in a value it was found that the value was the highest (44.346) when the sample was dyed for 60 minutes and value decreased when the sample was dyed for 70 minutes (43.750). On further increase in time duration to 80 minutes the value of a decreased to 42.232, indicating that the redness of the samples decreased with

increase in dyeing time. The value of 'b' was 1.801 for the sample dyed for 60 minutes. The values of b for the sample dyed for 70 and 80 minutes were found to be 1.900 and 2.100, respectively indicating that the sample was less yellow at 60 minutes of dyeing time and more yellow at 70 and 80 minutes of dyeing time. Looking in to the K/S values of the dyed samples, the dye absorption was the highest (7.363) for the sample dyed for 60 minutes.

Table 4.3 Optimization of dyeing time for cold reactive dye on the basis CIE Lab and K/S values

Time (min)	Wavelength (nm)	L	a	b	K/S values
60	520	48.833	44.346	1.810	7.363
70	520	48.240	43.750	1.900	7.290
80	520	48.000	42.232	2.100	7.050

The results for wash fastness grades have been depicted in table 4.4. In the case of sample dyed for 60 minutes, the grade for colour change was good whereas the staining was found to be negligible on both wool and silk fabrics. The wash fastness grades for the samples dyed for 70 and 80 minutes were good for colour change and negligible staining was found on wool fabric and slight staining was observed on silk fabric. The results are in line with the findings of Richard and Stephen (2002). Thus, 60 minutes of dyeing time was optimized for dyeing the fabric with cold reactive dye.

Table 4.4 Optimization of dyeing time for cold reactive dye on the basis of wash fastness

Time (min)	CC	C S	
		W	S
60	4	4/5	4/5
70	4	4/5	4
80	4	4/5	4

4.1.1.3 Optimization of dye concentration for cold reactive dye

The results of CIE Lab and K/S values for different dye concentrations of cold reactive dye have been furnished in table 4.5. The dyeing was carried out at the optimized pH and time for six dye concentrations separately. CIE Lab values were recorded for all the six dye concentrations. These indicated that with the highest L (52.410), a (49.415) and b (3.781) values the sample was the darkest when dyed at 6 percent dye concentration. The value of K/S was also found to be highest (49.005) for this dye concentration. According to the Kubelka-Munk theory, K/S is directly proportional to the colour strength (Marie *et al* 2008).

Table 4.5 Optimization of dye concentration for cold reactive dye on the basis CIE Lab and K/S values

Concentration (%)	Wavelength (nm)	L	a	b	K/S values
1	520	34.905	43.387	-.339	6.1777
2	520	36.876	45.786	1.665	15.681
3	520	38.098	46.098	2.815	19.012
4	520	42.278	46.568	2.870	24.010
5	520	48.2450	48.867	3.521	32.340
6	520	52.410	49.415	3.781	49.005

After the evaluation of wash fastness grades at different dye concentrations mentioned in table 4.6, it was observed that the sample dyed at 6 percent dye concentration showed fair to good grade for colour change and noticeable to slight staining on both wool and silk fabrics. The wash fastness grade for the samples dyed at 4 percent dye concentrations was found to be good for colour change whereas, slight staining was found on both wool and silk fabrics. The wash fastness grade for the sample dyed at 4 percent dye concentration was better than the samples dyed at 6 percent dye concentration. Thus, 4 percent dye concentration was considered optimum and the observed value of CIE Lab for 4 percent dye concentration were found to be 42.278, 46.568 and 2.870, respectively. At this dye concentration the K/S value was 24.010.

Table 4.6 Optimization of dye concentration for cold reactive dye on the basis of wash fastness

Concentration (%)	CC	C S	
		W	S
1	4	4	4
2	4	4	4
3	4	4	4
4	4	4	4
5	4	4	3/4
6	3/4	3/4	3/4

4.1.2 Optimization of dyeing conditions for hot reactive dye

4.1.2.1 Optimization of dyeing pH for hot reactive dye

The CIE Lab and K/S values regarding the optimization of pH for hot reactive dye are shown in table 4.7. The highest value (45.333) of L at pH 5 indicated that it was darkest among

all the samples dyed at pH 5, 6 and 7. The sample dyed at pH 5 was greener with the highest a value (-12.367). The value of 'a' decreased for the samples dyed at pH 6 and 7 indicating that the samples were less green. The value of b for the sample dyed at pH 5 was -2.310, which indicated that colour was more toward blue as compared to the sample dyed at pH 6 and 7. Colour strength (K/S) was also maximum (6.300) for the samples dyed at pH 5.

Table 4.7 Optimization of dyeing pH for hot reactive dye on the basis of CIE Lab and K/S values

pH	Wavelength (nm)	L	a	b	K/S values
5	520	45.333	-12.367	-2.310	6.300
6	520	43.857	-11.433	-2.060	6.167
7	520	42.367	-11.600	-1.920	6.000

Data pertaining to wash fastness grades for the samples dyed at pH 5, 6 and 7 are depicted in table 4.8 and the furnished results showed that the grades for colour change after washing was excellent for all the samples dyed at different pH. In terms of colour staining no staining was observed on both wool and silk fabrics. Hence, the pH 5 was considered optimum for dyeing the samples with hot reactive dye. Results were supported by the findings of Ahamad *et al* (2001). Uddin and Hossain (2011) in their study stated that higher pH may damage the wool in the mulberry silk waste/ wool blended fabric.

Table 4.8 Optimization of dyeing pH for hot reactive dye on the basis of wash fastness

pH	CC	C S	
		W	S
5	5	5	5
6	5	5	5
7	5	5	5

4.1.2.2 Optimization of dyeing temperature for hot reactive dye

It can be analysed from table 4.9 that the mulberry silk waste/wool blended fabric gave the best result at dyeing temperature 90°C than dyeing temperature 70°C and 80°C. The value of L increased with the increase in dyeing temperature thus indicating that the sample became the darkest when dyed at temperature 90°C with mean value 45.160. The values of L decreased at dyeing temperature 70°C and 80°C which were observed as 43.990 and 44.330, respectively. The higher negative value of a (-13.543) and b (-1.900) for the samples dyed at 90°C indicated that the sample was more green and more blue. Todorova *et al* (1991) recorded that the dye uptake values increased with increasing dyeing temperature and hot

reactive dyes are designed to give high fixation by exhaust dyeing methods when applied at the temperature between 85°C to 90°C.

The observed value of K/S of the sample dyed at 90°C was 7.871, depicting the maximum dye absorption at this temperature as compared to the dye absorption at 70°C and 80°C where the K/S value was 6.600 and 6.234, respectively.

Table 4.9 Optimization of dyeing temperature for hot reactive dye on the basis of CIE Lab and K/S values

Temperature (°C)	Wavelength (nm)	L	a	b	K/S values
70	520	43.990	-12.786	-1.810	6.600
80	520	44.330	-12.500	-1.100	6.234
90	520	45.160	-13.543	-1.900	7.871

Table 4.10 represents the results of wash fastness. The results of wash fastness were similar for all the samples dyed at three temperatures i.e. 70°C, 80°C and 90°C. The data in the table showed that change in colour after washing was excellent and no staining was observed on both wool and silk fabrics. Dyeing results were the best at 90°C thus, it was considered optimum for further work.

Table 4.10 Optimization of dyeing temperature for hot reactive dye on the basis of wash fastness

Temperature (°C)	CC	C S	
		W	S
70	5	5	5
80	5	5	5
90	5	5	5

4.1.2.3 Optimization of dyeing time for hot reactive dye

It can be elicited from the table 4.11 that the observed value of L for the samples dyed for 60 minutes was 42.070, which increased to 42.237 when the dyeing time was increased to 70 minutes. A further increase in the dyeing time to 80 minutes led to a decrease in the L value (41.733). Thus, the sample was the brightest when dyed for 70 minutes. The observed value of 'a' at 70 minutes dyeing time was found to be -13.510, which indicated that the sample was more green as compared to the samples dyed for 70 and 80 minutes where the value of a was found to be -13.510 and -1.345, respectively. The recorded value of b was -1.345 for the sample dyed for 70 minutes which showed that the sample was bluer.

The observed value of K/S was 5.800 for the sample dyed for 60 minutes. The K/S value increased to 7.100 for the sample dyed for 70 minutes and decreased to 6.600 when dyeing time was increased to 80 minutes. Findings of Marie *et al* (2008) revealed that the colour strength increased with raising the dyeing time, after reaching to its maximum, the colour strength started decreasing. According to Choudhury (2006) reactive dyeing should be carried out between 60 to 90 minutes for best exhaustion.

Table 4.11 Optimization of dyeing time for hot reactive dye on the basis of CIE Lab and K/S values

Time (min)	Wavelength (nm)	L	a	b	K/S values
60	520	42.070	-12.830	-1.283	5.800
70	520	42.237	-13.510	-1.345	7.100
80	520	41.733	-12.850	-1.310	6.600

The results pertaining to the wash fastness grades of samples dyed for 60, 70 and 80 minutes of dyeing time have been shown in table 4.12. The results depicted that wash fastness of the samples dyed for 60, 70 and 80 minutes in terms of colour change was excellent and no staining was found on both wool and silk fabrics. Thus, 70 minutes time was considered optimum for dyeing the fabric with hot reactive dye.

Table 4.12 Optimization of dyeing time for hot reactive dye on the basis of wash fastness

Time (min)	CC	C S	
		W	S
60	5	5	5
70	5	5	5
80	5	5	5

4.1.2.4 Optimization of dye concentration for hot reactive dye

It is apparent from the table 4.13 that the CIE Lab and K/S values of dyed samples increased with the increase in dye concentration. The sample dyed at 6 percent dye concentration showed the highest value for the CIE Lab. The value of L for 6 percent dye concentration was found to be 51.021, which was maximum and showed that the sample was brightest as compared to the samples dyed at other concentrations. Recorded a and b values were -14.987 and -2.00, respectively thus, the sample was more greener and more blue. A similar trend was observed for dye absorption where K/S value was found to be maximum (14.200) at 6 percent dye concentration.

Table 4.13 Optimization of dye concentration for hot reactive dye on the basis of CIE Lab and K/S values

Concentration (%)	Wavelength (nm)	L	a	b	K/S values
1	520	40.245	-13.612	-0.781	5.200
2	520	42.176	-13.342	-0.423	6.100
3	520	44.575	-14.021	-1.200	12.400
4	520	44.278	-14.716	-1.151	12.400
5	520	48.955	-14.200	-1.349	14.200
6	520	51.021	-14.987	-2.000	14.200

It can be concluded from that table 4.14 that an increase in dye concentration resulted in decrease in wash fastness grades. Samples dyed at 5 and 6 percent concentration showed good grades for colour change and negligible staining on wool fabric whereas slight staining on silk fabric. The sample dyed at 4 percent concentration showed good to excellent grade in terms of colour change and negligible staining was observed on both wool and silk fabrics, whereas the sample dyed at 3 percent dye concentration showed excellent grade for colour change and no staining on both the adjacent fabrics. At this concentration the value of L was 44.575, which indicated its darkness. Greenness was analysed in terms of 'a' value (-14.021) and the recorded b value was -1.200 which indicated its blueness. The value of the K/S for the sample dyed at 3 percent concentration was found to be 12.400. Considering the all above parameters 3 percent dye concentration was thought to be the best dye concentration for hot reactive dyeing.

Table 4.14 Optimization of dye concentration for hot reactive dye on the basis of wash fastness

Concentration (%)	CC	C S	
		W	S
1	5	5	5
2	5	5	5
3	5	5	5
4	4/5	4/5	4/5
5	4	4/5	4
6	4	4/5	4

4.1.3 Optimization of dyeing variable for levelling acid dye

4.1.3.1 Optimization of dyeing pH for levelling acid dye

The colour parameters of the dyed mulberry silk waste/wool blended fabric with levelling acid dye at different pH values were evaluated. Table 4.15 shows that the value of L was highest (52.459) at pH 5 that dropped to 51.541 and 50.416 when pH was increased to 6 and 7, respectively. The dye uptake was more in acidic medium (pH 5) as at this pH, covalent bonding of the dye molecules with the fiber predominates (Hunger 2003). Abdullah (2006) reported that higher acidic and alkali media can cause severe damage to protein fibers. The negative value of a (-15.538) and b (-8.732) of the sample dyed at pH 5, indicated that the sample was less green and less blue as compared to that when dyed at pH 6 and 7. The value of K/S was observed to be the highest (5.310) for the sample dyed at 5 pH indicating that the dye absorption was maximum at pH 5 as compared to the samples dyed at pH 6 and 7.

Table 4.15 Optimization of dyeing pH for levelling acid dye on the basis CIE Lab and K/S values

pH	Wavelength (nm)	L	a	b	K/S values
5	520	52.459	-15.538	-8.732	5.310
6	520	51.514	-15.560	-9.978	4.923
7	520	50.416	-15.746	-10.342	4.298

Data furnished in table 4.16 show the wash fastness results of levelling acid dyed samples. The samples dyed at 5, 6 and 7 pH showed good grades for colour change, whereas a slight staining was observed on wool fabric and noticeable to slight staining was observed on silk fabric. Hence, the pH 5 was considered to be optimum for dyeing the fabric with levelling acid dye as the dye absorption was maximum at pH 5 and the wash fastness grades were same for all the pH.

