PLASMA TREATMENT ON COTTON AND SILK FABRICS AND THEIR EFFECT ON DYEABLITY WITH NATURAL DYE

A PROJECT REPORT

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BONAFIDE CERTIFICATE

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ABSTRACT

Plasma treatment is gaining popularity in the textile industry due to their numerous advantages over conventional wet processing techniques. Cotton and silk offers a combination of cost, processability and performance. The natural dyes have less affinity towards the cotton and silk fabrics. In this study, cotton and silk fabrics are treated with air and oxygen plasmas and dyed with natural dyes and the treated sample are evaluated for changes in physical and functional characteristics in terms of absorbency, wickability, surface energy, moisture content, tensile strength and Colour strength.

Plasma treatment caused physical and chemical modifications in the substrate. The changes in the morphology caused chemical changes in the form of implantation of functional groups in addition to physical changes helps in obtaining mush improved moisture content, wettability and dyeability with natural dyes. The improvement has been confirmed by the absorbency test, wicking test. Among the gas plasmas studied, oxygen plasma is more effective in getting the desirable functional properties in case of cotton. While in case of silk, air plasma is more effective in getting the desirable properties.

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CHAPTER 1

INTRODUCTION

1.1 Plasma Technology

In various processes involved in the manufacturing of textile material of textile materials, chemical processing often remain as the centre of focus on account of various advantages and problems created in the processes. As the result, many unconventional methods and techniques are tried very often to provide an eco-friendly alternative method. Water-free processing, solvents assisted processing and low wet pick-up processes have been tried to reduce the pollution load created in the wet processing. The plasma treatments can be carried out on thin films, solids, natural fibers, man-made fibers and fabrics. Plasma processing methods have been explored in improving spinnability of cotton yarn, desizing and scouring of cotton and man-made fibers, dyeing and dyeing and printing of cotton and polyester fibers and also in imparting various functional finishes to the textile materials. In the medical front, plasma technique appears to be very much promising in obtaining bio-compatible surfaces and also in sterilization process.

1.2 Objectives of the Project

- To modify the surface structure of cotton & silk fabric by plasma treatment
- To study for Characterization of the fabrics for Surface morphology, Moisture absorption properties and Physical characteristics.
- To study the dyeing characteristics of treated fabrics with natural dyes.

CHAPTHER 2

LITERATURE REVIEW

2.1 Plasma – The fourth state of matter

Plasma is considered as the fourth state of matter, after solid, liquid and water, contains ionized gas comprising of electrons, atoms and molecules (jahala,2005;subbulakshmi,1988). A gas requires 1-30 eV per particle to change its state to plasma, which is higher than that required for solid-liquid or liquid-gaseous transitions. In the gaseous substance, if collisions between particles of matter caused by very high temperature increase then the initial gaseous state comprising of neutral molecules or atoms, develops into an ionized state which an equal density of positive and negative charged particles. Presence of free electrons make plasma electrically conducting, respond to electric and magnetic fields and can be an efficient sources of radiation. The plasma modifies the surface of the fabric by the bombardment with high energy electrons and ions.

2.2 Plasma treatment as a means to save water, materials and energy

This environmental aspect is without doubt the most exposed advantage of plasma technology. It wins in importance everyday due to ever increasing environment-related costs. However, its relevance depends on above mentioned cases. The largest advantage will be experienced when a plasma treatment can replace an existing wet process completely. Even though such cases are not common, and in view of a continuing cost increase for both water extraction and discharge, plasma technology becomes relatively more cost-effective every day.

Other affiliated cost savers are: Reduction of the amount of chemicals needed in wet treatment following the plasma treatment; better exhaustion of chemicals from the bath; reduced BOD/COD of discharged processing water; Shortening of the wet processing time; this compensates for the possible extra time required for the plasma treatment. Reduction in temperature needed for wet processing; saving heating energy. This adds to the efficient use of energy during the plasma treatment. Extra advantages can be that the finished textile shows better performance and improved fastness properties, ie has an extra added value.

2.3 Plasma treatment as a means to create unique textile properties

Though it is not currently produced in large amounts (square metres), this type of high-performance textiles will certainly grow in economic importance. Due to their high added value even small textile batches can be produced at high profit, though perfect process control is absolutely necessary. Typically, textiles for medical applications or in the sector of biotechnology are expected to increase in importance. Applications are special selective filtrations, biocompatibility, growing of biological tissues, etc. Especially in this case, high investment costs have a fast pay-off

2.4 Types of plasmas

The types of gaseous matters used in the plasma processes appear to vary depending upon the effects targeted. Reactive gases, inert gases, water vapour and combination of these are used on all types of textile materials depending upon the applications (Bhat, 2003; Kutlu, 2004). The combination of the gaseous are used to achieve better process performance and flexibility in the processing methods. Various types of plasmas used in the alteration of the properties of textile materials are listed I the following Table 1.1. Plasma surface treatment can be effected by either achieve or passive plasma exposure (Roth 2001).

Table 2.1 Types and characteristics of plasma

Types of Plasma	Characteristics
Glow Discharge, Formed by applying direct current (DC0, microwave (50Hz), RF (40KHz, 13.56 MHz) over a pair or series of electrodes.	Produced at low pressure, Offers highest possible uniformity and flexibility.
Vacuum Glow Discharge, Microwave (GHz) power supply	Produced at low pressure, Offers highest possible uniformity and flexibility.
OAUGDP*, RF frequency	Uniform active species, high average concentration
Corona Discharge, Formed at atmospheric pressure Using low frequency or pulsed high voltage over an electrode pair	Corona consist of small lightening type discharge Non-homogeneous, high local energy levels.
Dielectric Barrier Discharge, Filamentary, Formed by applying a pulsed Voltage Over an electrode pair with one covered by a dielectric material.	Lightening type Discharges are created provides uniform treatment

*- OAUGDP – One Atmospheric Unit Glow Discharge Plasma

2.5 Characteristics of plasma

The classification systems of plasma dwell on temperature, pressure, type of current used, type of gas used (Semyra Borucki 2001)

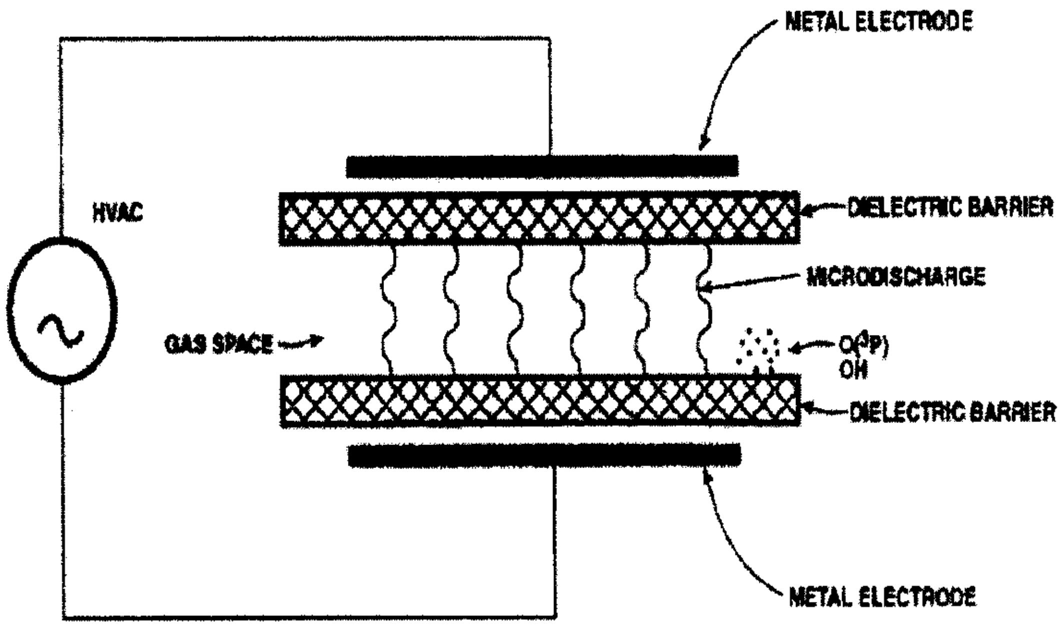


Fig.2.1 Plasma Chemical Reactivity

The two board categories, on the basis of temperature, of plasma include hot plasma and cold plasmas. In plasma, the electron temperature may reach 10^5 K whereas the plasma temperature is only about 10^2 - 10^3 K. The temperature of electrons in the plasma can reach as high as eV (1 eV = 11604 K) trough the temperature of plasma is below 40°C.

In hot plasma, which is also known as equilibrium plasma , appreciable ionization takes place, the ions and electrons are in thermal equilibrium and are operated at high pressure or atmospheric pressures. The applications of hot plasmas include deposition and spraying, welding and cutting, synthesis of ultra-fine powders, decomposition of toxic gases, liquid and solid wastes. The cold plasmas are often operated in low pressure conditions. Cold plasmas are further categorized into glow discharge, corona discharge, RF discharge plasma and are also known technological Plasmas. When the surface energy is less than 30-40 dynes/cm, the surface becomes relatively unwettable and water will bead upon its surface, above 60 dynes/cm, the surface becomes wettable and water drop spreads over a large area with a contact angle below 10°. Alteration of surface energy of material results the changes in wettability, wickability printability etc.\

Table 2.2 Plasma application and their effects on substrates

Nature of Process	Effects Obtained
Alteration of Surface Energy, Alters chemical nature of surface by active species, embedding or removing of changes	Wettability, wickability, printability, dyeability, washability, functional applications.
Alteration of Cohesive Properties, increases surface-to-surface contact cohesion, increases 3D cross linkages among fibers.	Crosslinking of polymers, washability, handle modification.
Alteration of Adhesive Properties, Adhesion, and Surface energy is increased by active species plasma. Adhesion results from combination of mechanical, chemical, micro-profile contributions.	Painting of surfaces without Volatile organic chemicals, composite structures, and medical applications.
Alteration of Electrical Characteristics, increases surface conductivity embeds or imparts electrostatic changes and,	Antistatic finish, charging and discharging action in photocopying, filtration, breathing masks, charge

deposits on the surface.	embedding in nonwovens.
Alteration of Surface Finish, Results in microscopic physical damage, removal of adsorbed monolayers, inducing chemical reactions.	Adhesion of liquids/ adhesives, etching, scratch resistance, altering optical characteristics.
Altering Bulk Properties, Changes occur due to change in surface energy and / or cohesive properties in microscopic scale.	Modification of tensile and compressive strength, elasticity, density, hand.
Removal of microorganism, Environmental stresses, physical/ chemical disruptions.	Sterilization, disinfection, cleaning and antisepsis

2.6 Role of textile structure and some process conditions

The structure of fibers, structure and construction of yarn and the fabrics play major role in deciding the efficiency of plasma processing.

