STUDIES ON EFFECT OF PLASMA TREATMENT ON POLYPROPYLENE NONWOVEN FABRICS

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BONAFIDE CERTIFICATE

Certified that this project report titled **"STUDIES ON EFFECT OF PLASMA TREATMENT ON POLYPROPYLENE NONWOVEN FABRICS"** is the bonafide work of Ms. **S. KANJANA** (Reg. No. 71206507004) who carried out the project work under my supervision. Certified further, that to the best of my knowledge the work reported herein does not form part of any other project report or dissertation on the basis of which a degree or award was conferred on an earlier occasion on this or any other candidate.

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ABSTRACT

Plasma treatments are gaining popularity in the textile industry due to their numerous advantages over conventional wet processing techniques. Polypropylene offer an excellent combination of cost, processability and performance. However, the inherent hydrophobic nature of polypropylene makes it unsuitable for some fiber/non-woven applications, where properties like aqueous absorption and wettability are required. A hydrophilic effect can be achieved through the application of topical hydrophilic treatments. However, while these treatments achieve an initial hydrophilic effect, they are not durable. In this study, Polypropylene spun bonded nonwoven fabrics are treated with air, oxygen and nitrogen plasmas with varying conditions of treatment such as distance between electrodes, time of treatment, pressure & types of electrodes and the treated samples are evaluated for changes in physical and functional characteristics in terms of absorbency, wickability, surface energy, moisture content, tensile strength, discolouration, durability of the effect obtained and dyeablity. Taguchi method , a proven advantageous method for designing the experiments over the other experimental designs, is used in this study with, distance between electrodes, time of treatment, type of gas plasma & pressure, as control factors and type of electrode & areal density, as noise factors.

Plasma treatment causes physical and chemical modifications in the substrate. The changes in the morphology causes increased tensile strength due to increased fibre-to-fibre friction. Chemical changes in the form of implantation of functional groups in addition to physical changes helps in obtaining much improved moisture content, wettability and dyeability with disperse and reactive dyes. The improvement was confirmed by absorbency by drop test, wicking and surface energy measurements. Distance between electrodes was found to play a dominant role than the other factors. Among the gas plasmas studied, air plasma was more effective in getting the desirable functional properties. Some degree of yellowing and loss in whiteness is inevitable in the plasma treatment. Though plasma treatment effect was found to be decaying with ageing period, it stabilises after a period of eight hours with significantly improved hydrophilicity in the fibre.

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(S.KANJANA)

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LIST OF SYMBOLS AND ABREVATIONS

FTIR	Fourier Transform Infrared Spectroscopy
λ_{max}	Wavelength of maximum absorbance
eV	Electron volt
RF	Radio Frequency
kHz	kilo hertz
UV	Ultra Violet
PP	Polypropylene
DFE	Directional Friction Effect
PET	Polyester
S/N ratio	Signal to Noise ratio
AATCC	American Association of Textile Chemists and Colourists
BS	British Standards
ASTM	American Society for Testing and Materials
PVA	Polyvinyl Alcohol
CRE	Constant rate of Elongation
ANOVA	Analysis of Variance
GSM	Grams per square metre
SEM	Scanning Electron Microscope
Cu	Copper
Al	Aluminium

CHAPTER 1

INTRODUCTION

1.1 PLASMA TECHNOLOGY

In various processes involved in the manufacturing of textile materials, chemical processing often remains 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, solvent 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 fibres, man made fibres and fabrics. Plasma treatment offers eco-friendly, water-free processes in many cases, to the existing range of various wet processing methods. Plasma processing methods have been explored in improving the spinnability of cotton yarn, desizing and scouring of cotton and man made fibres, dyeing and printing of cotton and polyester fibres 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 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 ions, electrons, atoms and molecules (Jhala,2005; Subbulakshmi, 1998). A gas requires 1 – 30 eV per particle to change its state to plasma, which is much higher than that required for solid - liquid or liquid - gaseous transitions. In the gaseous substance, if the 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 with an equal density of

positive and negative charged particles. Presence of free electrons makes 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.