Table 4.16 Optimization of dyeing pH for levelling acid dye on the basis of wash fastness

pH	CC	C S	
		W	S
5	4	4	3/4
6	4	4	3/4
7	4	4	3/4

4.1.3.2 Optimization of dyeing temperature for levelling acid dye

The CIE Lab and K/S values regarding the optimization of temperature are shown in table 4.17. Data in the table show that the sample dyed at 90°C had the highest value of L (53.876) which means that it was darker as compared to the samples dyed at 70°C and 80°C. In terms of a value for the samples dyed at 90°C, it was found to be more green as depicted by its higher negative a value (-16.147) as compared to dyeing temperature 70°C (-15.890) and 80°C (-15.543). As far as b value was concerned, more bluish value was depicted by the negative value (-11.498) of the sample dyed at 80°C however not much difference was found between the b values of samples dyed at 70°C and 90°C. It was observed from the table 4.19 that K/S value increased with the increase in the dyeing temperature and it was observed maximum (7.363) for the samples dyed at 90°C.

Table 4.17 Optimization of dyeing temperature for levelling acid dye on the basis of CIE Lab and K/S values

Temperature (°C)	Wavelength (nm)	L	a	b	K/S values
70	520	50.431	-15.890	-11.452	4.924
80	520	51.669	-15.543	-11.498	5.231
90	520	53.876	-16.147	-11.453	7.363

As far as the wash fastness grades are concerned, data presented in table 4.18 reveals that the samples dyed at 70°C and 80°C possessed good wash fastness in terms of colour change and the dyed sample showed slight staining on both wool and silk fabrics. Whereas, the sample dyed at 90°C showed good grade for colour change and slight staining on wool fabric whereas slight to noticeable staining on silk fabric. Speakman and McMohan (1938) stated that at low temperature all the animal fibers offered considerable resistance to dye absorption, due to dense molecular structure. Thus, 90°C temperature was considered optimum.

Table 4.18 Optimization of dyeing temperature for levelling acid dye on the basis of wash fastness

Temperature (°C)	CC	C S	
		W	S
70	4	4	4
80	4	4	4
90	4	4	4/3

4.1.3.3 Optimization of dyeing time for levelling acid dye

It was observed from table 4.19 that value of L was highest (51.980) the sample dyed for 80 minutes, of dyeing time which indicated that this sample was darker as compared to the samples dyed for 60 and 70 minutes, where the brightness decreased. The duration of dyeing time should be between one or two hours or more depending upon the depth of colour required as recorded by Abdullah (2006). The value of a increased with the increase in dyeing time. The value of a observed was -14.876, 15.609 and -16.121 at dyeing time 60, 70 and 80 minutes respectively, which indicated that the sample was the greenest when the dyeing time was 80 minutes. The value of b was found to be maximum (-10.645) when the sample was dyed for 80 minutes which indicated its maximum blueness. Sample was less blue when dyed for 60 and 70 minutes with b value -9.980 and -10.143 respectively.

Looking into K/S values of the samples dyed for 60, 70 and 80 minutes of dyeing time showed that the sample dyed for 80 minutes had the highest value of K/S with mean value of 5.231, whereas the value of K/S was observed 4.924 and 4.934 for the samples dyed for 60 and 70 minutes, respectively.

Table 4.19 Optimization of dyeing time for levelling acid dye on the basis of CIE Lab and K/S values

Time (min)	Wavelength (nm)	L	a	b	K/S values
60	520	50.765	-14.876	-9.980	4.924
70	520	51.078	-15.609	-10.143	4.934
80	520	51.980	-16.121	-10.654	5.231

The wash fastness grades for the samples dyed for 60, 70 and 80 minutes using levelling acid dye have been furnished in table 4.20. It was observed that irrespective of increase in time duration the grades remained good for colour change after washing and slight staining was recorded on wool fabric whereas, noticeable to slight staining was found on the silk fabric for all dyeing times. Although the wash fastness results were same at all the dyeing times but the dye absorption was found maximum at 80 minutes, hence it was considered optimum for further work.

Table 4.20 Optimization of dyeing time for levelling acid dye on the basis of wash fastness

Time (min)	CC	C S	
		W	S
60	4	4	3/4
70	4	4	3/4
80	4	4	3/4

4.1.3.4 Optimization of dye concentration for levelling acid dye

It is apparent from table 4.21 that the sample dyed at 6 percent dye concentration showed the highest value of L (55.654), which was the indicator of the darkness and brightness of the dyed samples. The values of a and b were recorded as -15.729 and -12.987, respectively. It means that the sample dyed at 6 percent concentration were more green and more blue. The value of K/S also showed the increasing trend with the increase in dye concentration.

Table 4.21 Optimization of dye concentration for levelling acid dye on the basis of CIE Lab and K/S values

Concentration (%)	Wavelength (nm)	L	a	b	K/S values
1	520	51.841	-12.253	-9.490	5.297
2	520	51.897	-12.558	-10.090	7.363
3	520	52.765	-13.564	-11.321	13.303
4	520	53.435	-13.870	-12.654	19.012
5	520	54.534	-15.457	-12.765	24.010
6	520	55.654	-15.729	-12.987	32.340

It was observed from the table 4.22 that the wash fastness grades remained same for the samples dyed at 1, 2, 3 and 4 percent dye concentrations. The wash fastness grades were good for colour change and slight staining was observed on both wool and silk fabrics. The sample dyed at 4 percent dye concentration showed higher value of CIE Lab as compared to the samples dyed at 1, 2 and 3 percent dye concentration. The observed value of L at 4 percent dye concentration which indicated its brightness was found to be 53.435. Greenness was evaluated in terms of a value and was found to be -13.870 whereas blueness was observed as b value (-12.654). The colour strength measured in terms of K/S was observed as 19.012. Further it was concluded from the table 4.22 that the wash fastness grades decreased after further increase in percent dye concentration. For 5 and 6 percent dye concentration the observed wash fastness grades were fair to good for colour change and noticeable to slight staining was observed on both wool and silk fabrics. Considering the all above results, 4 percent dye concentration was optimized as optimum dye concentration.

Table 4.22 Optimization of dye concentration for levelling acid dye on the basis of wash fastness

Concentration (%)	CC	C S	
		W	S
1	4	4	4
2	4	4	4
3	4	4	4
4	4	4	4
5	3/4	4	3/4
6	3/4	3/4	3/4

4.1.4 Optimization of dyeing variable for milling acid dye

4.1.4.1 Optimization of dyeing pH for milling acid dye

In order to optimize the pH of the dye bath for milling acid dye the samples were dyed in the dye bath at pH value 5, 6 and 7 separately. The colour strength of the dyed sample at different pH was measured and the data of which has been presented in table 4.23. It is evident from the table that L value increased when the pH was increased from 5 to 7 and was found to be maximum (47.347) at pH 7 depicting that the sample was brightest. The value of a also increased with increase in pH. The observed value of a was 50.367 which showed that the dyed sample was more towards green than the rest. The greater negative value of b (-12.876) at pH 7 showed that the sample was more blue. It is evident from table 4.23 that K/S value of the dyed sample increased with increase in pH value of the dye solution and showed highest value (15.025) for the sample dyed at 7 pH.

Table 4.23 Optimization of dyeing pH for milling acid dye on the basis of CIE Lab and K/S values

pH	Wavelength (nm)	L	a	b	K/S values
5	520	42.567	49.469	-12.093	9.681
6	520	44.859	49.857	-12.054	11.520
7	520	47.375	50.367	-12.876	15.025

Table 4.24 revealed that the grades were good in terms of colour change for all the samples dyed at pH 5, 6 and 7 and negligible staining was observed on wool fabric and slight staining was observed on silk fabric. Thus, pH 7 was considered optimum pH for milling acid dyeing as the dye absorption was maximum and results of wash fastness grades were same for

all pH values. The results for wash fastness grades are in line with the research findings of Wadia and Patel (2008).

Table 4.24 Optimization of dyeing pH for milling acid dye on the basis of wash fastness

pH	CC	C S	
		W	S
5	4	4/5	4
6	4	4/5	4
7	4	4/5	4

4.1.4.2 Optimization of dyeing temperature for milling acid dye

The CIE Lab and K/S values regarding the optimization of temperature for milling acid dye are shown in table 4.25. It is evident from the table that the L value increased with the increase in dyeing temperature. The L value was 42.271, 42.324 and 50.886 for dyeing temperature 70°C, 80°C and 90°C, respectively. The recorded value of a and b was found to be highest for the sample dyed at 90°C with mean value of 52.987 and -11.204, respectively. Higher value of a indicated that the colour of dyed sample was more towards green, whereas higher negative value of b indicated that colour was more toward blue. The observed value of K/S for 90°C was also recorded highest (19.012). Gohl and Vilensky (1983) reported that temperature of the dye liquor influences fiber swelling, making it easier for the dye molecule to penetrate into the amorphous region of the fiber.

Table 4.25 Optimization of dyeing temperature for milling acid dye on the basis of CIE Lab and K/S values

Temperature (°C)	Wavelength (nm)	L	a	b	K/S values
70	520	42.271	45.358	-10.133	10.133
80	520	43.324	50.654	-11.190	19.012
90	520	50.886	52.897	-11.204	19.012

Data presented in table 4.26 revealed that the sample dyed at 70°C temperature had good to excellent grade for colour change, in terms of colour staining, negligible staining on wool whereas, slight staining was observed on silk fabric. Good to excellent washing fastness properties of acid dyes indicates good substantivity of these dyes for wool, silk and nylon fabrics (Patel and Patel 2010). Wash fastness grades for colour change was good and slight staining was observed on both wool and silk fabrics for the sample dyed at 80°C and 90°C

dyeing temperature. Although K/S value and wash fastness grades were found to be similar for the samples dyed at 80°C and 90°C dyeing temperature but at 90°C temperature the CIE Lab values showed marked difference than CIE Lab values of sample dyed at 80°C dyeing temperature and were found to be higher. Hence, 90°C temperature was considered optimum for further work. According to Patel and Bhattacharya (2009) above 90°C temperatures the fibers gets shrunk considerably due to prolonged boiling and on the other hand below 90°C the exhaustion of acid dye on the fibers was found less compared to that obtained at 90°C temperatures.

Table 4.26 Optimization of dyeing time for milling acid dye on the basis of wash fastness

Temperature (°C)	CC	C S	
		W	S
70	4/5	4/5	4
80	4	4	4
90	4	4	4

4.1.4.3 Optimization of dyeing time for milling acid dye

It could be investigated from table 4.27 that the value of L was highest (52.342) when the sample was dyed for 80 minutes which indicates that it was the darkest than the samples dyed for 60 minutes (42.271) and 70 minutes (50.886). The highest positive value of a (50.886) and negative value of b (-11.876) depicted the redness and blueness of the dyed samples, respectively. It was also interpreted from the table 4.25 that K/S value was highest (19.012) for the sample dyed for 80 minutes which indicated that dye absorption was maximum at this dyeing time. Mari *et al* (2008) also reported that by increasing the dyeing time, the K/S value increase gradually.

Table 4.27 Optimization of dyeing time for milling acid dye on the basis of CIE Lab and K/S values

Time (min)	Wavelength (nm)	L	a	b	K/S values
60	520	42.271	45.358	-10.942	10.133
70	520	50.886	50.654	-11.204	13.303
80	520	52.342	50.886	-11.876	19.012

Analysing the wash fastness results from the table 4.28 it was found that the sample dyed for 60 minutes showed good to excellent grades for colour change and negligible

staining on both wool and silk fabrics. When the samples were dyed for 70 and 80 minutes, grades were good in terms of colour change and negligible staining was found on wool fabric, whereas a slight staining was observed on silk fabric. Wash fastness grades were similar for the samples dyed for 70 and 80 minutes but 80 minutes time duration was considered optimum because the sample dyed for 80 minutes showed comparatively higher CIE Lab values. K/S value was found to be highest for the sample dyed for 80 minutes. Hence 80 minutes dyeing time was thought to be optimum for dyeing the mulberry silk waste/wool blended fabric.

Table 4.28 Optimization of dyeing time for milling acid dye on the basis of wash fastness

Time (min)	CC	C S	
		W	S
60	4/5	4/5	4/5
70	4	4/5	4
80	4	4/5	4

4.1.4.4 Optimization of dye concentration for milling acid dye

It is apparent from the table 4.29 that CIE Lab and K/S values of dyed samples increased with the increase in the dye concentration. The sample dyed at 6 percent dye concentration showed the highest L value (52.76), which means that samples dyed at this concentration were darkest among all. Observed value of a and b for 6 percent dye concentration was found to be 52.876 and -13.987 respectively, which were the indicators of redness and blueness of the dyed sample. The value of K/S at 6 percent dye concentration was observed as 49.000. K/S value is directly proportion to the dye concentration so increase in the concentration of dye bath increases the colour strength (Vassileva *et al* 2008).