Table 2.3 Parameters of glow discharge processes

Parameter	Variations	Main effect
Apparatus parameters		
Reactor type Frequency Electrodes - Placement - Coupling - Shape - Surface area ratio	Tube or bell jar 0 (DC) -10^{10} Hz Internal or external Capactively or inductively	 Energy density of plasma Internal electrodes affect chem. Composition of plasma Homogeneity of plasma

Pumping - Base pressure - Capacity Magnetic field	10^{-10} - 10^{8} mbar 2-250 m^3 /h	Only important for internal electrodes Cleanness of system Residence time Confinement and homogeneity of plasma
Discharge parameters		
Gas Substrate Flow Pressure Power Biasing Substrate temperature	 0-1000cm3/min 10^{-2} – 10 mbar 1-1000W Self bias – 500V 77-500K	Type of process Etching or deposition Residence time in plasma Etching/deposition rate Energy density of plasma Energy density of plasma Etching rate Surface composition
Procedure Parameters		
Cleaning of reactors Evacuation time Treatment time Quenching gas	Chemical, thermal or Plasma Cleaning 1 s-several hours 0.01 s-several hours	Cleanness of system Intensity of modification Surface chemistry

The presence of impurities in the fibers, additives present in the yarn and fabrics also intervene during the processing. The mean free path of gas particles, typical distances in the fabric structure like inter-fiber distance and inter-yarn distances have to be considered in the plasma processing (poll,2001;Rashidi,2004). The ion bombardment action on fiber can reveal the detail of surface and internal structures of fibers and make the molecules of surface layers activated. The thickness of fabrics, generally, range up to several millimeters depending upon the applications and to ensure plasma effect o the access in all the regions in an acceptable time, with retaining modifying ability. The applied pressure value must be matched to the characteristic structure of the textile material to be plasma exceeds the typical distances in textile materials and the very low pressure causes a relatively low radical concentration per unit volume.

In the pressure range P >100 mbar especially at atmospheric pressure the mean free path in the gas phase is much lower than textile distances. Most of the collisions happens with other gas particles and reduce the life time of the radicals. The particles do not reach the reaction sites inside the voluminous fabrics. A process pressure between 1 and 100 mbar turns out to be optimum value for plasma modification of textile materials out the thickness.

2.7 Applications Areas of Plasma in Textile Industry

- Plasma treatment is used as a step in the total textile production cycle.

  Examples are proved wetting, adhesion properties, dyeing, coating, value addition etc.
- Plasma treatment is used as a means to say water, materials and energy.
- Plasma treatment has the following advantages over wet processing:
  Plasma processing requires no water; the treatment is done in the gas phase, only a small amount of chemicals is needed, there is virtually no waste production, the treatment is confined to the fiber surface, plasma processing is very energy efficient, Some special textile properties can only be obtained via plasma processing.

2.8 Plasma processing for Yarn manufacturing

Attempts have been made, earlier, to assess the effect of plasma processing of fibres and the subsequent influence in yarn manufacturing process in the short stable spinning system using cotton fibres. The spinnability, strength, twist requirement for required strength, end breaks in spinning and weaving preparation have been analyzed as the measure of process efficiency (Abbot, 1977). Corona treatment of (CO2) cotton card sliver, using multiple ends with 450 W powders, shows increased yarn tenacity (linear density-21 tax) from 116 to 142 mN/tex (CSP – 1970 to 2330) with no significant difference in elongation at break and unevenness.

2.9 Plasma processing in fabric preparation

Plasma processing has been attempted in desizing of PVA sized viscose rayon, starch sized cotton yarns, scouring of cotton and wool fiber using cold plasma (cai, 2003; Cai 2002 ;Sun, 2004). Current method of removal of hydrophobic impurities, starch involves the use of surfactants, alkalis and oxidants at high temperatures, which result in very high effluent loads. Rot steeped 100 % cotton fabrics shows a weight loss of about 6.0% after 24 hours while the loss in the case of plasma treated fabric increases with time of treatment and the power applied. At higher power level (15 W); the weight loss reached the maximum within 5 minutes. This happens mainly due to ablation of surface, the wetting time to less hydrophobic substances. The subsequent washing reduces the wetting time to less than 1 second. PVA is used as the secondary sizing agent to starch for cotton yarns due to better film forming abilities. The desizing of PVA size high temperatures (90-95°C) often leads to re-deposition of PVA over the fabrics due to gel nature. Capacitively coupled device with low frequency 1-12 kHz at 3.4 kPa using air-He plasma has been used to desize the cotton fabrics treated with PVA (size content 8 %) to analyze the weight loss, percent desizing ratio, tensile strength, SEM for surface appearance. The percent desizing ratio reached up to 97 % after 10 minute treatment whereas traditional peroxide results in 76 % removal.

Non- polymerizing plasma using oxygen gas has been used in a study to scour cotton and wool and also the dyeing processes. The contact angle decreases considerably after oxygen plasma treatment compared to untreated samples and the plasma treatment followed by scouring results in the residual wax content to 0.8% in cotton and wool. The dyeing rate also increases for both after plasma treatment.

2.10 Plasma processing for dyeing and finishing

Surface modification of textile materials to create a reactive surface suitable for dyeing and finishing treatments has been attempted by many researchers (Bozzi, 2005; De Geyter, 2006; Virk, 2004). The exhaustion levels of raw cotton fabric pre-treated with corona acid groups within the cuticle layer was suggested and also an increase in surface acidity. Corona treated fabrics tend to be darker in colour than the untreated samples with deviations in chroma and mainly due to differences in the initial un-dyed substrates.

Hydrophilisation has been carried out on cotton using ratio frequency plasma at 20 kHz with power of 0.64 W/cm^2 and pressure of 0.6-8 mbar to reach the saturation of hydrophilisation. Hydrophilisation increases with penetration depth and velocity of penetrating front. The effect of plasma treatment on fabrics made of various fibres and the relevant results are detailed in the following Table 2.4

Development of self cleaning cotton textiles through RF plasma, MW plasma and UV radiation to introduce functional groups to anchor Tio_2 on textile surface shows the formation of TiO_2 crystallites with 5-7 nm size immediately, form the precursor

Table 2.4 Effect of Plasma Finishing on various Fabrics

Fibre	Process	Gas, process Conditions	Effect
Cotton	Hydrophilicity and wettability (RF Plasma)	Air-O_2 Low pressure 0.6-8 mbar Air- O_2, Low Temp. at 9 Pa pressure, 70-120W	Weight loss, increase in C=O,COOH contents, increase in vertical wicking, strong etching Generally greater changes in fibres
	Hydrophobicity (RF plasma)	CF_4, C_3F_6,100,300W,50 & 75 m Torr for CF4 50, 160 W, 50-150 m Torr for C_3F_6	Lower results for CF4 than C3F6, polymerises by plasma and plasma inducing methods. CF6 yields plasma polymerization, atomic fluorine only.
Wool	Hydrophilicity (RF Plasma)	Water vapour,Power 100 W and pressure 100 Pa	Removal of fatty layer, generation of hydrophilic groups, Epicuticle is removed.
	Shrink resistance (Glow Discharge)	O_2, Low temperature at 10 Pa pressure	Felting decreases, becomes shrink resistance, alkaline solubility increases and dyes faster, Micropores and clef created. Subsequent enzyme treatment enhances handle and dyeability.

2.11 Effect of Plasma Treatment on Fibre Properties

The effect of plasma treatment on the textile materials depends on type of gases used and the type of substrates used (Bajaj 1998). The free radicals produced in the treatments can undergo reactions upon the gases present in the atmosphere. The substrate can be coated with different kinds of gases without changing the fabric bulk properties.

The yellowing effect on wool fibres reduce with plasma treatment during steaming operation carried out in setting and finishing processes. Abrasion resistance, breaking force also increase in the case of fabrics along with reduced time of half dyeing and barrier effects. Plasma treated cotton fabric source but this also results in reduction in weight loss subsequent cellulose treatment. However, oxygen plasma on to wool improves strength and higher rate of weight loss in subsequent process treatment. As the plasma treatment is carried out in dry condition, the fibres are not swollen and changes on surfaces are restricted. The DFE is reduced but fibre/fibre friction increases. Surface roughening caused by a plasma treatment of wool results in harsh handle.

Plasma treatment causes abrasion of fiber surface by introducing different kinds of surface roughness such as cracks and fissures. Even very high power supply with prolonged treatment results in no significant changes in tenacity and elongation at break values for both silk and cotton fabrics. Grafting methods employed using plasma treatment does not alter the mechanical strength, probably due to attachment of monomers in the surface not in entire bulk.

Plasma treatment offers effect which are decaying In nature i.e. ageing or durability effect. The effects produced by plasma decreases over a period of time e.g. Wettability, unless special measures are taken in the selection of gases. E.g. oxygen or oxygen containing gases this decaying nature of the effect affects the finishes imparted through the plasma treatment e.g. diapers, hygienic products.

2.12 Cotton fabrics treated by corona discharge after several laundering

Cotton fabric because of finishing process has shrinkage behavior after laundering and for improving this problem, chemical agents utilize for anti-shrinkage. This temporary finishing can reduce shrinkage but has many environmental problems. Corona discharge treatment has been used for cotton fabric, other fibers and polymers. This treatment can reduce costs and environmental impacts. In this study, cotton fabric was treated by corona discharge instrument at 2 - 40 passages and fabrics were washed with laundering method. After four times laundering, shrinkage behavior of corona discharge treated fabric as compared with un-treated fabric decrease. After 10 passages of corona discharge treatment, water, dye absorption and shrinkage are modified but after 14 passages, despite of shrinkage improvement, dyeing properties decrease.

Corona treatment consists on the application of an electrical discharge of high voltage (around 10.000 V) through air between two electrodes, using frequencies around 40 kHz, at normal atmospheric temperature and pressure, on dry cotton fabric (Ward et al., 1979). Physical and chemical surface changes in the cotton's structure are noticed after Corona discharge (Pavlath and Slater, 1971). Corona discharge has been proved to be a useful method for improving the polymer surface hydrophilic property, especially in the composite material strength improvement (Riccobono and Rolden, 1973; Stone and Barrett, 1962; Jung et al., 1977; Ward et al.1978), the treatment can improve the surface affinity and the sticking strength with some hydrophilic polymers, because the treatment can lead to the increasing of the high reactive free radical oxygen in the polymer surface.

Corona discharge was also applied in the chemical graft copolymerization (Shishoo, 1996; Lieberman and Lichtenberg, 1994), this is also based on that after the irradiation, new active group is produced and the monomer can be successfully grafted onto those active groups in the irradiated material. Some other researchers have introduced the corona discharge treatment into the textile surface treatment to improving the wool fabric printability (Rakowski et al., 1982) or the wool fabric shrinks resistance properties (Marsh, 1987; Venugopalan, 1971). Corona discharge technology is also widely used in the printing of some polymer films (Venugopalan, 1971; Benerito et al., 1981). The treatment has been confirmed to be simpler and more practical than any other chemical and physical methods because the samples can be quickly treated under atmosphere pressure (Ward et al., 1978).

2.13 Surface Functionality of Cotton Fibres by Atmospheric Plasma Treatment

Cotton is mainly composed of cellulose with some non- cellulosic components. These non-cellulosic components are waxes, pectin and some proteins, and they are mainly found in the cuticle layer and the primary wall which are the outermost layers of the cotton fiber. These hydrophobic impurities, especially cotton wax, affect the uptake of dyeing and finishing solutions. To remove these impurities from the cotton surface, certain chemical methods are used in the textile industry. These conventional methods are energy- consuming processes with negative environmental impact . In this concern, many alternative environmentally friendly methods were developed. Plasma treatment is one of those methods and can be used as an effective technique for modifying the surface properties of cotton fabric without altering the interior part of the fiber.

Plasma is generated when a gas is exposed to an electro- magnetic field at low pressure and near ambient temperature. The chemistry of the plasma takes place in non-equilibrium conditions. Plasmas can be classified as low pressure and atmospheric plasmas. Both plasmas can be used for surface modification of materials. Vacuum systems are time-, space-, and energy-consuming processes, and some material properties, such as thickness and size, are highly dependent on the dimension of the device and, in addition, the process is not a continuous one. On the other hand, atmospheric plasma can be generated under atmospheric conditions and does not require vacuum systems with continuous and open perimeter fabric flow. The efficiency of plasma treatments depend on treatment conditions of time, pressure, power and gas. The species that participate in plasma reactions, such as excited atoms, free radicals and metastable particles, electrons and ions, can interact either physically or chemically with substrates

Numerous researches have been carried out to improve wettability, water repellency and soil releasing property of textile fibers and fabrics by using plasma technology. Additionally, modifications of conventional dyeing, printing and finishing properties have been achieved by plasma treatment methods. However, low-pressure plasma was used in most of the studies. In this study, the effects of air and argon atmospheric plasma treatments on the functionality of cotton fabrics were investigated.