1.3 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 combinations of the gases 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 in the following Table 1.1. Plasma surface treatment can be effected by either active or passive plasma exposure (Roth, 2001).

Table 1.1 Types and Characteristics of Plasma

Types of Plasma	*Characteristics*
Glow Discharge, Formed by applying direct current (DC), microwave (50Hz), RF (40 kHz, 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 consists of small lightning 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	Lightning type discharges are created Provides uniform treatment

* - OAUGDP – One Atmospheric Unit Glow Discharge Plasma

1.4 CHARACTERISTICS OF PLASMAS

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

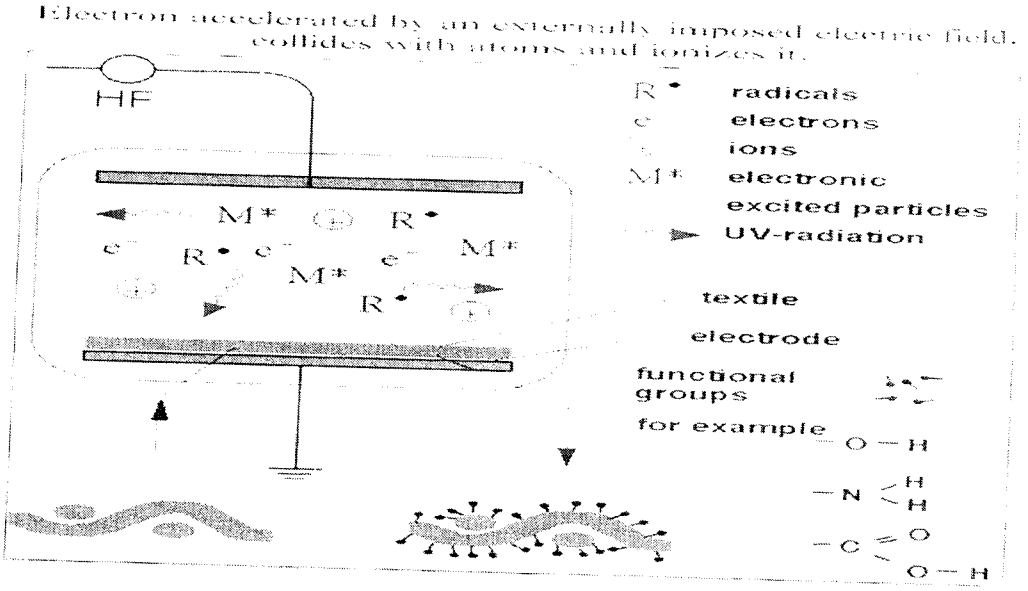

Fig. 1.1 Plasma Chemical Reactivity

The two broad categories, on the basis of temperature, of plasmas include hot plasmas and cold plasmas. In plasma, the electron temperatures may reach 10^5 – 10^6 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 3.56 eV (1 eV = 11604 K) though 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, liquids 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 plasmas 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 textile materials results the changes in wettability, wickability printability etc.

Table 1.2 Plasma Applications 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 charges	Wettability, wickability, printability, dyeability, washability, functional applications
Alteration of Cohesive Properties, Increases surface-to-surface contact cohesion, increases 3D cross linkages among fibres	Crosslinking of polymers, washability, handle modification
Alteration of Adhesive Properties, Adhesion, surface energy is increased by active species plasma. Adhesion results from combination of mechanical, chemical, electrostatic, permeation, diffusive, surface roughness, micro-profile contributions	Painting of surfaces without Volatile organic chemicals, composite structures, medical applications
Alteration of Electrical Characteristics, Increases surface conductivity, embeds or imparts electrostatic charges and deposits on the surface	Antistatic finish, charging and discharging action in photocopying, filtration, breathing masks, charge 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