Table 4.29 Optimization of dye concentration for milling acid dye on the basis of CIE Lab and K/S values

Concentration (%)	Wavelength (nm)	L	a	b	K/S values
1	520	47.310	47.397	-11.800	10.133
2	520	48.100	51.201	-10.571	10.451
3	520	48.650	51.450	-11.980	32.340
4	520	50.542	52.760	-12.110	32.340
5	520	51.870	52.876	-12.324	49.000
6	520	52.760	52.876	-13.987	49.000

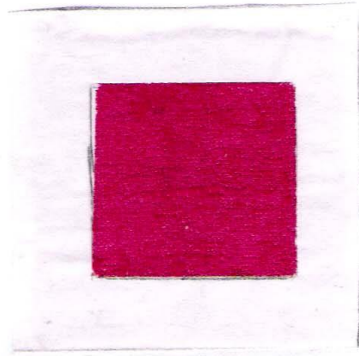
Observing at the results of wash fastness grades from the table 4.30 it was seen that the wash fastness grades for the samples dyed at 1, 2 and 3 percent dye concentrations were good to excellent for colour change and negligible staining was observed on the both wool and silk fabrics. The wash fastness grades decreased when percent dye concentration was increased 4 to 6. Wash fastness grade for the sample dyed at 4 percent dye concentration was good for colour change and slight staining was found on both wool and silk fabrics. Similar wash fastness grades were observed for the sample dye at 5 and 6 percent dye concentration except for the colour staining on wool fabric which was found slight for 5 percent dye concentration and noticeable to slight staining was observed for 6 percent dye concentration. As far as the colour change was concerned observed grades were fair to good for colour change and noticeable to slight staining was observed on silk fabric for both 5 and 6 percent dye concentration. Even though the absorption was maximum at 6 percent dye concentration, 3 percent dye concentration was optimized as optimum dye concentration as the value of L was observed to be 48.650, a value was 51.45 and b value was -11.980. The recorded value of K/S was 32.340 and this was found to be maximum among the samples dyed at 1, 2 and 3 percent dye concentration (Table 4.29).

Table 4.30 Optimization of dye concentration for milling acid dye on the basis of wash fastness

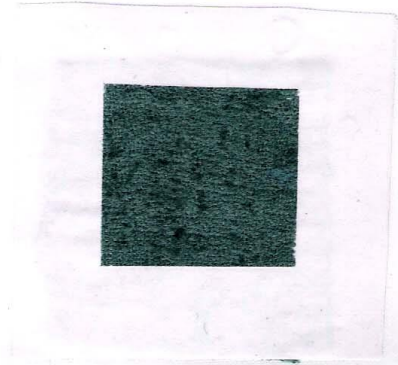
Concentration (%)	CC	C S	
		W	S
1	4/5	4/5	4/5
2	4/5	4/5	4/5
3	4/5	4/5	4/5
4	4	4	4
5	3/4	4	3/4
6	3/4	3/4	3/4

4.2 Final dyeing of mulberry silk waste/wool blended fabric

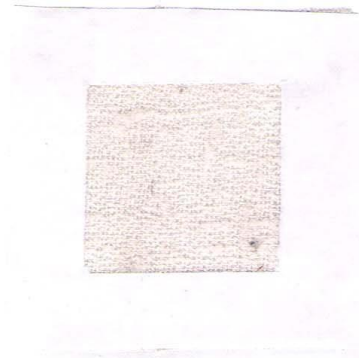
The optimized dyeing conditions were used for final dyeing of mulberry silk waste/wool blended fabric using four synthetic dyes viz. cold reactive dye, hot reactive dye, levelling acid dye and milling acid dye. The shades obtained have been displayed on sheet SS₁.



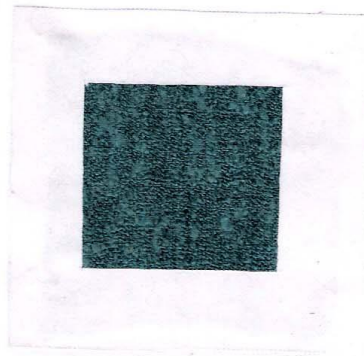
Cold reactive dye



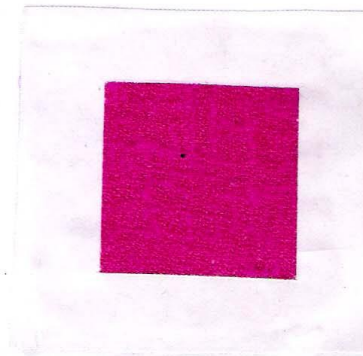
Hot reactive dye



Control



Levelling acid dye



Milling acid dye

SS 1: Undyed and dyed samples of mulberry silk waste/ wool blended fabric

4.3 Analysis of fabric properties

The undyed (control) and dyed fabric were analysed for physical and mechanical properties. The properties analysed were fabric weight, fabric thickness, bending length, fabric stiffness, drapeability, crease recovery, cover factor and tensile strength.

4.3.1 Analysis of physical properties of dyed and undyed fabric

Under the physical properties fabric weight, thickness, stiffness, flexural rigidity, drape coefficient, crease recovery and cover factor were analysed (Table 4.31 and figure 4.1-4.7).

4.3.1.1 Fabric weight (GSM)

Usually the weight of the fabric increases after dyeing as different dye molecules and the chemicals used in the dyeing process penetrate into the fabric at the time of dyeing which are trapped under the fiber polymer system thus, increase the weight of the fabric. Increase in the weight of the fabric after dyeing is also dependent on the size of the dye molecule as well as on the bond formation between dye and fiber polymer (Mulasavalagi 2005). Increase in dye concentration is also an important factor for the increase in the fabric weight. Higher the dye concentration, higher is the number of dye molecule which results in higher fabric weight (Hussain and Ali 2009).

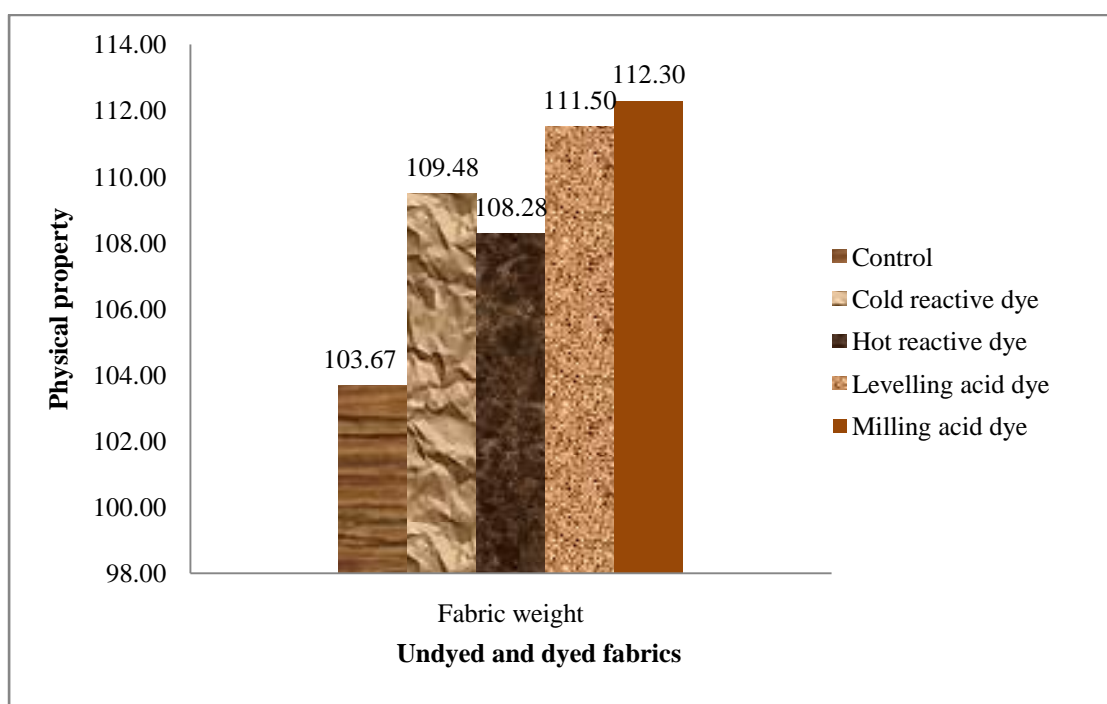


Fig.4.1: Fabric weight of undyed and dyed fabrics

It was apparent from the table 4.31 and fig.4.1 that the weight of the undyed fabric was ($103.667 \pm 0.576 \text{ g/m}^2$), which got increased significantly after dyeing. Fabric dyed with milling acid dye showed the highest mean value of fabric weight ($112.300 \pm 0.663 \text{ g/m}^2$). Milling dye molecules are larger in size which results in increase in the weight of the fabric as reported by Hunger (2003). Further, this increase in weight was followed by fabric dyed with levelling acid

dye with the mean value $111.500 \pm 0.3872 \text{ g/m}^2$. Although cold reactive dyed fabric with mean value $109.478 \pm 0.320 \text{ g/m}^2$ and hot reactive dyed fabric with mean value $108.280 \pm 0.574 \text{ g/m}^2$ also showed increase in the fabric weight but the difference between these two was not found to be significant. The increase in fabric weight was due to the structure of the reactive dye which forms the covalent bond with the fiber polymer and the polymer system of the fiber held the molecule with in itself. Cold reactive dyed fabric showed less increase in weight as compared to the hot reactive dyed fabric. This may be due to the reason that the fabric was dyed at room temperature which lead to the improper bond formation between dye molecule and fiber polymer.

4.3.1.2 Fabric thickness

It was observed that thickness of fabric increased after dyeing. Mulasavalagi (2005) reported that this may be due to deposition of dye molecules within the polymer system of the fabric. It can be attributed from table 4.31 and fig.4.2 that the thickness for undyed fabric was $0.305 \pm 0.006 \text{ mm}$. Fabric dyed using milling acid dye showed maximum thickness ($0.500 \pm 0.007 \text{ mm}$) followed by levelling acid dyed fabric with mean value $0.430 \pm 0.008 \text{ mm}$. It was observed from the table 4.31 that the difference in thickness between the fabrics dyed with these two dyes was significant. As regards the fabric dyed with reactive dyes, the thickness of the fabric was found to be minimum ($0.395 \pm 0.061 \text{ mm}$) for the fabric dyed using hot reactive dye and thickness for the fabric dyed with cold reactive dye was found to be $0.414 \pm 0.010 \text{ mm}$, but these values did not differ significantly. This may be due to the washing off many dye molecules that were being held loosely in the fiber polymer system.