The wet processes in textile finishing unfortunately require great amounts of water. This requisite leads enormous energy consumption along with the pollution of subterranean water with wastewater as well as thermal pollution. Therefore the main purposes of this study are to investigate the potential of viability of the atmospheric plasma methods for reducing energy consumption and water pollution as being an alternative method i.e. replacement of wet textile pre-treatment processes.

2.14 Low Temperature Plasma for Silk

Low temperature plasma treatment on silk fabrics, films and sutures have been attempted to improve wettability, Hydrophobicity and bio-compatibility of sutures and biological support materials. RF plasma has been applied to silk material using silicon hexafluoride gas to improve the Hydrophobicity of materials and also assessed with reference to the ageing effects using Rf frequency of 13.56 MHz, pressure of 1-7 mttor and the power in the range of 25 to 75 W efficient attachment of fluorine atoms on the polymer surface takes place during this plasma treatment, which means to water repellency.

Fibroine of the silk treated with low temperature plasma using oxygen, tetrafluoro methane gases to analysis the immobilization of the enzyme shows of alkaline phosphatase and improved activity with both the gases.
Surface flutes are also absorbed in the surface of the silk sutures treated with low temperature oxygen plasma with increased loading capacity. The etching effect obtains in the sutures also leads to reduction in weight of the fibre. On contrary to other findings a reduction in the crystallinity has been absorbed in this study.

2.15 Plasma Treatment on Wool Fibre and Fabrics

Extensive work has been carried analyze the effect of various low temperature plasma on wool fibres and fabrics to improve the processability physical and chemical properties.

The chemical composition of wool fibre surface varies differently with the gases used in the plasma generation. Low temperature oxygen plasma on wool incorporates-OH, C=O, -COOH functionality and is considered to be alternative for wet clolorinatrion for improving wettability and shrink resistance. Conversion of disulphide to acid is almost 90% which is higher than UV or ozone oxidization of the wool fires. Nitrogen plasma introduces $-NH_2$ groups in the fibres which become dye sites on wool, increasing dye aborbtion. Low temperature plasma using oxygen, nitrogen and mixture nitrogen and hydrogen result in formation of bunte salt, cysteic acid due to cleavage of di sulphide linkage.

Detailed analysis on wool fibre, yarn and fabrics have been carried out using glow discharge plasma to analysis the effect like surface changes in chemical composition and various physical properties. The effect of glow discharge plasma on wool fibres on spinnability and the substitution in yarn properties has been studied extensively. Fibre to fibre frictions increase but directional friction effect decreases. The treatment does not change the strength and elongation. Under the influence of low temperature plasma, tearing strength of wool fabrics reduce both in warp and weft direction, mainly due to decreases in the sliding of the yarns.
The plasma treatment followed by scouring, results in the residual wax content about 8% in the wool fibres. The dyeing rate also increases for plasma treated

fibres. The contacts angle decreased considerably after oxygen plasma treatment compared to untreated samples. Plasma treatments are also explored in highly pollution causing chrome dyeing for dark shades. Oxygen, nitrogen and mixture of oxygen (25%) and nitrogen (75%) plasma results in reduction in half time of dyeing and increases in the final exhaustion.

In case of dyeing regular dyeing process poor wettability of untreated fibers shows poor dye exhaustion initially but then proceeds similar to the treated fabrics. However, the final time to reach exhaustion was shorter for plasma treated specimen. Alteration of fiber surface provides a path way for dye stuffs of diffuse in to the fibre easily. Similar reflectance curves or obtained for both the samples show the similar depth of dyeing. Dry rubbing fastness showed improvement slightly no such thinks in wet rubbing. Wash fastness and fastness to perspiration increases compared to un treated fabrics.

The presence of scale structure on wool fibre surface introduces a number of problems such as felting and surface barrier of dye stuff. The shrinkage and fabric takes place due to relaxation, consolidation and felting shrinkages. A dimensional changes obtained in the warp are higher than weft in terms of decreases in shrinkage. Consolidation shrinkage is higher for untreated fabrics than treated ones. Felting dimensional changed reduces from 9.6% to 1.1% for the treated fabrics in warp direction and 12.3 % to 1.5% in the area of shrinkage. That happens mainly etching effect with decreases the natural shrinking tendency. Combination of oxygen low temperature plasma and silicon polymer treatment improves the dimensional stability more than silicon treatment alone.

Breaking load, elongation at break of low temperature plasma treated fabrics is comparatively larger than those of the untreated fabrics. The inter-yarn, inter bond fiber friction play major role in tensile strength of fabrics which increases either low temperature plasma due to surface roughness. Cleavage of disulphide linkage appears to make the scales more elastic, besides increases in the friction and modify the elongation at break.

Plasma treatment offers effect which are often decaying in nature e.g. ageing or durability effect the effects produced by the plasma decrease over a period of time e.g. wettability, unless special measures are taken in the selection gases e.g. oxygen or oxygen containing gases. This decaying nature of the effect affects the future imparted through the plasma treatment e.g. diapers, hygienic products.

2.16 Characterization of Annatto

Family: Bixaceae

Genus: Bixa

Species: orellana

Synonyms: Bixa acuminata, B. americana, B. odorata, B. platycarpa, B. purpurea, B. tinctoria, B. upatensis, B. urucurana, Orellana americana, O. orellana

Common names:achiote, achiotec, achiotl, achote, annatto, urucu, beninoki, bija, eroya, jafara, kasujmba-kelling, kham thai, onoto, orleanstrauch, orucu-axiote, rocou, roucou, ruku, roucouyer, unane, uruku, urucum, urucu-üva

Plant Description

Ti is an evergreen shrub found throughout America, Caribbean, India and Africa. Most of the world's supply comes from Kenya, Peru and Brazil. An attractive pink flower made it popular as a hedge plant in colonical gardens. The fruit capsule is heart-shaped, like a beech pod, with opposing clefts and red prickly spines. When ripe, the pod splits an half to reveal about fifty seeds encased in a red pulp. The pulp is used in many commercial dye products.

The Bixa orellana is commercially grown for the dye products and for its seeds as a spice. It requires a tropical setting in a loamy soil at altitudes below 1000 M (3000 ft). it is sown from cuttings. The ripe fruits are collected then macerated in water. The dye settles and is collected and into cakes and the seeds is separated and washed.

Annatto trees produce crimson pods containing numerous seeds. The outer pericarp of the seed is covered with a thin layer of Bixin pigment. Seeds from both cultivated and wild plants are harvested and dried prior to processing. The dye extracted from the seeds is called Bixin. Extract color with alkali solution to obtain is called Nor-Bixin.

2.16.1 Plant Chemicals

Analysis of annatto seeds indicates that they contains 40% to 45% cellulose, 3.5% to 5.5% sucrose, 0.3% to 0.9% essential oil, 3% fixed oil 4.5% to 5.5% pigments, and 13% to 16% protein, as well as alpha- and beta-carotenoids and other constituents. Annatto oil is extracted from the seeds and is the main source of pigments named bixin and norbixin, which are classified as carotenoids. Bixin, extracted and used as a food colorant, has been shown to protect against ultraviolet rays and to have antioxidant and liver protective properties in clinical research.

Annatto Dye

Annatto dye is basically a red orange pigment known as Bixin, extracted from the seed coat of Sinduri (Bixxaa Orellana). The quantity of Bixin in the seeds varies from 0.73% to 1.3 % by weight and contains carotenoid of various types out of which cisbixin alone accounts for 82%. Since bixin is the principle colouring matter, the chemistry and performance of Annatto colour is essentially of the bixin. Bixin is highly unsaturated compound.

Fig. 2.2 Chemical structure of Annatto Dye

The Bixin dissolves in vegetable oil, undergoes complex series of isomerisation and degradation reactions when heated to extraction temperatures. A yellow pigment transbixin and cisbixin are the major carotenoids in oil soluble annatto colour. The total pigment content of the commercial annatto butter colour varies from .2 to 2.6 percent, at least 30 percent of which is bixin.

Annatto based colors are used in

Cheese processing, fish processing, confectionery manufacturing, skin care products formulation, medical products formulation, vegetable shortening, and oils, Diary, cosmetic, dye, and leather industry.

CHAPTER 3

MATERIALS AND METHODS

3.1 MATERIALS

The cotton and silk fabrics of following specification were taken

Cotton Fabric Specifications

- Bleached and woven fabric is taken
- Fabric warp count -80'Ne
- Fabric weft count -80'Ne
- Ends per inch -156
- Picks per inch -108
- GSM -1.27 grms/sq.m

Silk Fabric Specifications

- Bleached and woven fabric is taken
- Fabric warp count -41 denier
- Fabric weft count -49 denier
- Ends per inch -118
- Picks per inch -98
- GSM -0.43grms/sq.m

3.1.1 Dyeing of Plasma Treated material with Natural Dyes

- Natural dye Annatto supplied from Gandhigram Trust, Dindugul was used for dyeing.

Mordanding Technique:

Post Mordanting:

In post mordanting, the cotton sample was treated with 6% shade at optimized time and temperature and then mordanted with alum(5%) for half an-hour at 50^0C. Then, the samples were removed from the dye bath and rinsed to remove the excess dye particles, washed and dried.

3.1.2 Plasma Reactor

A capacitively coupled atmospheric pressure DC plasma system with provision up to 3Kv and 500mA power supply, supplied by M/s hydro pneoVac, Bangalore, available at Bannari Amman Institute of Technology, Sathyamangalam, was used for this study .design allowed, the distance between two electrodes to be varied from few millimeters to 15cms. The plasma was generated using a power supply with variable output. It is also possible in the system to detach the electrodes and replace with another electrode. Aluminum and copper electrodes were used independently in the present study. The schematic line diagram of the reactor is given below.

3.2 METHODS

3.2.1 Plasma Treatment

Samples of 20 × 20 cm placed in the middle of two parallel electrode plates, were treated with Atmospheric Air and oxygen plasma with electrodes such as Aluminum and Copper with spacing between electrodes of 3 cm for same durations. Fabric samples were placed between the electrodes, the working pressure was adjusted with a rotary vacuum pump. Evacuations were carried out for duration of 10 min. and then the power supply was initiated. The delivering power between the electrodes was adjusted using the power setting knob. The glow discharge was maintained and the fabric samples were exposed to plasma for the required duration after which it was switched off and the samples was allowed to be in vacuum for 10 minutes; the vacuum pump was then turned off after which the chamber was purged with air and the treated samples was removed for study.