1.5 ROLE OF TEXTILE STRUCTURE AND SOME PROCESS CONDITIONS

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

Table 1.3 Parameters of glow discharge processes

Parameter	*Variations*	*Main effect*
Apparatus parameters		
Reactor type	Tube or bell jar	
Frequency	0 (DC) -10^{10} Hz	Energy density of plasma
Electrodes		
- Placement	Internal or external	Internal electrodes affect chem.
- Coupling	Capactively or inductively	Composition of plasma
- Shape		Homogeneity of plasma
- Surface area ratio		
Pumping		Only important for internal electrodes
- Base pressure	10^{-10} – 10^{8} mbar	
- Capacity	2-250 m^3/h	Cleanness of system
Magnetic field		Residence time
		Confinement and homogeneity of plasma
Discharge parameters		
Gas		Type of process
Substrate		Etching or deposition
Flow	0-1000cm^3/min	Residence time in plasma
Pressure	10^{-2}-10mbar	Etching/deposition rate
Power	1-1000W	Energy density of plasma
Biasing	self bias-500V	Energy density of plasma
Substrate temperature	77-500K	Etching rate
		Surface composition
Procedure parameters		
Cleaning of reactor	Chemical, thermal or Plasma Cleaning	Cleanness of system
Evacuation time	1 s-several hours	Intensity of modification
Treatment time	0.01 s-several hours	Surface chemistry
Quenching gas		

The presence of impurities in the raw fibres, additives present in the yarn and fabrics also intervene during the plasma processing.The mean free path of gas particles, typical distances in the fabric structures like inter-fibre distance and inter-yarn distances have to be considered in the plasma processing (Poll,2001; Rashidi,2004) .The ion bombardment action on fibres can reveal the detail of surface and internal structures of fibres 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 on the surface of all single fibres in the whole fabric, the modifying particles must gain 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 treated. In the low pressure regions (P < 1 mbar) the mean free path in the gas phase 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 happen with other gas particles and reduce the life time of the radicals. The particles do not reach the reaction sites inside the voluminous fabric. A process pressure between 1 and 100 mbar turns out to be an optimum value for plasma modification of textile materials through out the thickness.

1.6 POLYPROPYLENE NONWOVENS

Polypropylene (PP) nonwovens find its applications in many fields. Melt blown PP is used as an absorbable dressing material in medical textiles. PP as a facing material coupled with an absorbent substrate and PP non-woven orthopedic cushion bandages are used under plaster casts and compression bandages for padding and comfort. Because of its excellent acid, alkali resistance and less cost, it has been used widely as filter fabrics. Especially in liquid filtration, needle punched, adhesive

bonded, impregnated and calendered non-wovens are used. PP non-wovens are used in clothing, for example, anoraks in bed covers and coverlets and in furniture in padding. This padding improves heat insulation and offers greater comfort where important criteria are fatigue strength is of care. Some of the major application of PP non-woven fabrics are given in Table 1.4

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Table 1.4 Applications of polypropylene fibre in industries

Applications	***Fibre Grade***	***Industry***
Cigarette filter	Stable fibre 3 denier	Cigarette
Technical filters	Stable fibre 5 denier, needle punched non-woven	Wet filtration, excellent, chemical resistance, used in water, milk, bear, paints, Coatings, petrochemicals, Pharmaceuticals, filtration
PP woven socks	PP film fibre, with 10-15% LDPE to reduce fibrillation and cost	Fertilizers, flour, Wheat, sugar, cement
Ropes and Twins	PP film and fibre	Agriculture
PP bale Warp	Spun Bonded PP	Synthetic fibres
PP tapes	High modules PP obtained by increasing draw ratio	Construction material like asphalt and concrete
PP construction / Industry fabrics	Filling grade and stable fibre	Construction material like asphalt and concrete
Substrate fabrics	Non-woven needle punched 3-4 denier stable fibres.	Furniture fabrics as backing material for visual furniture fabrics, it serves as reinforcement. Also used for wall covering, Luggage, table-clothes, tarpaulins, and automobile.
Outdoor Application	Heavy deniers containing stabilizers, UV absorber, etc.	Sports
Non-electric fuses for initiating explosives	PP slit film tapes	Mining Industry
Medical / surgical disposable fabric	PP stable fibre non-woven, Face masks, spun bond / melt blown / spun bond top sheets in diapers	Hospital