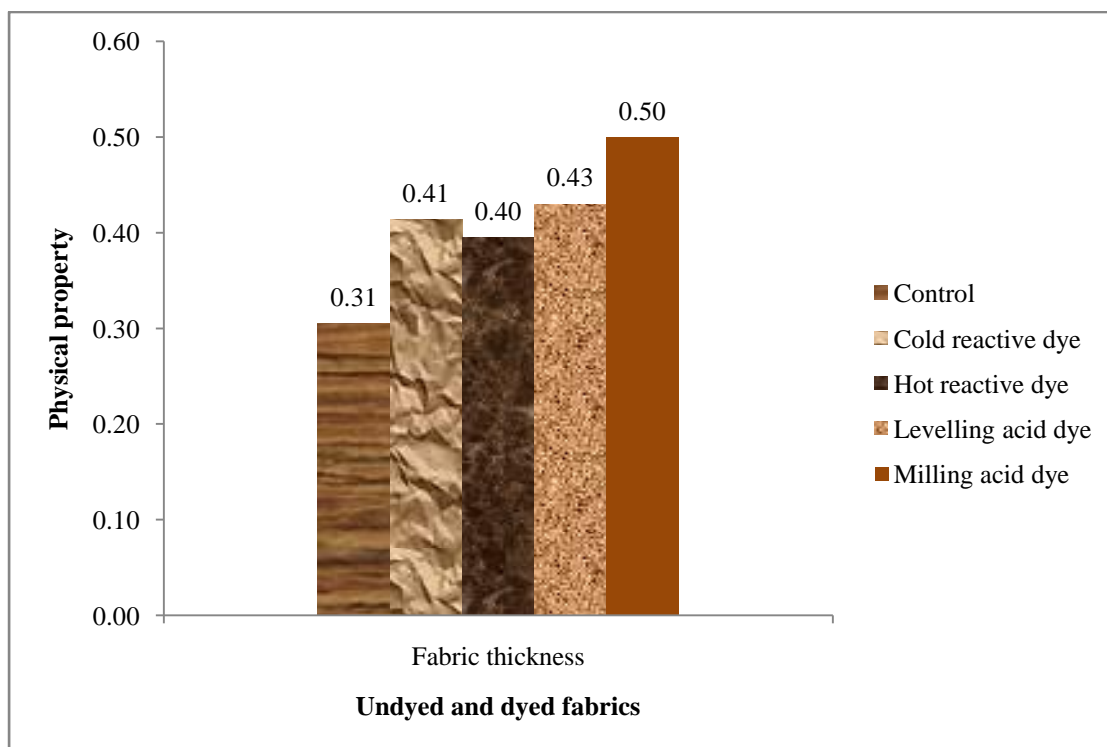


Fig.4.2: Fabric thickness of undyed and dyed fabrics

Table 4.31 Analysis of physical properties of undyed and dyed samples

Physical properties	Undyed sample	Cold reactive dyed Sample	Hot reactive dyed sample	Levelling acid dyed sample	Milling acid dyed sample	CD	CV%
Fabric weight (g/m²)	103.667±0.576	109.478 ± 0.320	108.280 ± 0.574	111.500 ± 0.3872	112.300 ± 0.663	1.536	1.076
CV%	1.244	0.661	1.171	0.776	1.320		
Fabric thickness (mm)	0.305 ± 0.006	0.414 ± 0.0105	0.395 ± 0.0618	0.430 ± 0.008	0.500 ± 0.00706	0.042	3.960
CV%	5.0281	5.716	5.440	4.348	3.160		
Bending length (cm)							
Warp	3.920 ± 0.0581	2.940 ± 0.102	2.450 ± 0.582	2.060 ± 0.0244	2.100 ± 0.509	0.189	4.110
CV%	3.599	5.304	3.639	1.850	3.851		
Weft	2.500 ± 0.196	1.300 ± 0.0316	1.540 ± 0.0399	1.020 ± 0.039	1.000 ± 0.373	0.277	2.763
CV%	5.457	3.074	3.521	3.992	4.019		
Flexural rigidity (mg/cm)							
Warp	149.676 ± 0.827	136.344 ± 0.290	134.886 ± 0.813	121.620 ± 1.99	120.624 ± 0.3725	3.103	3.578
CV%	3.724	1.940	3.375	3.570	1.231		
Weft	127.618 ± 0.375	118.580 ± 0.192	114.768 ± 0.2648	94.348 ± 0.381	96.104 ± 0.509	1.068	3.84
CV%	3.888	2.323	2.391	5.390	4.386		
Overall flexural rigidity	135.800 ± 0.094	127.350 ± 0.520	126.174 ± 0.349	107.936 ± 0.317	108.681 ± 0.769	1.380	2.86
CV%	0.646	4.982	1.620	1.650	4.820		
Drape coefficient	0.722 ± 0.005	0.956 ± 0.007	0.829 ± 0.111	0.595 ± 0.0283	0.568 ± 0.024	0.133	5.225
CV%	1.847	1.857	3.048	7.968	9.672		
Crease recovery (degree)							
Warp	145.412 ± 1.224	152.501 ± 0.999	150.000 ± 1.499	135.000 ± 1.220	130.000 ± 0.999	3.332	2.017
CV%	1.761	1.58	2.280	1.914	2.329		
Weft	140.000 ± 0.948	140.500 ± 1.999	130.050 ± 1.870	130.250 ± 1.741	125.550 ± 1.580	6.582	4.405
CV%	1.395	4.140	4.700	8.537	2.941		
Warp	26.013 ± 0.540	20.810 ± 1.324	22.544 ± 0.098	21.970 ± 0.980	24.279 ± 1.021	2.098	NS
CV%	2.876	1.678	2.547	3.609	2.510		
Weft	6.917 ± 0.870	6.194 ± 1.087	7.084 ± 0.867	6.584 ± 1.90	6.201 ± 0.809	1.654	NS
CV%	1.650	2.380	3.540	1.609	3.560		

4.3.1.3 Bending length

From the data furnished in table 4.31 and fig. 4.3 the mean bending length of undyed fabric in warp direction was perceived to be 3.920 ± 0.0581 cm. Among all the dyed fabrics, the levelling acid dyed fabric showed the minimum bending length with mean value 2.06 ± 0.024 cm. The fabric dyed with cold reactive dye showed highest value of the bending length with the mean value 2.940 ± 0.102 cm. The bending length for fabric dyed with hot reactive dye was 2.450 ± 0.582 cm, this value was found to be significantly lower than the mean bending length of the fabric dyed using cold reactive dye. The observed value of bending length for fabric dyed with milling acid dye was 2.100 ± 0.509 cm. Hence, it can be concluded that the acid dyed fabric imparted more softness and smoothness to the dyed fabric as compared to the reactive dyed fabric, the making it more pliable than the other dyed fabrics.

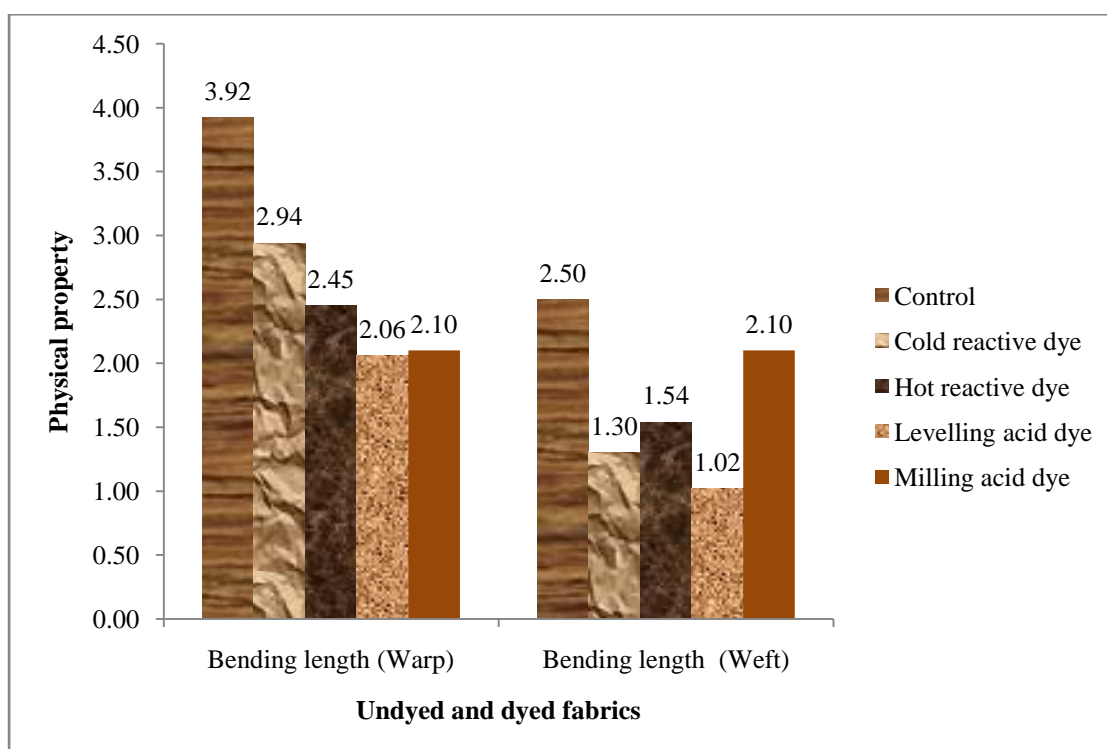


Fig.4.3: Bending length of undyed and dyed fabrics

The bending length of undyed fabric in weft direction was 2.500 ± 0.196 cm. Hot reactive dyed fabric showed highest value (1.540 ± 0.039 cm) for bending length in weft direction as compared to the fabric dyed with other three dyes. Although this value was not significantly different from the mean bending length of cold reactive dyed fabric which was found to be 1.300 ± 0.031 cm. Bending length of the fabric dyed using levelling and milling acid dye were 1.020 ± 0.039 cm and 1.000 ± 0.373 cm, respectively but the values did not differ significantly with each other. The difference between the bending length of reactive and

acid dyed fabric was found to be significant. Thus, the reactive dyed fabric showed significantly higher bending length than the acid dyed fabric (Table 4.31 and fig.4.3).

4.3.1.4 Flexural rigidity

Flexural rigidity is the measure of stiffness. Flexural rigidity in warp direction for undyed fabric was 149.676 ± 0.827 mg/cm. Flexural rigidity for cold reactive dyed fabric was 136.344 ± 0.290 mg/cm and it was observed 134.886 ± 0.813 mg/cm for the fabric dyed using hot reactive dye. These two values were not significantly different from each other. For levelling acid dyed fabric the flexural rigidity was measured 121.620 ± 1.990 mg/cm, where as for milling dyed fabric it was found to be 120.624 ± 0.3725 mg/cm. Although these two values were not significantly different from each other but flexural rigidity for acid dyed fabric were significantly lower than the flexural rigidity of the fabric dyed using reactive dye (Table 4.31 and fig.4.4).

It was observed from the data furnished in table 4.31 and fig.4.4 that undyed fabric showed highest value (127.618 ± 0.375 mg/cm) of flexural rigidity in weft direction. Observed value of flexural rigidity in weft direction for the fabric dyed with cold reactive dye was 118.58 ± 0.192 mg/cm, which was significantly higher than the mean value of the fabric dyed using hot reactive dye (114.768 ± 0.264 mg/cm). Flexural rigidity was found to be 94.348 ± 0.381 mg/cm for the fabric dyed using levelling acid dye and this value was significantly lower than the observed value of flexural rigidity for milling acid dyed fabric, which was observed as 96.104 ± 0.509 mg/cm.

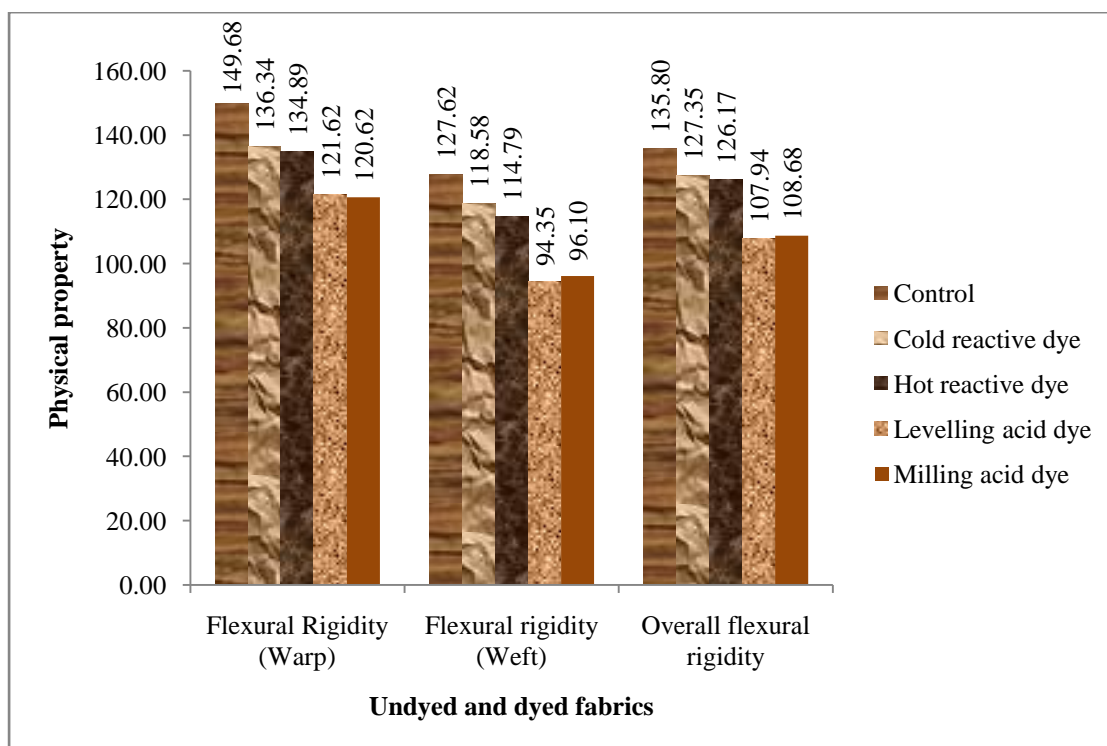


Fig. 4.4: Flexural rigidity of undyed and dyed fabrics

Maximum overall flexural rigidity observed for the undyed fabric with the mean value was 135.80 ± 0.094 mg/cm. For cold reactive dyed fabric it was measured as 127.350 ± 0.520 mg/cm. This mean value did not differ significantly from the mean overall flexural rigidity of the fabric dyed using hot reactive dye which was analysed to be 126.174 ± 0.349 mg/cm. The overall flexural rigidity for the fabric dyed using levelling acid dye and milling acid dye was 107.936 ± 0.317 mg/cm and 108.681 ± 0.769 mg/cm respectively. These values did not differ significantly, but these values showed significant difference if compared with the cold and hot reactive dyed fabric, thus indicating that the fabric dyed with reactive dyes was stiffer than the fabric dyed with acid dyes.