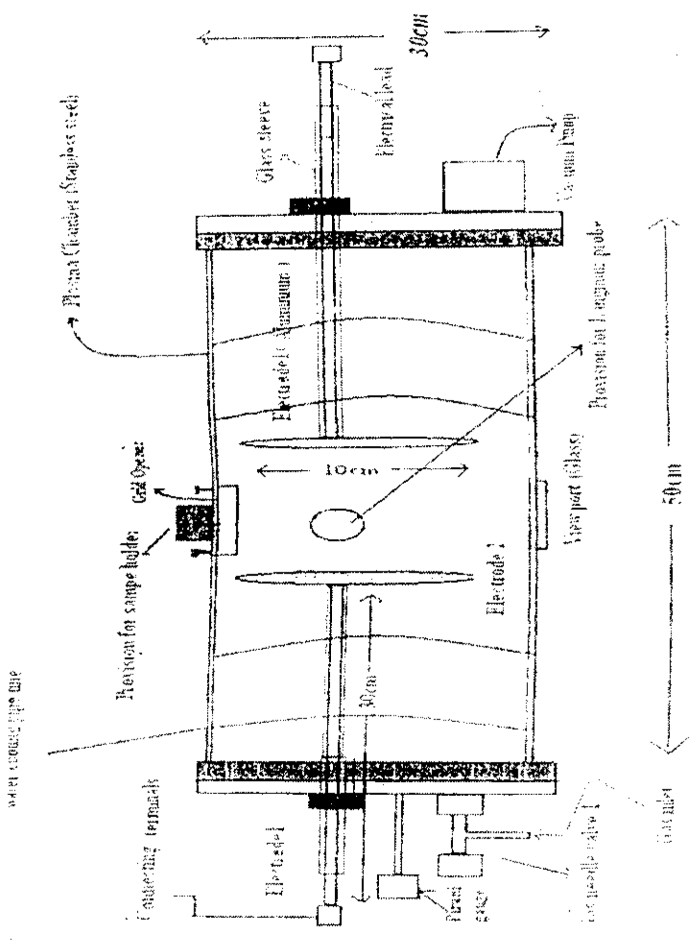


Fig. 3.1. Plasma Chamber

Gas flow rate and output power were maintained at 10 cc/min. 300v respectively throughout the study. Treatment time was 6min. and the pressure was 300mtorr.

3.2.2 Dyeing

The treated and untreated fabrics were dyed using the natural dyes with 5% shade at optimized time and temperature, keeping m:l ratio as 1:20. The dyed fabrics were then mordanted with 5% Alum for half-an-hour by the optimized mordanting technique according to the dye.

Annatto – post mordanting

Then, the samples were removed from the dye bath and rinsed to remove the excess dye particles, washed and dried

3.3 Characterization of Plasma Treated Fabrics

3.3.1 Hydrophilicity

The hydrophilicity of the treated and untreated samples was measured using the following 2 methods.

Drop test

The absorption time of a water drop was measured according to the standard AATCC Test method 79-2000. A drop of water was allowed to fall from a fixed height of 2.5cm on to the taut surface of the test specimen. The time required for the specular reflection of water drop disappears is measured and recorded as wetting time. Average of 5 reading is reported.

Wickability

The procedure used to measure the fabric wickability was based on the standard BS: 3432. A sample of 20*5 cm was vertically suspen b-magnification-10,000X ded with an end immersed in water to a height of 2mm and water claimed height due to the capillary action was measured after 30 minutes.. 5 tests each for treated and untreated fabric a were tested, and the average was reported. Higher the height of water climbed, better the wicking property.

3.3.2 Etching Loss

Etching loss calculated using analytical weighting balance with accuracy of 0.001g. fabric was first dried at 60°C for 10 min. and then plasma treated, dried at the same conditions and the etching loss was calculated by the following formula.

$$\%\, Etching\ loss = \frac{W1 - W2}{W1}\ X\ 10C$$

Where W1 and W2 are the weights of the untreated and plasma treated samples respectively

3.3.3 Moisture Content

Moisture content of the samples was determined after conditioning using the weighing bottle and balance as per ASTM D29 -1999 following the formula given below.

$$\%moisture\ content = \frac{WC - WD}{WC} X100$$

Where WC and WD are the weights of treated samples and dry samples respectively.

3.3.4 FTIR Analysis

Shimadzu Fourier Transform Infrared Spectroscopy (FTIR) 8400S in ATR mode was used to assess the surface modifications in terms of chemical groups implanted in the plasma treated samples by comparing with untreated samples. The finger prints were obtained with wave numbers in the range of 4000 to 400 cm^{-1}, resolution of 4cm^{-1} and final 20 scans to reduce noise effects in measurements.

3.3.5 SEM Micrograph

Fibers from the samples were gently unraveled and Surface Morphology was examined in a Hitachi Model S-3200 Scanning Electron Microscope at a magnification of 2500X and 6000X at 5.0kV.

3.3.6 Tensile Strength

Tensile strength of the plasma treated and untreated samples were measured in Premier Tensomax 7000 tensile testing instrument according to ASTM D 5035. The instrument works by the principle of CRE and a constant rate of 100 mm/m was used for testing. Size of the test specimen was 158 10 cm and the average for test results for each samples was reported.

3.3.7 Conditioning

The treated and untreated samples were conditioned as per ASTM D1776 at 65% RH and 25°C for 24 hours in an environmental chamber before taking them for any testing.

3.3.8 Colour Strength

Annatto dyed samples were measured for the colour strength (K/S) at $?_{max}$ (wavelength of maximum absorbance) using Kubelka-munk function, in Gretag Magbeth computer colour matching system.

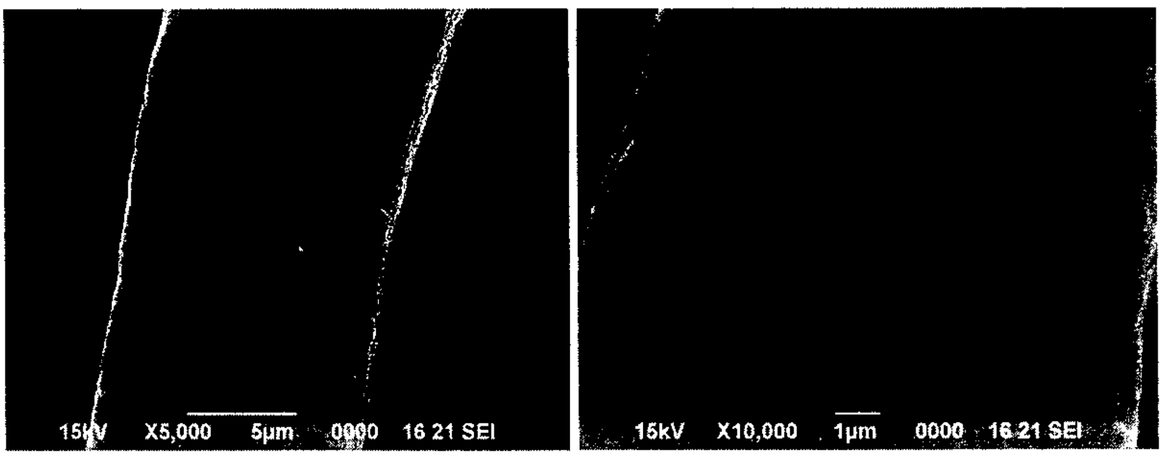
$$\frac{K}{S} = \frac{(1-R)^2}{2R}.$$

CHAPTER 4

RESULT AND DISCUSSION

4.1 Surface Morphology Analysis

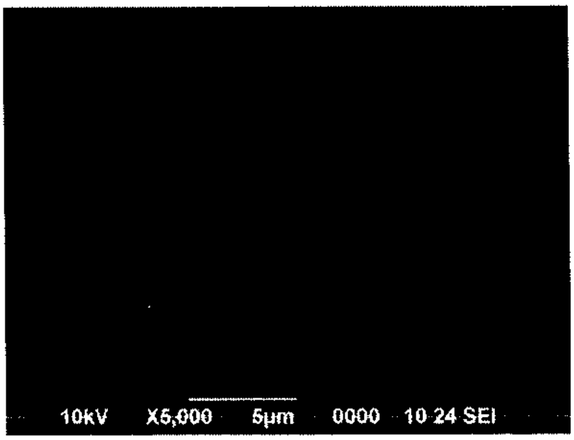
The effect of plasma treatment on morphological changes on the untreated and treated samples are shown in fig (4.1, 4.2, 4.3) the untreated cotton had a normal surface and silk has smooth surface , while all plasma treated sample exhibited surface morphological changes. Increased surface roughness can be produced by the etching effect of plasma active species bombardment on the cotton and silk fabric

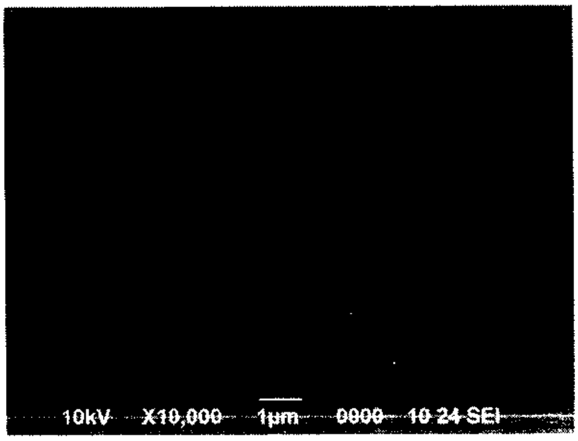


(a) (b)

(a) untreated cotton with 5000X magnification

(b) untreated cotton with 10000X magnification





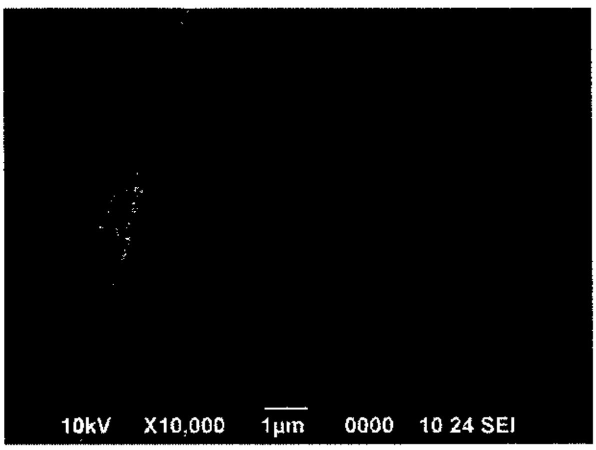
(c) (d)

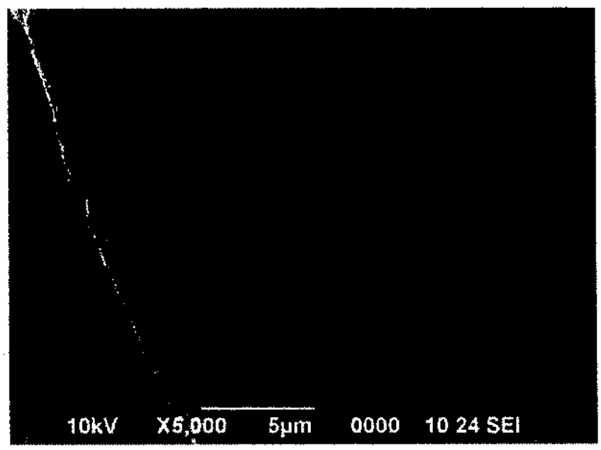
(c) untreated silk with 5000X magnification

(d) untreated silk with 10000X magnification

Fig. 4.1(a, b, c, d) SEM micrograph of untreated cotton and silk fabrics

After exposure for five minutes, the surface morphology of cotton treated with oxygen plasma showed more surface roughness than cotton treated with air plasma.