1.6.1 The Limitations of Polypropylene

Polyolefins offer an excellent combination of cost, processability and performance. However, the inherent hydrophobic nature of polypropylene makes it unsuitable for s ome fiber/non-woven applications, where properties like aqueous absorption and wettability are required.

A hydrophilic effect can be achieved through the application of topical hydrophilic treatments. However, while these treatments achieve an initial hydrophilic effect, they are not durable and are essentially removed after exposure to an aqueous medium or "insult". These treatments also have certain limitations in the process--the quality of the treatment depends on the non-woven structure and the non-woven has to be dried after treatment. As a result, Polypropylene becomes less suitable for applications where there is repetitive or continuous exposure to an aqueous medium. Applications such as wipes must rely on the absorptive capacity of other fibers, or fiber blends, to quickly absorb and hold water. Existing hygienic products are not suitable for extended or repetitive use.

1.7 APPLICATIONS AREAS OF PLASMA IN TEXTILE INDUSTRY

- Plasma treatment is used as a step in the total textile production cycle. Examples improved 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 fibre surface, plasma processing is very energy efficient, Some special textile properties can only be obtained via plasma processing.

1.8 OBJECTIVES OF THE PROJECT

The broad objectives of the project are

- To modify the surface characteristics of polypropylene fibres through plasma treatment and to assess the related changes in terms of physical and chemical properties
- To convert hydrophobic polypropylene in to hydrophilic, in order to improve its wettability, dyeability so as to enable enlarged application areas of PP fibre

In the present work an attempt has been made to give plasma treatment to polypropylene spun bonded nonwoven fabric and to study the effect of the treatment on wettablity, moisture regain, discolouration, surface energy, functionalisation, dyeability, tensile strength and the permanency of the effect by aging study.
The main objectives of the project are:

1. To identify the scaling factors in the Plasma finishing that influence the treatment process significantly.
2. To identify the adjustment factors in the Plasma treatment those may be useful to modify the process to accommodate minor variations.
3. To identify optimum process conditions using statistical technique, in particular, Taguchi methods to include the effects of noise factors in the experiments
4. To arrive a set of robust design parameters to achieve the uniform Plasma treatment efficiency, regardless to the noise factors.

CHAPTER 2

LITERATURE REVIEW

2.1 PLASMA PROCESSING FOR YARN MANUFACTURING

Attempts have been made, earlier, to assess the effect of plasma processing of fibres and the subsequent influence in the yarn manufacturing process in the short staple spinning system using cotton fibres. The spinnability, strength, twist requirement for required strength, end breaks in spinning and weaving preparation have been analysed as the measure of process efficiency (Abbot,1977) . Corona treatment of (CO_2) cotton card sliver, using multiple ends with 450 W power, shows increased yarn tenacity (linear density-21 tex) from 116 to 142 mN/tex (CSP-1970 to 2330) with no significant difference in elongation at break and unevenness.

2.2 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 fibres 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, removal of starch and 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 at 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 analyse 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-polymerising 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 8.0% in cotton and wool. The dyeing rate also increases for both after plasma treatment.

2.3 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 shows the similar values as that of bleached fabrics. Formation of carboxylic 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 hue mainly due to differences in the initial un-dyed substrates.

Hydrophilisation has been carried out on cotton using radio 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 2.1

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, from the precursor.