4.3.1.5 Drape coefficient

There is strong correlation between flexural rigidity and drape parameters. Drapability under the influence of gravity is significantly affected by bending properties. A higher flexural rigidity prevents the fabric to form folds (Behera and Pattnayak 2008). It is noticed from table 4.31 that drape coefficient of the undyed fabric was found to be (0.722 ± 0.005) . Drape coefficient was observed 0.9562 ± 0.007 for fabric dyed using cold reactive dye and 0.8298 ± 0.111 for the hot reactive dyed fabric, these two mean values were not significantly different from each other.

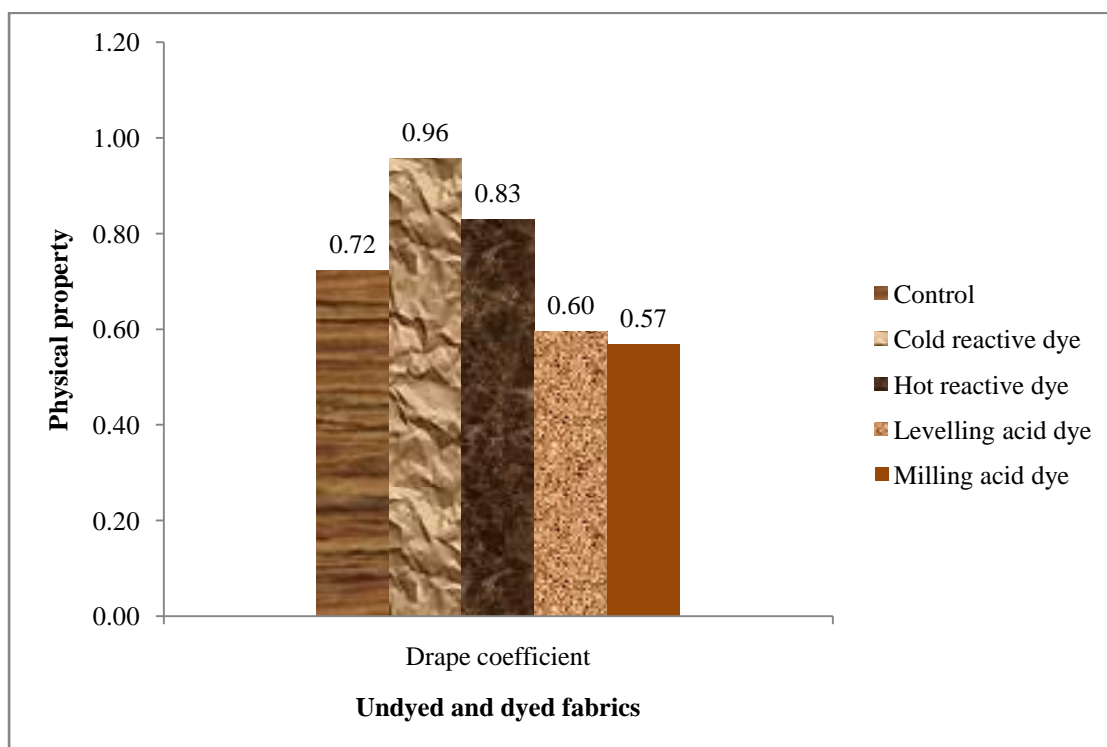


Fig. 4.5: Drape coefficient of undyed and dyed fabrics

Drape coefficient for the fabric dyed using levelling and milling acid dyes was found to be 0.5956 ± 0.0283 and 0.5688 ± 0.0245 respectively. The difference between these two values was not found significant. But the drape coefficient value of acid dyed fabric was

significantly lower than the undyed as well as with reactive dyed fabric. Comparing both reactive and acid dyed fabric it was concluded that the drape ability of acid dyed fabric was better than reactive dyed fabric. Mulasavalagi (2005) reported that cloth stiffness is inversely proportional to its drape quality therefore levelling and milling acid dyed fabric showed better drapability with the lower value of drape coefficient. Hence, increase in stiffness eventually affected the drape negatively (Table 4.31 and fig.4.5).

4.3.1.6 Crease recovery

Table 4.31 and fig.4.6 elicits the crease recovery angle of the undyed and dyed fabric. The observed crease recovery angle in warp direction for undyed fabric was 145.412 ± 1.224 . Crease recovery angle in warp direction was maximum (152.501 ± 0.999) for fabric dyed using cold reactive dye which was followed by hot reactive dyed fabric with the mean value of (150 ± 1.499). There might be cross-linking between the fiber molecules which hinder the molecular and fibrillar slippage and stabilise the structure, thereby increasing the crease recovery angle (Periyasamy *et al* 2011). Crease recovery angle of the fabric dyed using milling acid dye was found to be minimum (130 ± 0.999) which was significantly lower than the crease recovery angle of the fabric dyed with the levelling acid dye which was found to be 135.00 ± 1.220 . Difference between the crease recovery angles of cold and hot reactive was also significant. Crease recovery angle of acid dyed fabric were significantly lower than the crease recovery angle of reactive dyed fabric, which explained that fabric dyed with acid dyes were more soft and pliable. Lower the bending path, softer the fabric and lesser is the crease recovery angle (Mulasavalagi 2005).

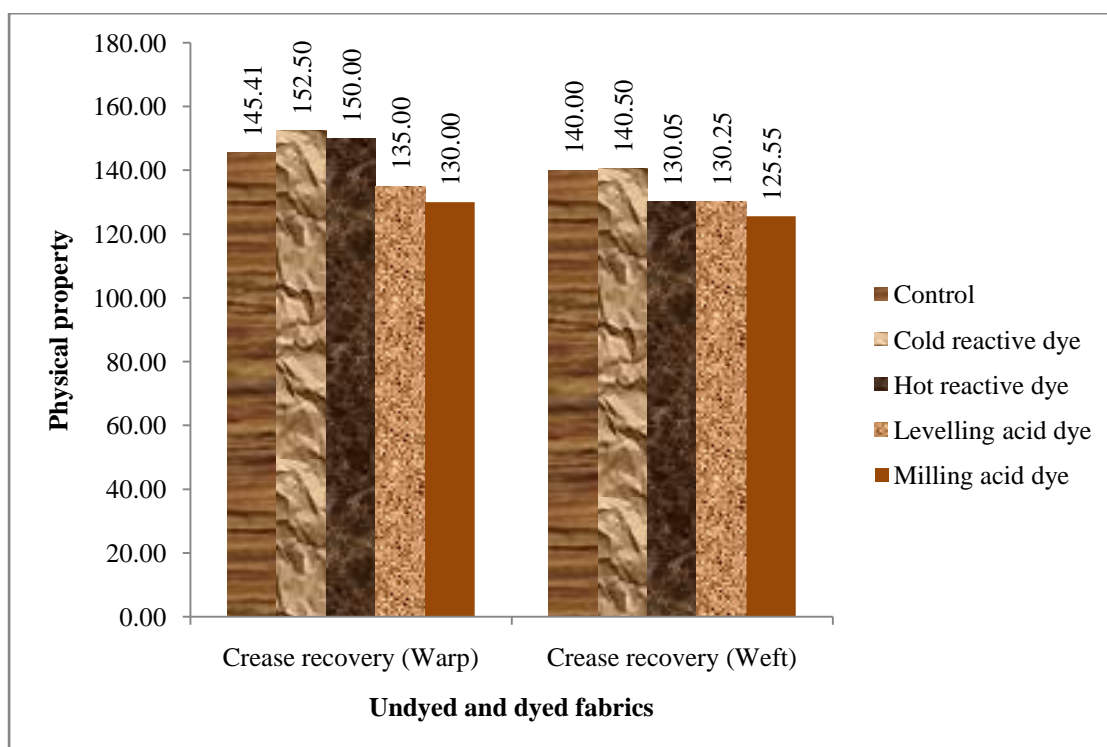


Fig. 4.6: Crease recovery of undyed and dyed fabrics

Crease recovery in weft direction was lowest (125.55 ± 1.580) for the fabric dyed with milling acid dye. The crease recovery angle of undyed fabric was (140 ± 0.948) this value did not differ significantly with the crease recovery angle (140.5 ± 1.999) of the fabric dyed with cold reactive dye. Crease recovery angle for levelling acid dyed fabric was 130.250 ± 1.741 it was not significantly different than the crease recovery angle of the hot reactive dyed fabric which was found to be 130.05 ± 1.870 , these values were significantly lower than mean crease recovery angle of undyed and cold reactive dyed fabric (Table 4.31 and fig.4.6).

4.3.1.7 Cover factor

Data presented in table 4.31 fig.4.7 revealed that cover factor for undyed fabric was found to be 26.013 ± 0.540 in warp direction. Cover factor in warp direction was found to be 20.810 ± 1.324 and 22.544 ± 0.098 for the fabric dyed using cold and hot reactive dye respectively. The observed value of cover factor in warp direction for levelling acid dye was 21.970 ± 0.980 , whereas it was found to be 24.279 ± 1.021 for milling acid dyed fabric. All these values of cover factor for dyed and undyed fabric were not significantly different from each other.

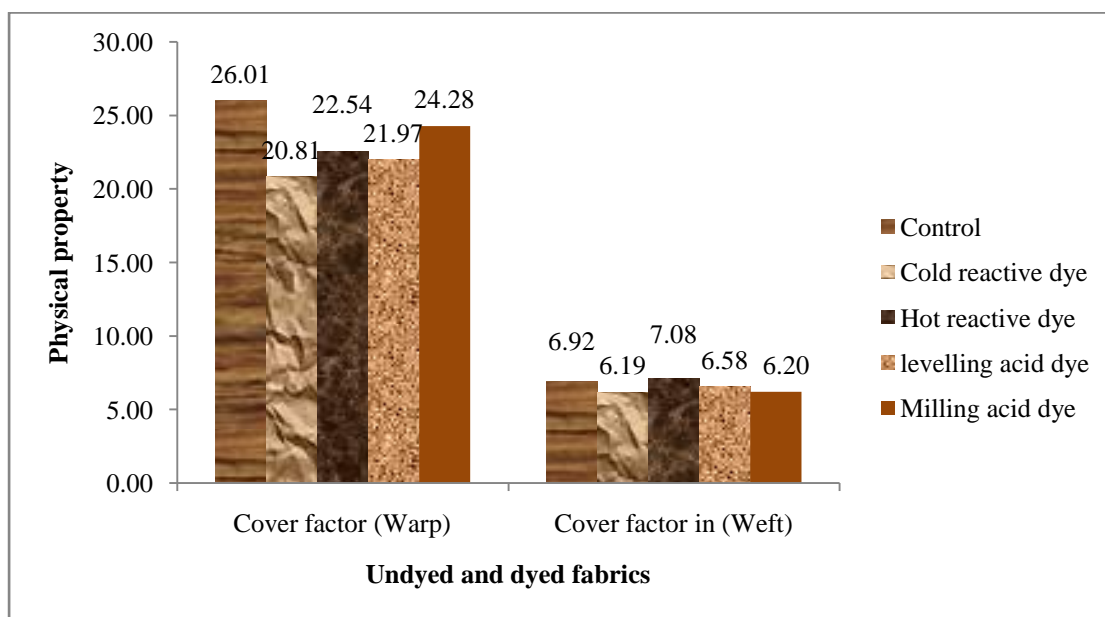


Fig. 4.7: Cover factor on undyed and dyed fabrics

As regards the value of cover factor in weft direction it was found to be 6.917 ± 0.870 for the undyed fabric. Value of cover factor was observed 6.194 ± 1.087 , 7.084 ± 0.867 , 6.584 ± 1.90 and 6.201 ± 0.809 for the fabric dyed using cold reactive, hot reactive, levelling acid and milling acid dye respectively. Following the similar trend as of warp direction, these values again did not show any significant difference.

4.3.2 Analyses of mechanical properties of undyed and dyed fabric

Tensile strength and elongation were studied for undyed and dyed fabric for evaluating the mechanical properties (Table 4.32 and figure 4.8 and 4.9).