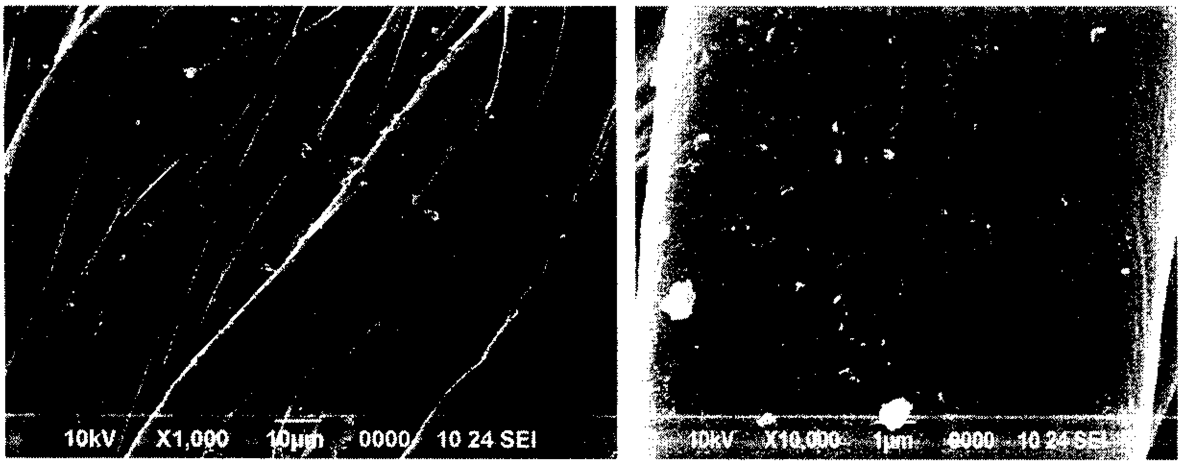




(a) (b)

(a) Oxygen plasma treated cotton with 10000X magnification

(b) Oxygen plasma treated cotton with 5000X magnification



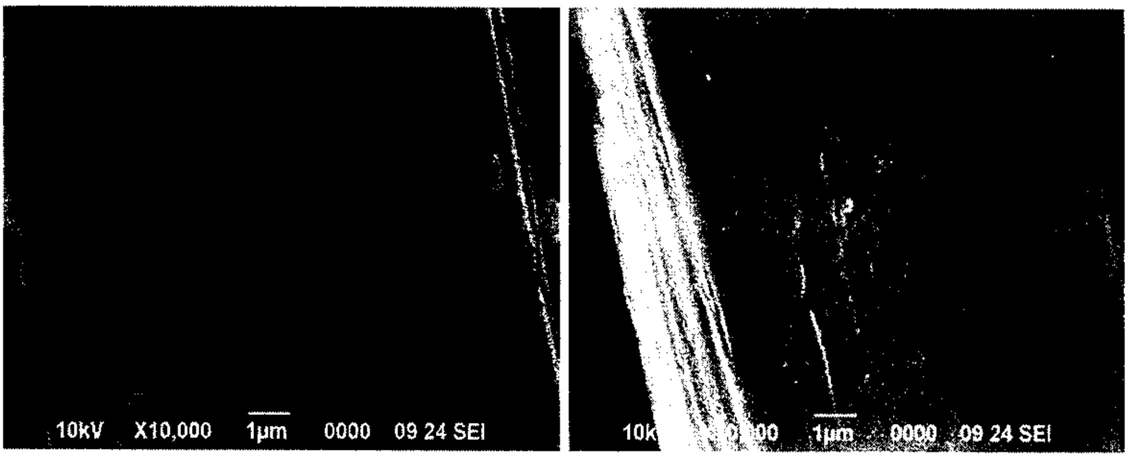
(c) (d)

(c) Air plasma treated cotton with 1000X magnification

(d) Air plasma treated cotton with 10000X magnification

Fig. 4.2(a, b, c, d) SEM micrograph of treated cotton fabric Sample

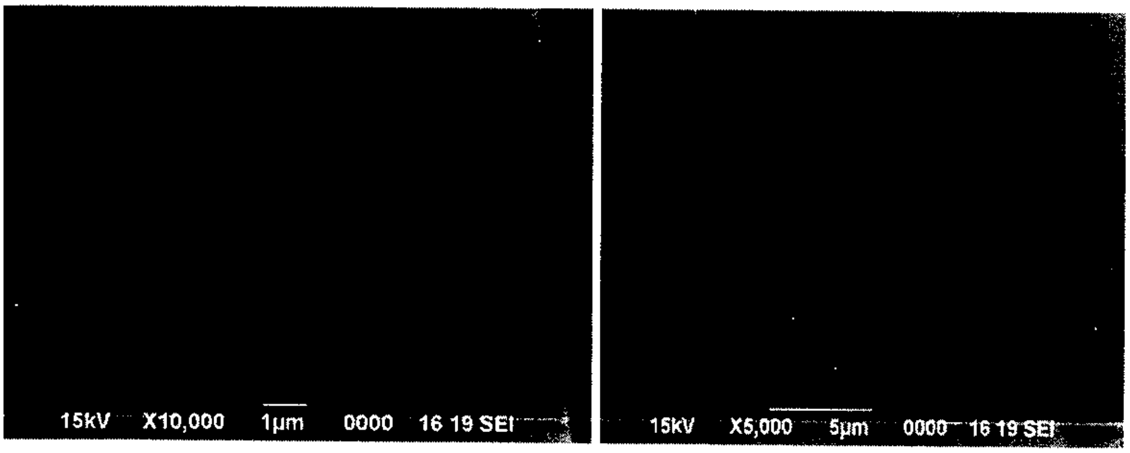
In case of silk, after exposure for five minutes the surface morphology of silk treated with air plasma showed more surface roughness than silk treated with oxygen plasma.



(a) (b)

(a) Oxygen plasma treated silk with 10000X magnification

(b) Oxygen plasma treated silk with 10000X magnification



(b) (d)

(c) Air plasma treated silk with 10000X magnification

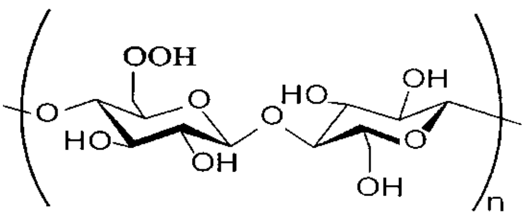
(d) Air plasma treated silk with 5000X magnification

Fig. 4.3(a, b, c, d) SEM Micrograph of treated silk fabric Sample

4.2 FTIR Spectra Analysis

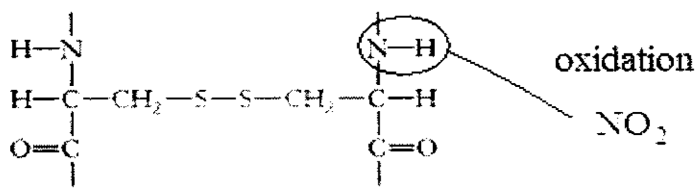
The FTIR spectra obtained for untreated and treated samples shown in Fig. Formation of hydrogen bonds and oxidation of compounds are the various happenings in the fabric during plasma treatment.

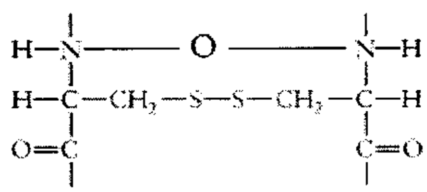
The spectra obtained for the oxygen plasma treated cotton sample showed oxidation of alcohol group to carboxylic group which causes more etching effect and thus increasing the water absorbency and dye uptake and in case of air plasma treated sample less effect compared to oxygen plasma had taken place.



Change occurred in Cotton

The spectra obtained for oxygen plasma treated silk sample showed the formation of hydrogen bonds between the amine groups and also oxidation of amine groups into nitro groups in some places. And in case of air plasma treated silk less effect compared to oxygen treated plasma had taken place.





Changes occurred in Silk

4.3 Effect on Plasma Treatment on Etching Loss

Etching or cleaning to remove the surface layer basically with gas plasmas caused a cold burning process which transforms the surface in to typical burning products like water, carbon di oxide, nitrous oxide. The results are shown in table4.1. Insignificant weight loss obtained, the roughness effect seen in SEM micrograph was predominant the highest weight loss occurs with oxygen followed by air-plasmas in case of cotton and the highest weight loss occurs with air followed by oxygen-plasmas in case of silk. The difference in weight loss with respect to gas plasmas conforms the physical etching nature and rates are different for different gases. This is also reflecting in the SEM micrograph.

Table 4.1 Effect of Plasma Treatment on Etching Loss

S.no	Fabric	Untreated Sample Wt (gram)	Treated Sample Wt (grams)		weight loss in %	
			Oxygen	Air	Oxygen	Air
1	COTTON	3.65	3.61	3.63	1.09	0.54
2		3.64	3.59	6.63	1.37	0.27
3		3.66	3.61	3.63	1.33	0.81
1	SILK	2.85	2.84	2.80	0.35	1.07
2		2.83	2.81	2.79	0.70	1.40
3		2.84	2.83	2.81	0.35	1.05

4.4 Effect on Plasma Treatment on Wettability

The wettability/hydrophilicity for treated and untreated cotton and silk samples were assessed by two methods viz. absorbency by drop test (time. of wetting) and wicking height. The results of the test are showed in table significant reduction in wetting time i.e., improver wettability. The untreated cotton and silk samples showed less absorbency than the treated samples. The gas used for the treatment had significant effect on the absorbency. In case of cotton oxygen plasma showed greater absorbency than the air plasma and in case of silk air plasma showed greater absorbency than the oxygen plasma. This is agreement with the FTIR analysis.

Table 4.2 Effect on Plasma Treatment on Wettability

FABRIC	SAMPLES/TREATMENT	Water level (cm)
Cotton	Untreated	4.8
	Air treated plasma	8.2
	Oxygen treated plasma	5.8
silk	Untreated	5.8
	Air treated plasma	6.9
	Oxygen treated plasma	6.7

Table 4.3 Effect on Plasma Treatment on Drop Test

FABRIC	SAMPLES/TREATMENT	TIME (sec.)
Cotton	Untreated	39
	Air treated plasma	6
	Oxygen treated plasma	4
silk	Untreated	132
	Air treated plasma	114
	Oxygen treated plasma	83

Wetting and wicking are two related process, the liquid does not wet fibers cannot wick in to fabric, so the fiber wettability is a prerequisite of wicking. The higher wicking by plasma treated samples can explained several possibilities which are

1. The higher level of polar hydroxyl, carboxyl and carbonyl groups generated by the fibre damage during plasma treatment, yield a more polar and wettable fibre surface.
2. The physical effect of plasma treatment which, through surface erosion removes the layers on the fibre surfaces, may thereby render the fibre more wettable by water.
3. Plasma treatment results in weight loss as a result of surface etching of the fibre. Does the shape and size distribution of the interfiber capillary spaces will be modified.

This may also lead to the ulocking of surface capillaries and hence promote more rapid wetting.

4.5 Colour Strength (K/S) in Natural Dyeing

To evaluate the affinity of natural dyes on cotton and silk fabrics, they were dyed with natural dye. While the natural dye using annatto was carried out according to the standard procedure.

The improvement in wettability improves the dyeing characteristics and the changes taken place in the fibre molecule either by breaking of chemical bonds or by oxidation helps the dye to interact with the fibre and the etching caused by the plasma has created rough spots in the fibre and the dye particles deposit into these spots, resulting in increased color strength which is confirmed by the reasonably good color strength values obtained in dyed plasma treated samples as compared to only tinting seen in the untreated fabric samples. The increase in the surface roughness could be the reason for the imparted darker appearance to the dyed fabrics.

Table 4.4 Colour Strength (K/S) in Natural Dyeing

FABRIC	SAMPLES/TREATMENT	K/S at $?_{max}$
Cotton	Untreated	5.40
	Air treated plasma	3.86
	Oxygen treated plasma	7.92
silk	Untreated	19.93
	Air treated plasma	54.56
	Oxygen treated plasma	24.52

4.6 Effect on Plasma Treatment on Moisture Content

While the untreated cotton and silk had 8% and 11 % moisture content respectively, an increase up to 3% for silk was demonstrated by the plasma treatment which is considered as a significant improvement in the moisture content. But for cotton the moisture content increased upto 2.5%.