Table 2.1 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 Generates greater changes in fibres
	Hydrophobicity (RF Plasma)	CF_4, C_3F_6 ,100, 300W, 50& 75 mTorr for CF_4 50,160W, 50 – 150 mTorr for C_3F_6	Lower results for CF_4 than C_3F_6 C_3F_6, polymerises by plasma and plasma inducing methods. CF_4 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 cleft created. Subsequent enzyme treatment enhances handle and dyeability
BOPP *	Increased surface energy and wettability (RF Plasma)	O_2, Low pressure (1-10 Pa) Low temperature Glow Discharge	Formation of new groups like –OH, -C=O. Surface energy increased from 24 to 71 mJ/m^2 ,Creation of surface roughness, loss of hydrogen, appearance changes due to low reflectance
Nylon	Water Repellency (RF Plasma)	CF_4 , Power 100 W and 4 Pa	Absorption of F atoms on surface, Air resistance and glossiness improves

* BOPP – Biaxially Oriented Polypropylene film

2.4 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 depending 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 fabrics shows reduced strength but this also results in reduction in weight loss in subsequent cellulase treatment. However, oxygen plasma onto wool improves its strength and higher rate of weight loss in subsequent protease treatment. As the plasma treatment is carried out in dry conditions, the fibres are not swollen and changes on surfaces are restricted. The DFE is reduced but fibre / fibre friction increases. Surface roughening caused by plasma treatment of wool results in harsh handle.

Plasma treatment causes ablation of fibre 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 change in tenacity and elongation at break values for both PET and cotton fabrics. The voids and cracks developed in PET also aid penetration of moisture. Grafting methods employed using plasma treatment does not alter the mechanical strengths, probably due to attachment of monomers in the surface, not in entire bulk. Moisture content of the hydrophilised polyester fabrics increases to 2%.

Plasma treatment offers effect which are often decaying in nature i.e. ageing or durability effect. The effects produced by plasma decrease 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, hygiene products.

2.5 PLASMA TREATMENT FOR NYLON FIBRES AND FABRICS

The various approaches on plasma treatment of polyamide fibres such as nylon, wool and silk have been reviewed by Saravanan et. al. (2007). An attempt has been made to study the effect of non-polymerising gases like oxygen, argon, tetrafluoromethane on Nylon 6 woven fabrics using glow discharge generator with a discharge power of 100W and 4 Pa of pressure. Prolonged treatment results in ripple-like patterns oriented perpendicular to the fibre axis. Oxygen plasma gives more distinct effects than argon plasma due to its inertness.

The effect of plasma treatment on low stress mechanical properties of nylon fabrics have been analyzed with reference to the control fabrics. This results in changes in various properties to different extent. Removal of uneven surfaces at the beginning that results in smoothness, however, the prolonged treatment causes ripples for both oxygen and argon plasmas. Surface roughness voids, and spaces created in the plasma treatment increase the air trapped in the fabrics and yarns and the trapped air act as good insulation medium and prevent heat loss of fabrics.

2.6 LOW TEMPERATURE PLASMAS 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 mtorr 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 leads to water repellency.

Fibroin of the silk treated with low temperature plasma using oxygen, tetrafluoro methane gases to analyse the immobilization of the enzymes shows successful immobilization of alkaline phosphatase and improved activity with both the gases.

Surface flutes are also observed in the surface of the silk sutures treated with low temperature oxygen plasma with increased loading capacity. The etching effect obtained in the sutures also leads to reduction in weight of the fibre. On contrary to other findings a reduction in the crystallinity has been observed in this study.

2.7 PLASMA TREATMENT ON WOOL FIBRES AND FABRICS

Extensive work has been carried out analyse the effect of various low temperature plasmas 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 alternatives for wet chlorination for improving wettability and shrink resistance. Conversion of disulphide to acid is almost 90% which is higher than UV or ozone oxidation of the wool fibres. Nitrogen plasma introduces $-NH_2$ groups in the fibres which become dye sites on wool, increasing dye absorption. Low temperature plasma using oxygen, nitrogen and mixture nitrogen and hydrogen result in the formation of Bunte salt, cysteic acid due to cleavage of disulphide linkage.