Table 4.32 Analysis of mechanical properties of undyed and dyed samples

Mechanical properties	Undyed sample	Cold reactive dyed Sample	Hot reactive dyed sample	Levelling acid dyed sample	Milling acid dyed sample	CD	CV%
Tensile strength(kg/sq.cm)							
Warp	59.062 ± 0.986	62.388 ± 0.671	61.342 ± 0.369	53.930 ± 0.401	56.948 ± 1.245	1.398	3.156
CV%	3.734	2.527	1.466	1.587	4.881		
Weft	42.154 ± 0.559	45 ± 0.374	43.03±0.755	38.034 ± 0.067	39.933 ± 0.532	1.754	3.251
CV%	3.039	2.145	4.020	3.646	2.854		
Elongation (%)							
Warp	13.940 ± 0.050	14.933 ± 0.211	15.608 ± 0.155	16.744±0.369	17.640 ± 0.496	0.892	4.895
CV%	0.817	3.316	2.664	5.600	8.857		
Weft	20.478 ± 0.1913	18.944 ± 0.039	20.760 ± 0.916	19.345 ± 0.380	21.000 ± 0.291	1.452	6.080
CV%	2.409	3.940	2.230	5.201	3.319		

4.3.2.1 Tensile strength

Table 4.32 and fig.4.8 elicits that the tensile strength in warp direction for undyed fabric was $(59.062 \pm 0.986 \text{ kg/sq.cm})$, which was significantly lower than the mean tensile strength of the fabric dyed using cold reactive dye, which was found to be $62.388 \pm 0.671 \text{ kg/sq.cm}$. In case of fabric dyed using hot reactive dye the mean tensile strength was $61.342 \pm 0.369 \text{ kg/sq.cm}$ and this was found to be non significant with the mean tensile strength of undyed and cold reactive dyed fabric. This may be attributed to fact that reactive dyes form covalent bonds with one of the many groups in the protein fiber. However, most of the covalent bonds ocean with amino groups, since these are numerous than the other bonds which increase the single yarn strength (Gohl and Vilensky 1983). The mean tensile strength for milling and levelling acid dyed fabric was found to be $53.930 \pm 0.401 \text{ kg/sq.cm}$ and $56.948 \pm 1.245 \text{ kg/sq.cm}$ respectively and these values did not differ significantly with each other. But these mean values are significantly lower than the mean value tensile strength of undyed fabric.

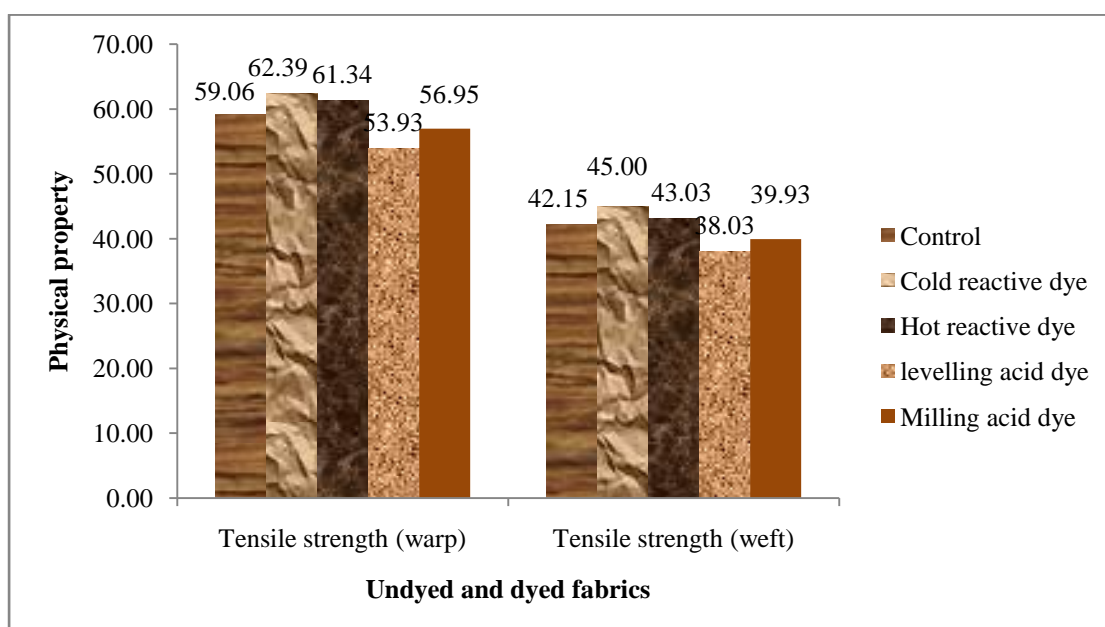


Fig. 4.8: Tensile strength on undyed and dyed fabrics

Data presented in the table 4.32 and fig.4.8 revealed that the tensile strength for undyed fabric in weft direction was $42.154 \pm 0.559 \text{ kg/sq.cm}$. The value of tensile strength for fabric dyed using cold reactive dye was $45 \pm 0.374 \text{ kg/sq.cm}$, where as the mean value of tensile strength for hot reactive dyed fabric was $43.03 \pm 0.755 \text{ kg/sq.cm}$. Increased tensile strength may be due to progressive consolidation of yarns on wet treatment that in turn enhanced the cloth count (Nagal 2006). Tensile strength for levelling acid dyed fabric observed was $38.34 \pm 0.0672 \text{ kg/sq.cm}$ and $39.933 \pm 0.532 \text{ kg/sq.cm}$ for the fabric dyed using milling acid dye, which was not significantly different than the mean tensile strength of the fabric dyed with levelling acid dye. During acid dyeing, hydrogen bonds as well as van der waals forces anchor the dye

molecular securely to polymer system which not only prevent dye stripping but also increase the strength of dyed fabric (Gohl and Vilensky 1983).

4.3.2.2 Elongation

Research findings revealed that the mean percent elongation of the undyed fabric was 13.94 ± 0.050 % and mean value of elongation was highest (17.640 ± 0.496 %) for fabric dyed using milling acid dye than the other dyed fabric. Among the all dyed fabric the elongation was found to be lowest (14.933 ± 0.211 %) for cold reactive dyed fabric. The value of the percent elongation was observed 15.608 ± 0.155 % for the fabric dyed using hot reactive dye and this value was significantly higher than the mean elongation of the fabric dyed with levelling acid for which the observed value of elongation was 16.744 ± 0.369 %.

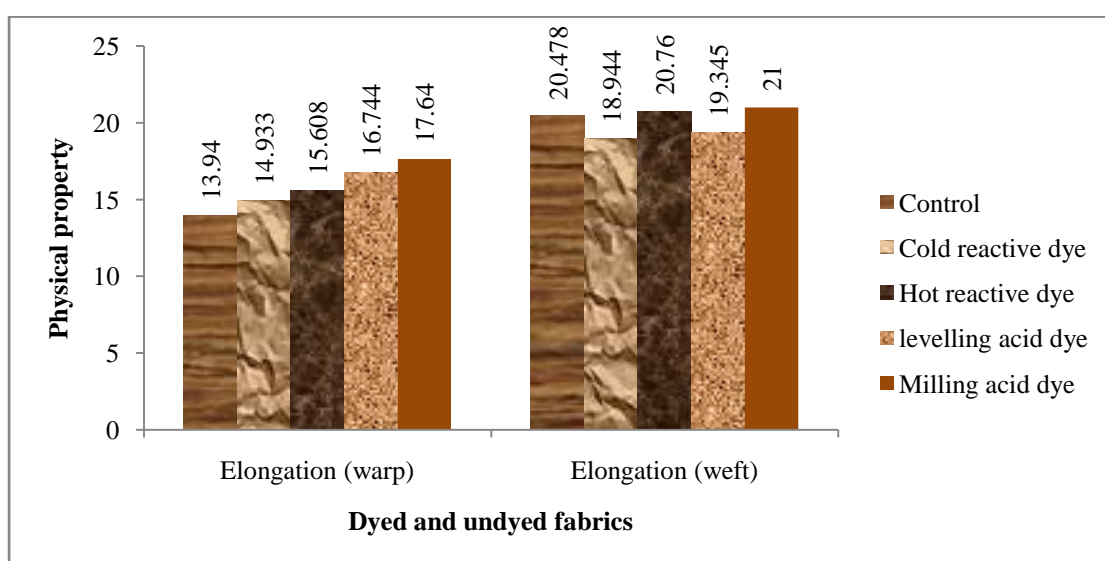


Fig. 4.9: Elongation on undyed and dyed fabrics

Elongation for undyed fabric in the weft direction was 20.478 ± 0.1913 % which decreased to 18.944 ± 0.039 % when the fabric was dyed using cold reactive dye. This was followed by the fabric dyed using levelling acid dye with mean value 19.345 ± 0.380 %. Percent elongation for milling acid dyed fabric was found to be 21.000 ± 0.291 % and this value did not differ significantly with the mean value of the hot reactive dyed fabric which was found to be 20.76 ± 0.916 % (Table 4.32 and fig.4.9).

4.4 Assessment of colour fastness properties of dyed fabric

Colourfastness is the ability of the dye to retain its colour after exposure to external agencies namely sunlight, washing, rubbing, perspiration, atmospheric conditions, or other chemical agents. The colour fastness properties of textile material depends on the type of fiber and class of dye used (Nagal 2006).

The colour fastness tests were conducted to study the effect of dyeing on fastness properties of the dyed fabric. Results of fastness properties have been furnished in table 4.33 and figure 4.10- 4.13.

Table 4.33 Colour fastness grades of dyed fabric

Fabric	Light fastness Grades	Washing fastness grades			Rubbing fastness grades				Perspiration fastness grades						
					Dry		Wet		Acidic			Alkaline			
		CC	CS		CC	CS	CC	CS	CC	CS		CC	CS		
			W	S						W	S		W	S	
Cold reactive dyed fabric	5	4	4	4	4	3/4	3	3/4	4	4/5	4	4	4	4	4
Hot reactive dyed Fabric	6	5	5	5	4/5	4	4	3/4	5	4	4	4/5	4/5	4	4
Levelling acid Dyed Fabric	6/7	4	4	4	4	4	3	3	4	3/4	3	4	4	4	3/4
Milling acid dyed Fabric	7	4/5	4/5	4/5	4/5	4/5	4	4	4/5	4	3/4	4	4	4	4

4.4.1 Colour fastness to light

There is no universal accepted explanation for the fading of dyed textile goods in sunlight. It is suggested that fading may be due to some kind of breakdown in light energy, absorption capacity of the electrons from the chromospheres or breakdown in structure of the dye molecule. Fading in light is partly due to ultraviolet radiation which initiates chemical degradation of the dye molecule through loosely held electrons of the chromospheres. Fading of dyed textile materials do not occur so readily in artificial light especially incandescent and fluorescent lights since these light sources do not emit significant quantities of ultraviolet radiation.

Data furnished in table 4.33 elicited that fabric dyed with cold reactive dye had good colour fastness to the light. Gohl and Vilensky (1983) also reported that some of cold reactive dyes show fair light fastness grades.

The fabric dyed with hot reactive dye also showed very good fastness rating and the results of the study are in line with the findings of Hussain and Ali (2009).

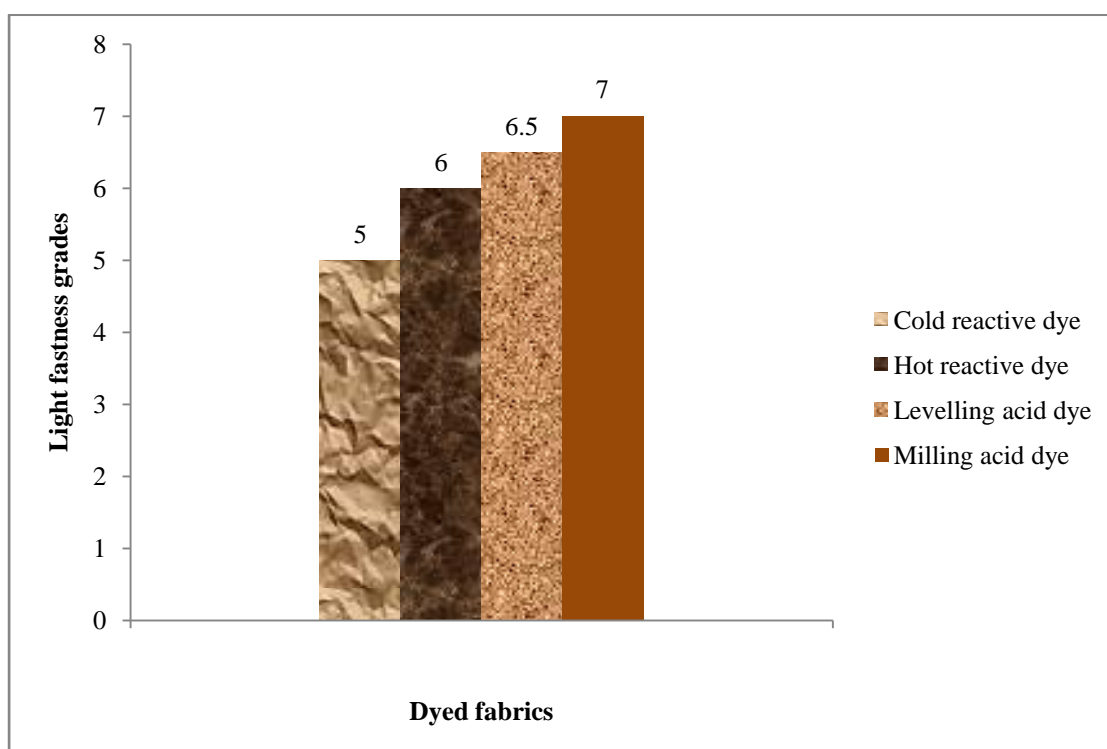


Fig 4.10: Light fastness grades of dyed fabrics

It is observed from table 4.33 and fig.4.10 that the fabric dyed using levelling acid dye had very good to excellent colour fastness to sunlight. The electron arrangement in the chromospheres of the dye is such that these dyes can resist the degrading effect of sun's UV radiation for considerable time (Gohl and Vilensky 1983).