The moisture content of plasma treated cotton and silk will depend on two main factors: the changes in the surface morphology due to the etching action of the plasma and the changes in the molecule of the fibre, which will help moisture penetration and binding on the surface. These could be the possible reason for the improved moisture content observed.

4.7 Effect on Plasma Treatment on Tensile Strength of the Fabric

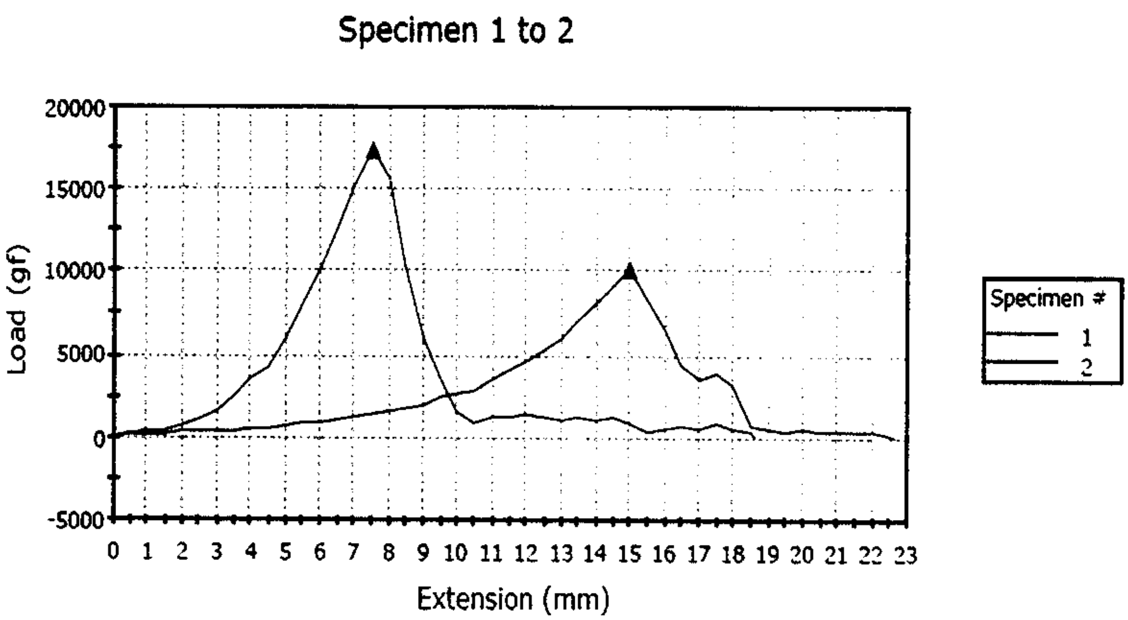
Tensile strength measurement of the untreated and plasma treated samples results are attached in annexure. Significant change in tensile strength was absorbed with plasma treatment in the samples. While consideration of cotton, there is a decrease in strength of oxygen treated sample when compared to air treated sample. This is due to increase in other functional properties like absorption and dye uptake etc.

In silk oxygen treated plasma sample show good tensile strength when compared to air and untreated samples because here the oxygen plasma treatment does have much effect on silk. There is improvement in tensile properties of air treatment of silk and this is confirmed by test results.

Fig. 4.4 TENSILE STRENGTH OF TREATED AND UNTREATED FABRICS

Raw Cotton

Text Inputs: Specimen label
Test: Rate 1 300. mm/min



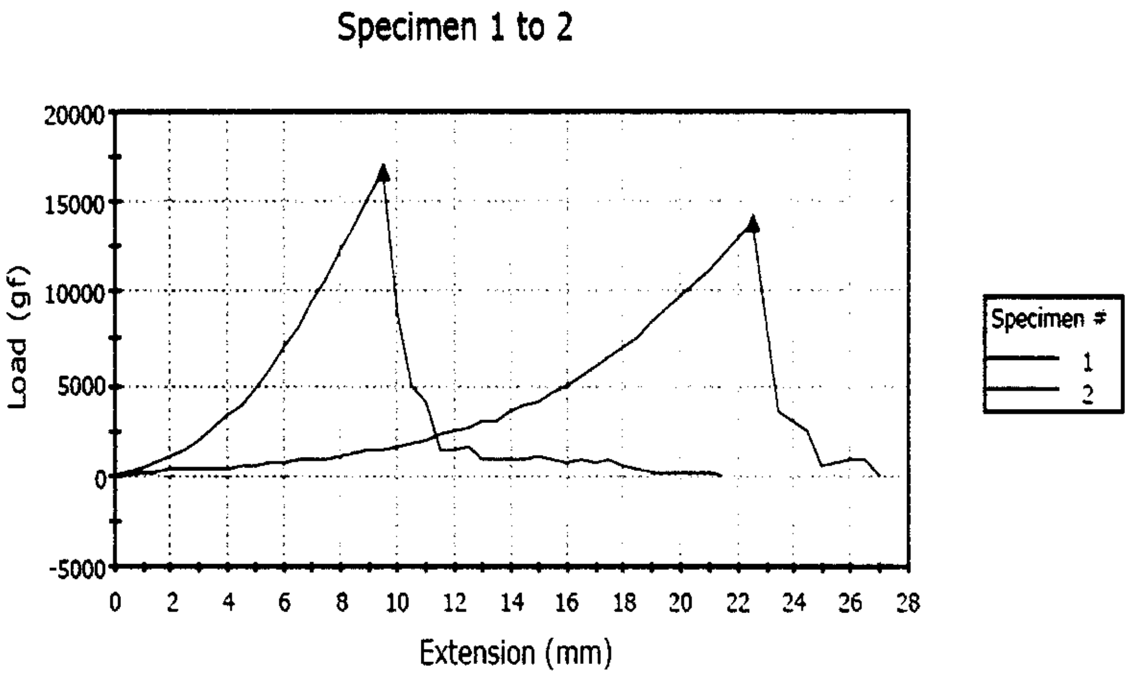
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	17332.17	7.50	173.32	7.50
2	10015.81	15.00	100.16	15.00

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	7.50	28.33
2	20.00	16.37

Tensile Strength of untreated cotton Sample

ATM Cotton

Text Inputs: Specimen label	ATM Cotton
Test: Rate 1	300. mm/min



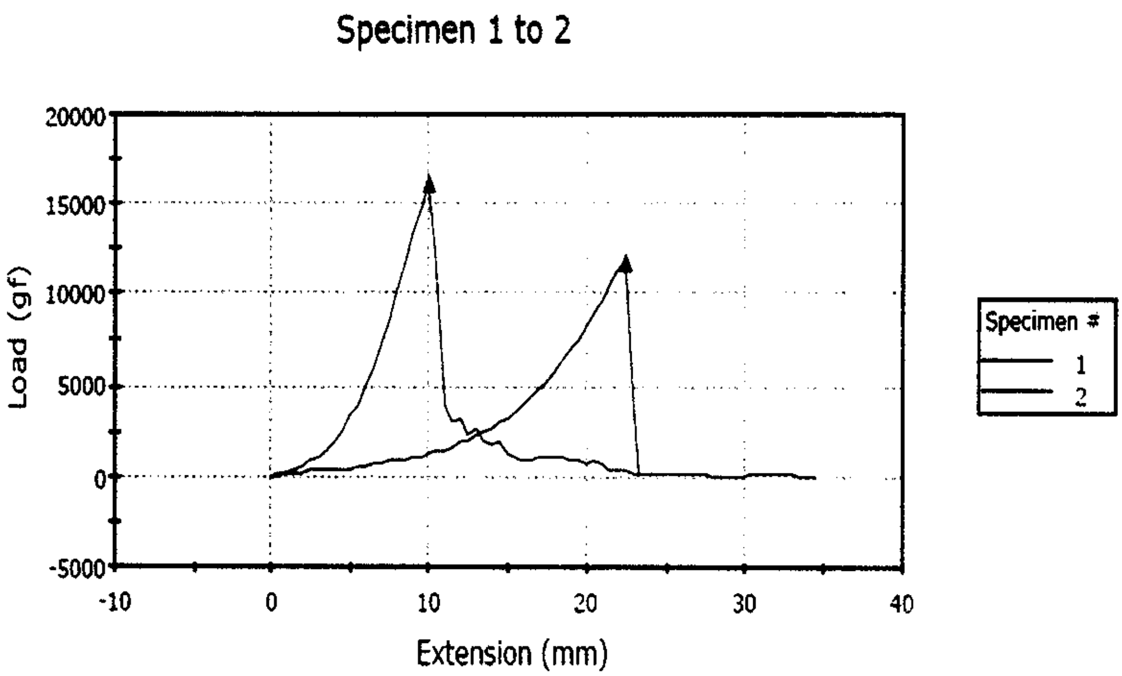
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	16548.52	9.50	165.49	9.50
2	13783.80	22.50	137.84	22.50

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	12.67	27.05
2	30.00	22.53

Tensile Strength of Air treated Plasma Cotton sample

Oxygen Cotton

Text Inputs: Specimen label	Oxygen Cotton
Test: Rate 1	300. mm/min



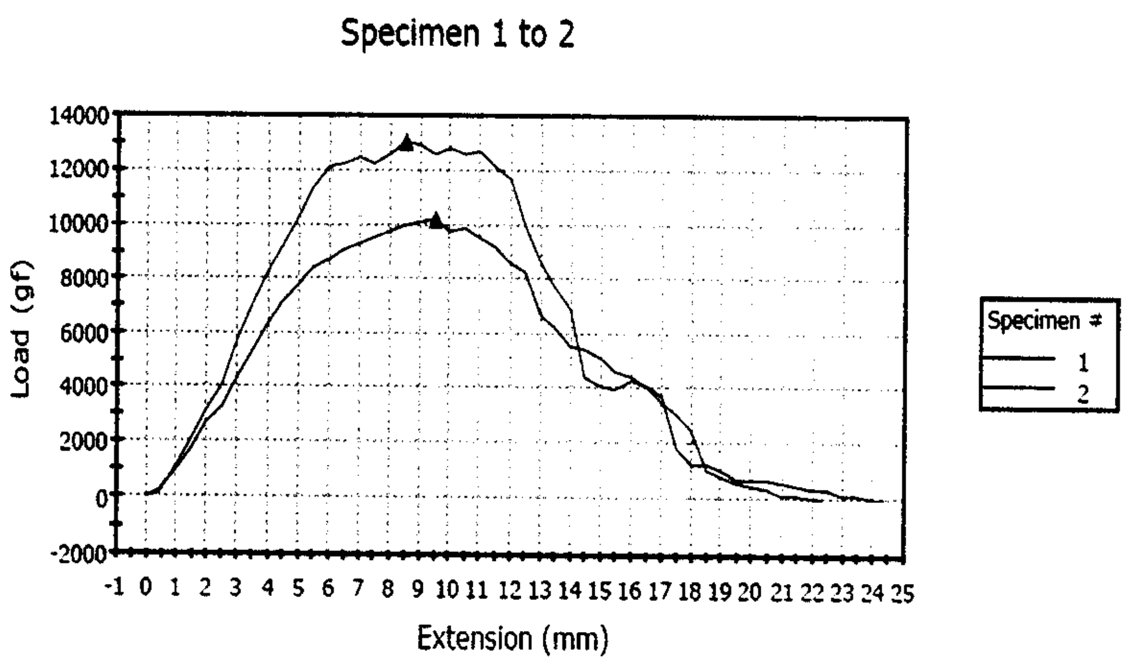
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	16117.99	10.00	161.18	10.00
2	11748.23	22.50	117.48	22.50