Detailed analyses on wool fibres, yarn and fabrics have been carried out using glow discharge plasmas to analyse the effects like surface changes, changes in chemical compositions and various physical properties. The effect of glow discharge plasma on wool fibres on spinnability and the subsequent changes in yarn properties has been studied extensively. Fibre-to-fibre frictions increases 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 directions, mainly due to decrease 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 contact 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 hydrogen (25%) and nitrogen (75%) plasma result in reduction in half time of dyeing and increase in the final exhaustion.

In the case of dyeing regular dyeing process poor wettability of untreated fibres show 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 fibre surface provides a path way for dyestuffs to diffuse into the fibre easily. Similar reflectance curves are obtained for both the sample show the similar depth of dyeing. Dry rubbing fastness showed improvement slightly while no such things in wet rubbing. Wash fastness and fastness to perspiration increases compared to untreated fabrics.

The presence of scale structure on wool fibre surface introduces a number of problems such as felting and surface barrier to dye stuffs. The shrinkage in fabric takes place due to relaxation, consolidation and felting shrinkages. The dimensional changes obtained in the warp are higher than weft in terms of decrease in shrinkage. Consolidation shrinkage is higher for untreated fabrics than treated ones. Felting dimensional change reduces from 9.6% to 1.1% for the treated fabrics in warp direction and 12.3% to 1.5% in the area shrinkage. This happens mainly due to etching effect which decreases the natural shrinking tendency. Combination of oxygen low temperature plasma and silicone polymer treatment improves the dimensional stability more than silicone 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-fibre frictions play major role in tensile strength of fabrics which increases with low temperature plasmas due to surface roughness. Cleavage of disulphide linkage

appears to make the scales more elastic, besides increase in the frictions and modify the elongation at break.

Plasma treatment offers effect which are often decaying in nature i.e. ageing or durability effect. The effects produced by plasma decrease 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, hygiene products.

2.8 PLASMA TREATMENT ON POLYESTER

Several techniques have been used to tailor the properties of polyester surfaces which are reviewed by Nalankilli et. al.(2007).

2.8.1 Surface Modification

Surface modification such as ablation / etching by breakdown of weak covalent bonds through bombardment by high energy particles or surface cross linking between molecular chains can be achieved using inert gas plasma. Reactive gases can be used for breaking the weak bonds and converting them into functional groups such as carbonyl, carboxyl, hydroxyl etc

Glow discharge plasma technique (of a few 100 V to a few kV) is particularly useful for fictionalization of polyester surface because with these techniques, it is possible to specifically modify the outermost surface. Corona (10-50 kV) treatment is also used. It is a simpler, more practical method than any other chemical / physical methods. Samples are also treated only under atmospheric pressure.