Results for the fabric dyed with milling acid dye depicted that it possessed very excellent light fastness grade. This may be due to the electron stability of chromospheres

in the acid dyes molecules. This electron stability enables the acid dye molecule to resist the photochemical degradation of ultraviolet rays in the white beam (Nagal 2006).

4.4.2 Colour fastness to washing

The loss of colour during laundering is referred to as lack of wash fastness or ‘bleeding’. Colour loss will occur during laundering if the dyes have been used which are held loosely by the fiber, that is, dyes that have not penetrated sufficiently or dyes which are held only by weak forces such as hydrogen or van der Waals’ forces (Gohl and Vilensky 1983).

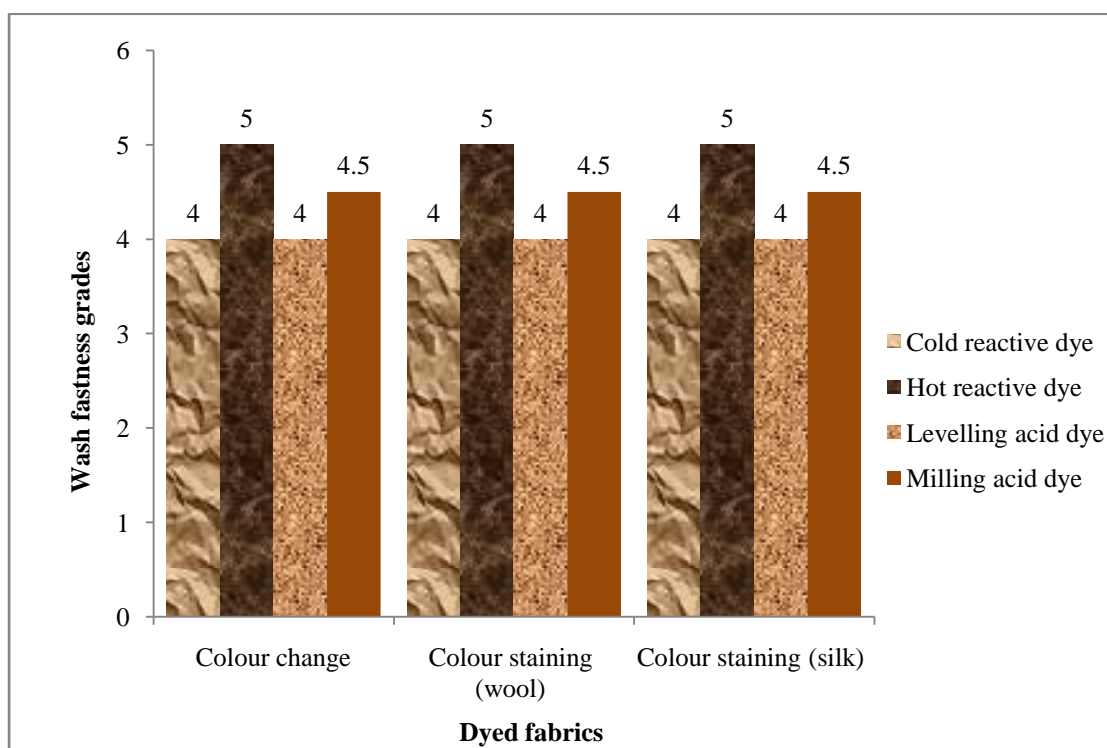


Fig 4.11: Wash fastness grades of dyed fabrics

Results furnished in table 4.33 and fig.4.11 depicted that cold reactive dyed fabric had good grade for colour change and slight staining was found on both wool and silk fabrics after washing.

The wash fastness grade for fabric dyed with hot reactive dye in terms of colour change was found to be excellent and the dyed fabric showed no staining on both wool and silk fabrics. Reactive dye attaches to the fiber by forming a covalent bond and is known for their bright colours with excellent wash fastness (Chooiling 2009).

It is concluded from the table 4.33 that colour fastness of the fabric dyed with levelling acid dye in terms of colour change was good and slight staining was observed on both silk and wool fabrics.

The fabric dyed with milling acid dye had good to excellent grade for wash fastness in terms of colour change and this dye showed negligible staining on both wool and silk fabrics. Lewis (1996) stated that these dyes are colloidal dispersions and are complex molecules that often contain hydrophobic alkyl chains that increase attraction through vander Waals forces, charge transfer and hydrophobic interaction and thus enhance wash fastness.

4.4.3 Colour fastness to rubbing

During dyeing process, some amount of dye molecules might be held superficially on the yarns beyond saturation level. It is essential to wash off the physically held dye molecules with appropriate after treatments to avoid colour bleeding in subsequent washes. In general it was noticed from table 4.33 and fig.4.12 that the colourfastness for dry rubbing was better than wet rubbing for both acid and reactive dyes.

From the table 4.33 it is evident that the fabric dyed with cold reactive dye showed good colour fastness to dry rubbing and noticeable to slight staining was observed on the adjacent fabric. This may be attributed to better penetration of the dye molecules into fiber polymer system and affinity of dyes to the fibers. The colour fastness of cold reactive dyed fabric for wet rubbing was relatively low. The colour change was rated fair and colour staining ranged between noticeable to slight. During reactive dyeing the dye molecules get hydrolyzed in presence of water that may result into poor wet fastness. Fair to poor fastness to wet rubbing may be due to superficial deposition of colour, poor dye fixation and affinity for water molecules is the limitation to wet fastness (Karmakar and Shah 1993).

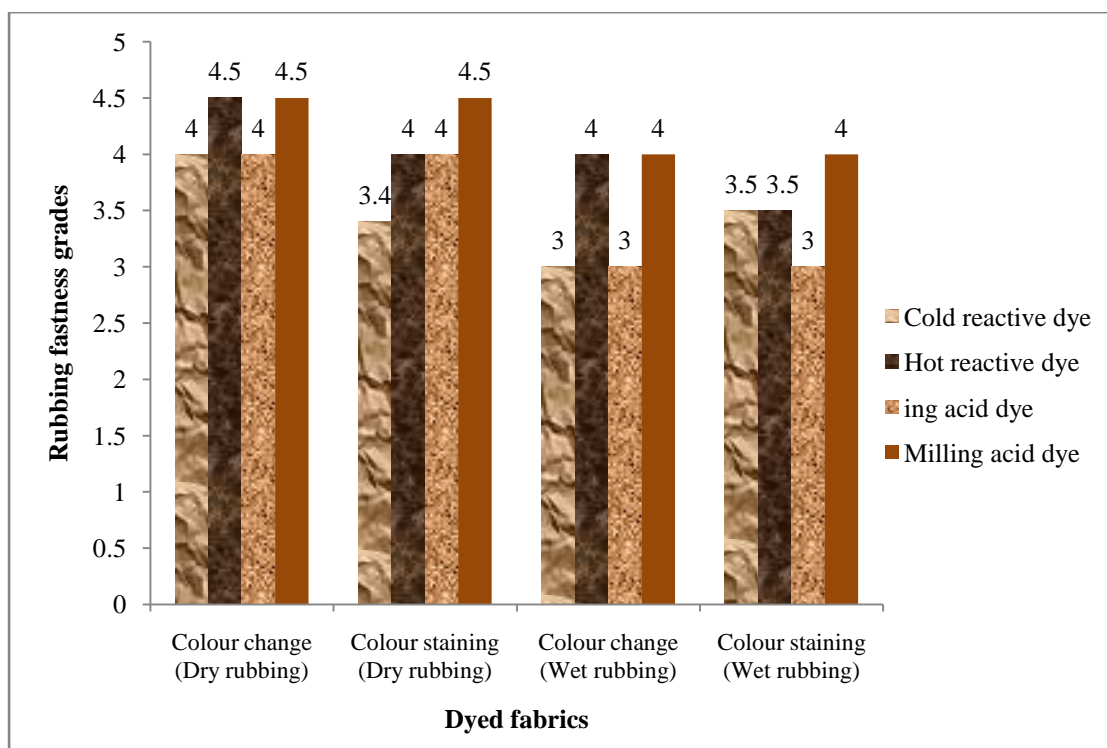


Fig 4.12: Rubbing fastness grades of dyed fabrics

The grade for colour change for fabric dyed using hot reactive dye ranged between good to excellent and slight staining was observed for dry rubbing on adjacent fabric. In case of wet rubbing good grade were observed for colour change and noticeable to slight staining was observed on the adjacent fabric. The results are in line with the findings of Uddin and Hussain (2010).

The colour fastness of levelling acid dyed fabric showed good results for colour change in dry rubbing and slight staining was found on adjacent fabric. Table 4.33 and fig.4.12 showed that levelling acid dyed fabric had fair colour fastness in terms of colour change for wet rubbing and noticeable staining was observed on the adjacent fabric.

The fabric dyed with milling dye showed good to excellent results for colour change and negligible staining on adjacent fabric for dry rubbing. But the result for wet rubbing fastness was good for colour change and slight staining on adjacent fabric. The findings of Nagal (2006) support these results.

4.4.4 Colour fastness to perspiration

Perspiration is a complex combination of body oils, fats and saline solution. It may results⁴ in loss of colour. The constituents of perspiration may react chemically and cause the chemical degradation of the dye.

Table 4.33 and fig.4.13 depicts the results of perspiration fastness of the dyed fabric. Data observed from the fig.4.13 showed that the perspiration fastness grade in terms of colour change in acidic medium for cold reactive dyed fabric was good and negligible staining was observed on wool fabric whereas slight staining was observed on silk fabric. When the medium was alkaline it showed good grade for colour change and slight staining on both wool and silk fabrics.

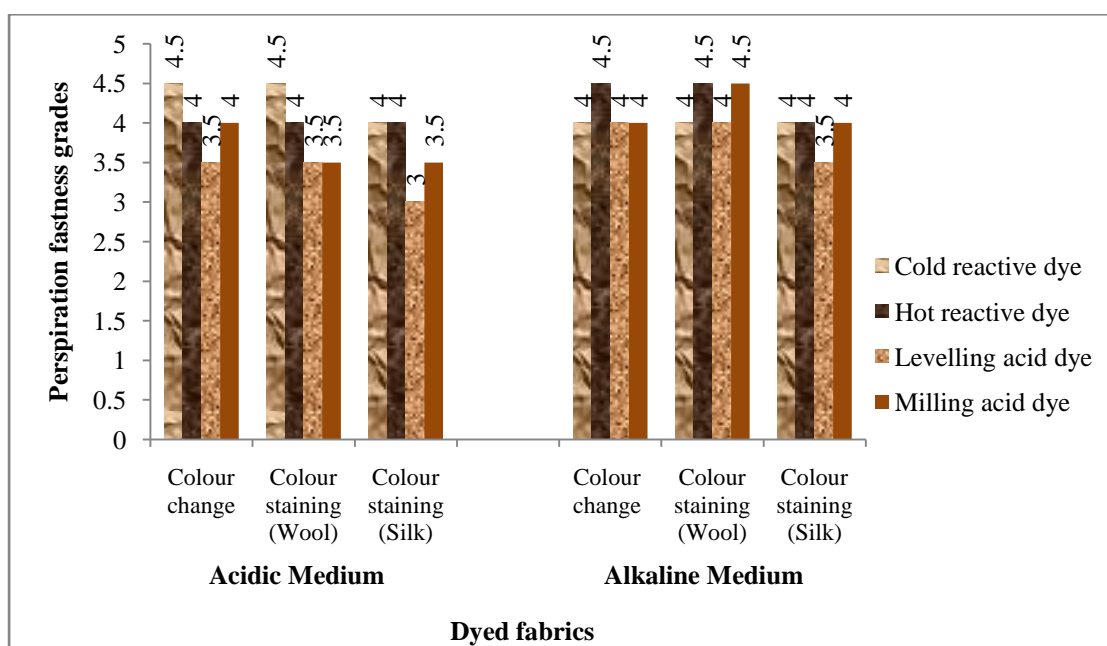


Fig 4.13: Perspiration fastness grades of dyed fabrics

The perspiration grade of hot reactive dyed fabric was excellent for colour change in acidic medium and slight staining was observed on both wool and silk fabrics. When the dyed fabric was kept in the alkaline medium the colour change ranged between good to excellent, negligible staining was found on wool fabric whereas slight staining was observed on silk fabric.

Results for perspiration fastness for the fabric dyed with levelling acid dye showed that the grade for colour change in acidic medium was good. Noticeable to slight staining was observed on wool fabric and noticeable staining was observed on silk fabric. In alkaline medium the grade observed for change in colour was good, slight staining was observed on wool fabrics and noticeable to slight staining was found on silk fabric.