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	13.33	26.34
2	30.00	19.20

Tensile Strength of Oxygen treated Plasma Cotton sample

Raw Silk

Text Inputs: Specimen label	Raw Silk
Test: Rate 1	300. mm/min



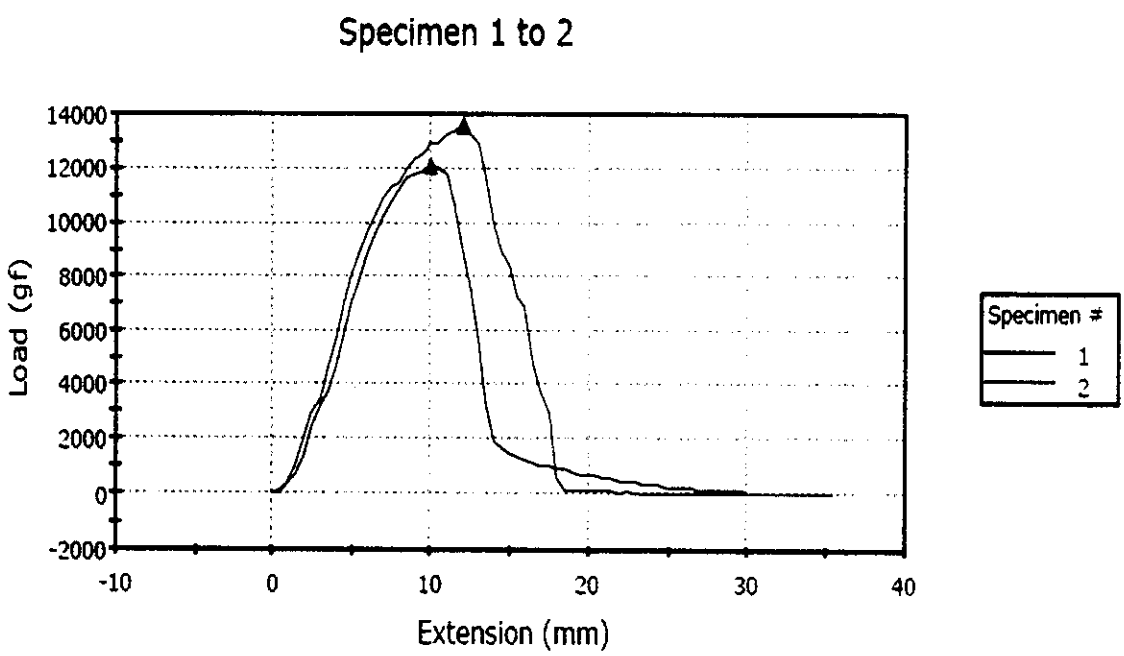
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	12972.87	8.50	129.73	8.50
2	10156.94	9.50	101.57	9.50

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	11.33	42.41
2	12.67	33.20

Tensile Strength of untreated Silk sample

ATM Silk

Text Inputs: Specimen label	ATM Silk
Test: Rate 1	300. mm/min



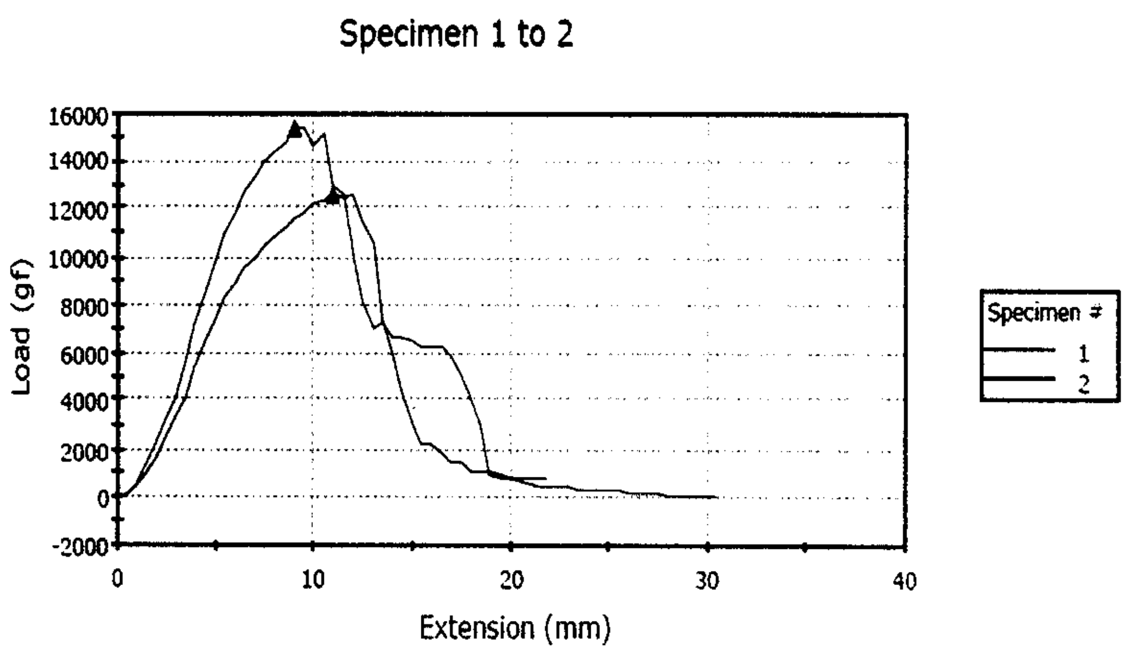
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	13597.35	12.00	135.97	12.00
2	12077.76	10.00	120.78	10.00

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	16.00	44.45
2	13.33	39.48

Tensile Strength of Air treated Plasma Silk sample

Oxygen Silk

Text Inputs: Specimen label	Oxygen Silk
Test: Rate 1	300. mm/min



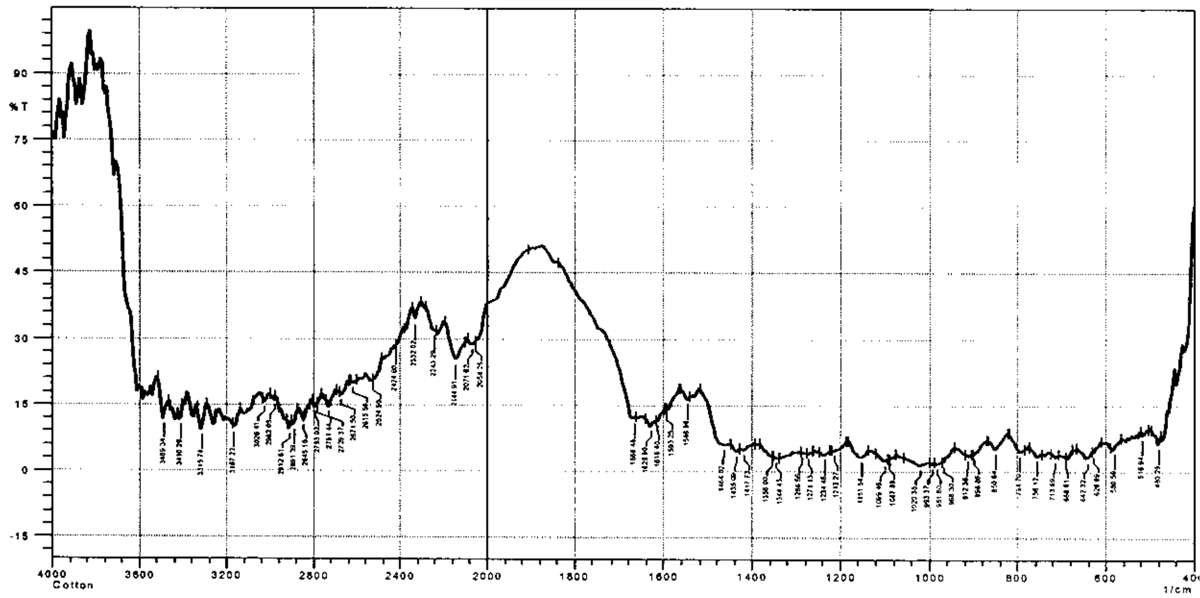
	Maximum Load (gf)	Extension at Maximum Load (mm)	Tenacity at Maximum Load (gf/tex)	Tensile extension at Maximum Load (mm)
1	15342.93	9.00	153.43	9.00
2	12620.47	11.00	126.20	11.00

	Tensile strain at Maximum Load (%)	Tensile stress at Maximum Load (N/mm^2)
1	12.00	50.15
2	14.67	41.25

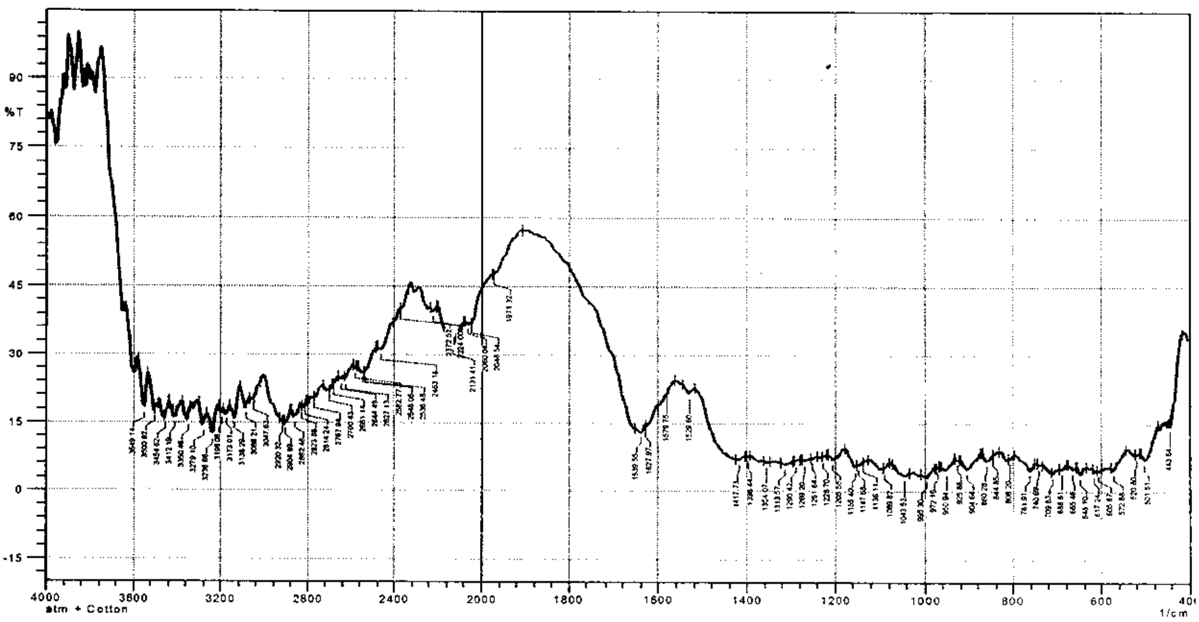
Tensile Strength of Oxygen treated Plasma Silk sample