Data furnished in table 4.33 and fig.4.13 showed that milling acid dyed fabric showed good to excellent grade for colour change in acidic medium whereas slight colour staining was observed on wool and noticeable to slight staining on silk fabrics in acidic medium. Good grade was observed in terms of colour change in alkaline medium and colour staining in alkaline medium was observed good for both wool and silk fabrics.

CHAPTER V

SUMMARY

Dyes are substances that can be used to impart colour to materials such as textiles, foodstuffs and paper (Chooiling 2009). A fast dye refers to a substance that does not fade when the material to which it is applied and exposed to the conditions associated with its intended use. Whereas, a dye that loses its colouring during proper usage is referred to as a fugitive dye. Some of the conditions that could cause such a change in the properties of a dye include exposure to acids, sunlight or excessive heat as well as various washing and cleaning procedures.

The most notable contribution to the science of colour and its constitution was due to Witt Whoin, who proposed that dyes contain two types of group namely chromophore and auxochrome which are responsible for their colour. Chromophore is a group of atoms principally responsible for the colour of the dye and this is capable of interacting with light and auxochromes are the 'salt forming' groups of atoms whose role is to provide an essential enhancement of the colour.

A dyestuff is a molecule which contains a chromophoric group capable of interacting with light, thus giving the impression of colour. Dyeing is a method for colouring a textile material in which a dye is applied to the substrate in a uniform manner to obtain an even shade with a performance and fastness appropriate to its final use. The process of dyeing is carried out in a variety of ways depending on the specific dye utilized as well as the properties of the substrate. Textile dyeing involves the use of a number of different chemicals and auxiliaries to assist the dyeing process. Some of them are process-specific, while others are also used in other operations.

Effect of dyeing varies according to the type of fiber. Similarly a dye responds differently on blended fabric. This is because different fiber has different affinity for the dye and a different fiber absorbs dyes at different rate (Bae *et al* 2006).

Natural silk is blended in an equal proportion with wool to make high class apparel goods, contributing lustre and strength; silk yarn is also used in wool piece goods as effect threads. Wool is strong, resilient and warm, absorbing 50% of its weight in moisture before becoming saturated and is wrinkle resistant. It is easy to spin and dye. Wool and silk blended fabric improves its physical properties from their union with wool becoming smoother, shinier, better drape and easier to wash without fear of felting, while silk acquires resiliency and warmth. The resulting yarn has a firm twist and easy to weave. Wool/silk blends are mainly used for apparel in woven or knitted form to give them a luxury character. A large variety of blend ratio can be found ranging from 5 to 50% of silk and wool. The wool/silk blend unites the best qualities of these natural fibers, creating a truly unique textile. The wool gives soothing warmth and breathability, and it is also super-absorptive & temperature-

regulating. The silk adds even more softness, plus durability, as well as its own temperature regulating properties.

Both silk and wool are protein fiber due to the presence of many amines, carboxylic acid, amide, and other polar groups, so they possess similar chemical properties. Silk and wool blends are dyed in piece form or as yarn on packages and hanks. Silk is chemically related to wool because amino groups are integral components of both fiber, thus both can be dyed with the same dyes. Silk and wool are hydrophilic in nature, wetted by water and are dyed with either acid or basic dyes through the formation of ionic bonds (salt linkages). They may also be dyed with reactive dyes that form covalent bonds with available amino groups. Dyeing system offers the advantages of a wide range of colours, including brilliant shades, one-bath dyeing method and excellent solid shade (Choudhury 2006).

Silk and wool have a slightly cationic character with an isoelectric point at about pH 5.0. Thus, in an acidic solution, silk fibroin or wool takes on a positive charge through absorption of hydrogen ions. The resulting electric charge can be counter balanced with negatively charged anions of the dye, whilst dyeing with anionic dyes such as acid, metal complex, direct and reactive dye. The quality and type of silk and wool fiber affect the distribution and shade depth on the fiber (Oswal 2010).

Wool dyed with acid dyes has low wash fastness because of the weak, secondary forces between the fiber and the dye molecule. The amount of covalently fixed dye increases when after treating the dyed wool samples with salts (Trezl *et al* 1998).

Silk has an affinity for acid dyes, but the colour tends to be less fast than on wool. Silk will exert its affinity for acid dyes at lower temperature than in the case of wool, and dyeing is usually commenced at 40°C and the temperature is not allowed to rise above 80°C (Tortman 1994).

Studies suggested that silk and wool both are protein fiber can be dyed successfully with the reactive and acid dyes. Less work has been done regarding the dyeing of silk/wool blend with synthetic dyes. An attempt in the present study has made to dye the mulberry silk waste/wool blended fabric with reactive and acid dyes with following objectives:-

- To optimize the dyeing conditions for dyeing silk/wool blended fabric with acid and chrome dyes.
- To compare the selected physical and mechanical properties of the undyed and dyed silk/wool blended fabric.
- To compare the dye absorption by the silk/wool blended fabric with selected dyes.
- To evaluate the colour fastness properties of dyed silk/wool blended fabric.

To carry out the investigation hand woven blend of mulberry silk waste/wool in the ratio of 65:35 was used for the research work. The mulberry silk waste was procured from

Srinagar (Jammu and Kashmir) and wool fiber was procured from the local market of the Ludhiana city. Blending and spinning was done at Kullu (Himanchal Pradesh) and weaving was done in Ludhiana.

Two different categories of reactive and acid dyes were selected to study the effect of dyeing on mulberry silk waste/wool blended fabric. While selecting dyes cold reactive and hot reactive dyes were selected for reactive dyeing whereas levelling acid and milling acid dyes were selected for acid dyeing. Dyeing parameters optimized for each dye included dyeing pH, dyeing temperature, dyeing time and dye concentration. The conditions were optimized on the basis CLE Lab, K/S values and wash fastness grades of the dyed samples.

Later the physical and mechanical properties of the undyed fabric was tested and compared with dyed fabric. Physical properties studied included fabric weight, fabric thickness, bending length, flexural rigidity, drape coefficient, crease recovery and cover factor. For mechanical properties tensile strength and elongation were studied. The colour fastness properties for all the sets of dyed fabric were evaluated.

Results regarding the optimum dyeing conditions for cold reactive, hot reactive, levelling acid and milling acid dyes are illustrated in the table 5.1. It could be concluded from the table that dyeing at pH 7 for 60 minutes at room temperature using 4 percent dye concentration was optimum for cold reactive dye. Whereas 5 pH for 70 minutes at 90°C for 3 percent dye concentration was considered optimum for dyeing the fabric with hot reactive dye. In case of levelling acid dye optimum dyeing conditions were, pH 5 for 80 minutes at 90°C with 4 percent dye concentration. In order to optimize the dyeing condition for milling acid dye best results were obtained at 7 pH with dyeing time 80 minutes, temperature 90°C and 4 percent dye concentration.

Table 5.1 optimum dyeing condition for dyeing mulberry silk waste/wool blended fabric

Dye	Dyeing pH	Dyeing time (min)	Dyeing Temperature (°C)	Dye Concentration (%)
Cold reactive dye	7	60	Room temperature	4
Hot reactive dye	5	70	90	3
Levelling acid dye	5	80	90	4
Milling acid dye	7	80	90	3

After dyeing the mulberry silk waste/wool blended fabric was dyed with cold reactive, hot reactive, levelling acid and milling acid dyes the dyed fabric was evaluated for physical and mechanical properties. Bending length was found 3.920 ± 0.0581 cm in warp direction and 2.500 ± 0.196 cm in weft direction whereas the observed value of overall flexural rigidity was 135.800 ± 0.094 mg/cm for the undyed fabric, all these values were found to highest.

Mean crease recovery angle was found 152.501 ± 0.999 and 140.500 ± 1.999 in warp and weft direction respectively for cold reactive dyed fabric which was found to be higher as compared to the other dyed fabric. Tensile strength of the fabric dyed using cold reactive observed was 62.388 ± 0.671 kg/sq.cm and 45 ± 0.374 kg/sq.cm in warp and weft direction, respectively which were found to be the highest. Fabric dyed using milling acid dyes showed highest thickness (0.500 ± 0.00706) and drapability with lowest (0.568 ± 0.024) drape coefficient. Value of GSM was found to highest for levelling acid dyed fabric with mean value 111.500 ± 0.3872 gm. Percent elongation was found highest for the levelling acid dyed fabric with mean value 17.640 ± 0.496 % and 21.000 ± 0.291 % in warp and weft direction respectively.

The fabric dyed with cold reactive dye showed good colour fastness grade to the light and hot dyed fabric showed very good colour fastness whereas very good to excellent light fastness grade was observed for the fabric dyed using levelling acid dye. Milling acid dyed fabric showed excellent colour fastness to sunlight. Cold reactive dyed fabric had good grade as regards colour change and slight staining was found on both wool and silk fabrics after washing. The wash fastness grade for fabric dyed with hot reactive dye in terms of colour change was found to be excellent and the dyed fabric showed no staining on both wool and silk fabrics. Colour fastness of the fabric dyed with levelling acid dye in terms of colour change was good and slight staining was observed on both silk and wool fabrics. The fabric dyed with milling acid dye had good to excellent grade for wash fastness in terms of colour change and this dye showed negligible staining on both wool and silk fabrics. Cold reactive dyed fabric showed good colour fastness to dry rubbing and noticeable to slight staining was observed on the adjacent staining fabric. The colour change was rated fair and colour staining ranged between noticeable to slight. The grade for colour change for fabric dyed using hot reactive ranged between good to excellent and slight staining was observed for dry rubbing on adjacent fabric. In case of wet rubbing good grade were observed for colour change and noticeable to slight staining was observed on the adjacent fabric. The colour fastness of levelling acid dyed fabric showed good results for colour change in dry rubbing and slight staining was found on adjacent fabric. Levelling acid dyed fabric showed fair colour fastness in terms of colour change for wet rubbing and noticeable staining was observed on the adjacent fabric. The fabric dyed with milling dye showed good to excellent results for colour change and negligible staining on adjacent fabric for dry rubbing. But the result for wet rubbing fastness was good for colour change and slight staining was observed on adjacent fabric. The perspiration fastness grade in terms of colour change in acidic medium for cold reactive dyed fabric was good and negligible staining was observed on wool fabric whereas slight staining was observed on silk fabric. When the medium was alkaline it showed good grade for colour change and slight staining was found on both wool and silk

fabrics. The perspiration grade of hot reactive dyed fabric was excellent for colour change in acidic medium and slight staining was observed on both wool and silk fabrics. When the dyed fabric was kept in the alkaline medium the colour change was ranged between good to excellent, negligible staining was found on wool fabric and slight staining was observed on silk fabric. Perspiration fastness for the fabric dyed with levelling acid dye showed that the grade for colour change in acidic medium was good. Noticeable to slight staining was observed on wool fabric and noticeable staining was observed on silk fabric. In alkaline medium the grade observed for change colour was good, slight staining was observed on wool fabrics and noticeable to slight staining was found on silk fabric. The milling acid dyed fabric showed good to excellent grade for colour change in acidic medium whereas slight colour staining was observed on wool and noticeable to slight staining on silk fabrics in acidic medium. Good grade was observed in terms of colour change in alkali medium and colour staining in alkaline medium was observed good for both wool and silk fabrics.

On the basis of the present study it can be concluded that the mulberry silk waste/wool blended fabric can be dyed at slightly acidic or neutral medium for 60-80 minutes at 90°C except cold reactive dyes as they were dyed at room temperature. The study revealed that the physical and mechanical properties like crease recovery angle and tensile strength increased after dyeing the fabric with reactive dye. Whereas fabric dyed using acid dyes showed better thickness and drapability however dyeing had no effect on cover factor. Fabric dyed using reactive dyes exhibited better fastness properties as compared to fabric dyed using the acid dyes.

The study has following limitations:-

- The study was limited to the use of mulberry silk waste and wool fiber.
- The study was limited to the use of plain weave.
- The study was limited to the use of one acid and one reactive dyes only as chrome dyes are now banned.
- The study was limited to the only two shades of each dye.

In the present investigation an attempt was made to give a clear picture about the dyeing of mulberry silk waste/wool blended fabric with reactive and acid dyes and its impact on fabric properties. From the results of the present study it can be concluded that the mulberry silk waste/wool blended fabric can be dyed easily and effectively. The value addition of mulberry silk waste/wool blended fabric through dyeing, can lead to its diversified uses in the apparel and home textiles. This study will be helpful for the traditional dyers in improving the dye uptake and colour fastness quality of traditional textiles which will enhance their domestic and export marketability and consequently preserve the textile heritage of India. It will also help dyers in diversifying their product range.

The future studies can be taken up in the following fields:-

- A similar study can be conducted on the similar blend with different weave effect such as taffeta, bengaline and denim.
- Pure mulberry silk fiber can be used in blending with wool rather than waste it would give better dyeing result.
- Different silk/wool ratio can be used.
- Another class of synthetic dyes can be used for dyeing mulberry silk waste/wool blended.
- Use of natural dyes instead of synthetic dyes could be proved as a revolutionary step towards the green fashion.
- A product line can also be developed according to the suitability of the fabric.

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