Fig. 4.5 FTIR Spectra Analysis

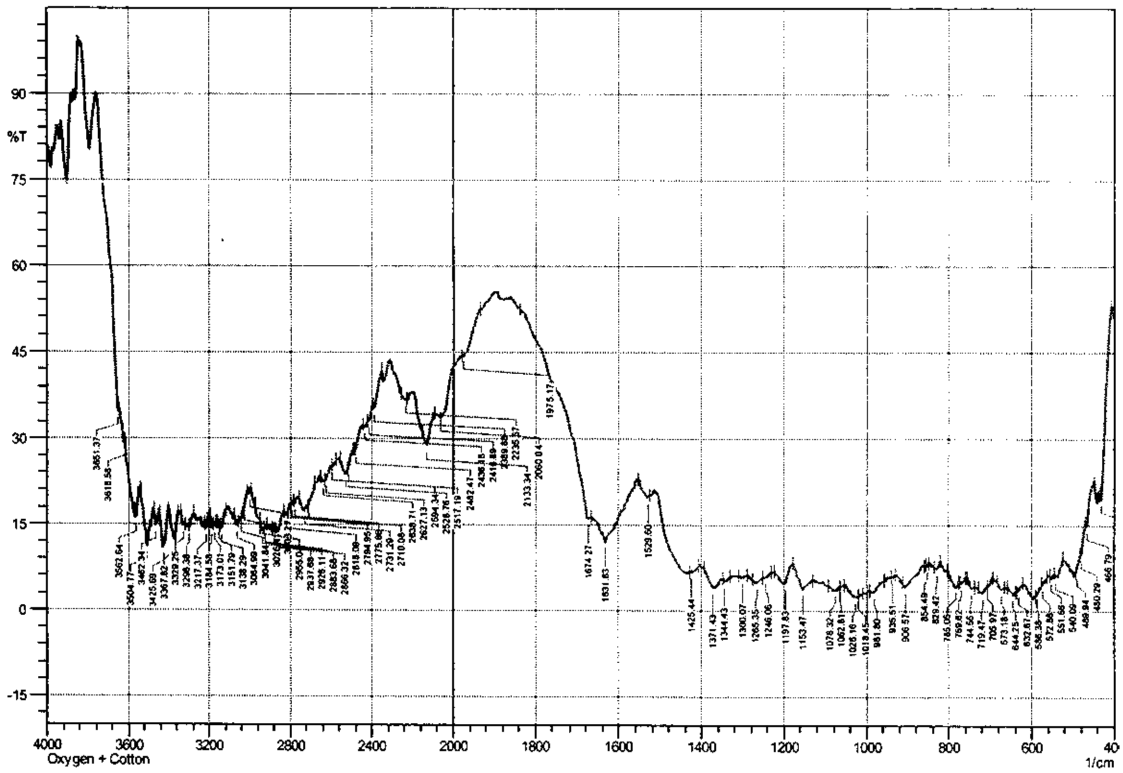
FTIR Spectra of untreated and plasma treated samples



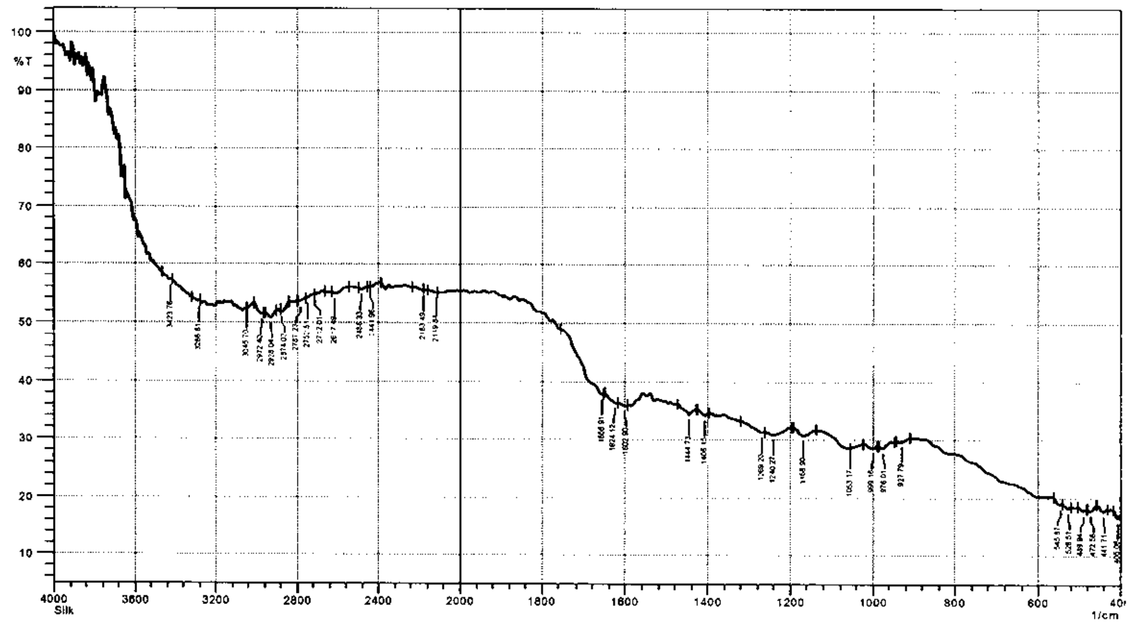
FTIR Spectra of untreated cotton Sample



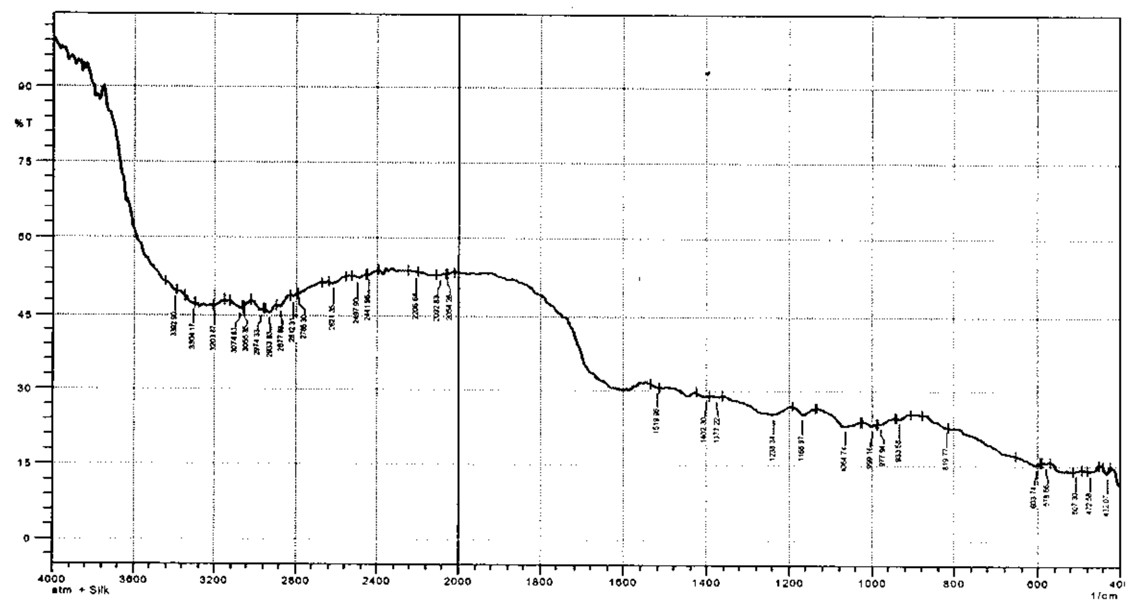
FTIR Spectra of Air treated Cotton Sample



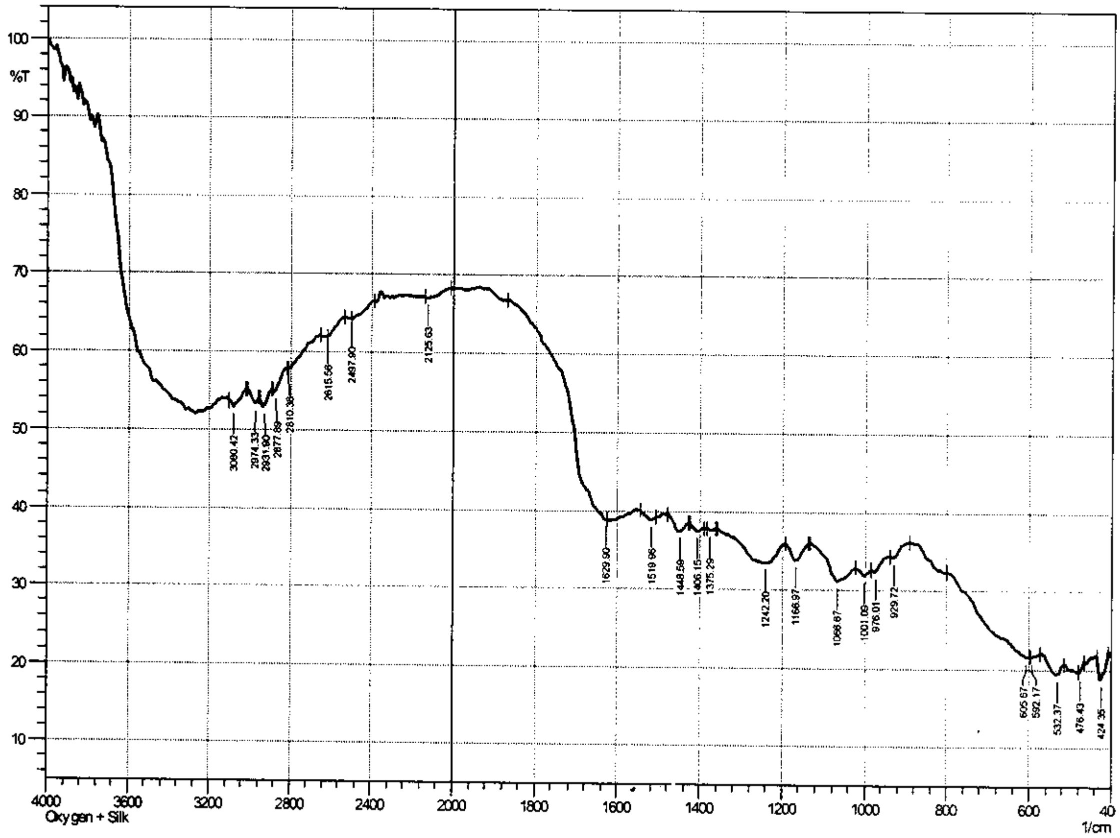
FTIR Spectra of Oxygen treated Cotton Sample



FTIR Spectra of untreated Silk Sample



FTIR Spectra of Air treated Plasma Silk Sample



FTIR Spectra of Oxygen treated Plasma Silk Sample

CHAPTER 5

CONCLUSION

From the study carried out, the following conclusions can be derived.

1. Plasma treated samples exhibited surface morphological changes and increased surface roughness by the etching effect of plasma active species bombardment in the cotton and silk surface
2. Plasma treatment causes oxidation and exchange of ionic groups depends on the gas plasma used for treatment. These groups are responsible for providing better wettability and dyeability in substrate.
3. Plasma treatment, results in insignificant weight loss in the substrate.
4. Plasma treatment shows significant improvement in wettability of cotton and silk fabrics which was confirmed by absorbency evaluation through vertical wicking height measurement and drop test.
5. Due to the etching effect of plasma, fine pores are formed on the surface of the fibre which leads to increase in colour strength of the dye due to accumulation of dye particles on these spots.
6. Plasma treatment shows significant change in tensile properties. For cotton tensile strength has significantly decreased and for silk tensile strength has increased.
7. Thus plasma treatment on cotton and silk fabric shows affinity for water and natural dye.

CHAPTER 6

SCOPE FOR THE FUTURE

- The usefulness of plasma treated samples can be evaluated for dialysis micro/ultra filtration, medical and hygiene applications such as top sheets in diapers etc.
- Various plasmas with inert gases as nitrogen, argon, helium and reactive gases such as ammonia, fluorocarbons, carbon di oxide, etc. can be studied independently and combinations to obtain different surface changes and applications.
- Surface modifications, in order to impart soil repellency, flame retardency and anti-microbial kinds of effect, can be studied with the help of plasma polmerisable gases such as acrylic acids, organo silaxanes, etc.,
- Effect of flow rate of gas, break down voltage for gases in different plasma methods can be investigated

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