Table 2.2 Various plasma treatments and their effects on Polyester

Treatment	*Effects*
Low temp plasma / O_2, N_2, H_e, A_r, H_2 CH_4	Surface tension of PET increased, O, N content in PET increased. Increase in H bonding force, increased wettability. Decreased surface tension, decreased oxygen content, reduced hydrogen bonding force, reduced wiettablility
Carona discharge / Acrylic acid (grafting)	Improves wettability
Electrical discharge RF plasma / $SiCl_4$	Silicon & oxygen containing groups grafted enhances surface roughness, increased oxygen content & lower carbon content, hydrophiliciity improved
RF plasma / O_2	Oxygen containing functional groups increased (Co, CooH, OH)
Low pressure RF / N_2	Contact angle increased, polar groups introduced on surface, chain scission takes place, unstaple surface effect, wettablility decays with time, prolonged exposure and / or high power level ablate the surface, differential etching in amorphous and crystalline regions.
Low pressure / RF / Air and O_2	Surface oxidation occurs, intensify the initial water uptake, decreased surface activity due to cross linking reactions, wettablility lower than N_2 plasma
Carona Discharge / Irradiation	More hydrophilic property, durable, improved dye uptake ratio and dyeing speed, shortened dyeing time, improved affinity with starch enabling sizing with modified starches.
Atmospheric plasma / PET Film / inert gases	Improved adhesion property
Atmospheric pressure / He / Ar or Acetone / Ar	Increased wettablility, He / Ar more effective than argon plasma
Atmospheric plasma / N_2 and Vinyl tri ethoxy silane (organic – Inorganic precursor)	Barrier properties imparted
Vacuum plasma / dichloromethane	Enhanced moisture content and dyeability, other properties not affected
Vacuum / Ar / perflouro acrylate	Water repellant properties
Vacuum plasma / Ar / Acrylic acid	Increase wettablility, soil resistance better colour strength (post plasma better than insitu)
Vacuum plasma / Hexamethyl disilane / tri's (trimethyl slyloxy) vinyl silane	Good dielectric properties, high thermal stability, scratch resistance, increased colour intensity
Carona discharge / acrylic acid (Graft co polyester)	Improvement in antistatic property

Glow discharge / Acrylic acid & Acrylamide	Subsequent ionization improves antistatic property remarkably
Carona / glow discharge / O_2	Adhesion property improved, metals interact with hydroxyl, carbonyl and ester groups after plasma treatment
Glow discharge / O_2	Adhesion with aluminium improved
Glow discharging / NH_3, O_2	Adhesion with aluminium improved
Atmospheric processes / N_2	8 fold increase in adhesive strength of monofil / epoxy
SO_2 + O_2 and $N_2^+H_2^+$ He	Introduction of polar functional groups, Increased dyeability with acids dyes, Decreased dyeability with disperse dyes, Increased crystallanity
Air	Crystalline phase affected, Reduction is dyeability, Etching away of dyeable macromolecules
O_2 / Glow Discharge	Crystyllanity increased Decrease in saturation dye uptake
ST Plasma / $SiCl_4$	Improved dyeing property
Atmospheric Plasma / PC Blend	Light fastness improved
Chlorohydrocarbon	Flame resistance
Trifluro Ethene	Bio compatibility / Vascular Grafts
Allyl Amine	Blood compatibility
Allyl Alcohol	- OH, C = O Functional Groups introduced
Allyl Amine	Introduction of NH_2 groups
Argon	Barrier coatings, prevention of plasticizer leakage
Air	Soil resistance
Ar / N_2 / Air	Wettability improved
N_2O / Ar	C = O: introduced
CF_6, C_2F_4 / H_2 Cold plasma	Water repellency
H_2 / RF, low pressure Glow Discharge	Barrier properties, Oxygen content decreased

2.9 PLASMA TREATMENT ON POLYPROPYLENE

Polypropylene has a very low value of the surface free energy (approximately 20-25 mJ/m^2. Due to low surface energy, polypropylene has very weak hydrophilic properties. In many industrial applications, there is a need to modify the polymer surface with keeping their desired bulk properties unchanged. Chemical activation of the surfaces is the most often used method for their activations; however, the ecological requirements force the industry to search alternative environmental safety methods. The surface modification of polymers by low temperature plasma appears as one of the most prospective and cheap solutions.

Plasma treatment of Polypropylene proceeds by a free radical mechanism that introduces a wide variety of oxidized functional groups onto the surface of the treated polymer. These oxidized functional groups may include C-OH, C=O, COOH, C-O-C, epoxy, ester, or hydro peroxide and they are responsible for the change in the polymer surface properties.

Plasma treatment of Polypropylene has a great degree of impact on surface morphology and fabric wetability. Plasma treatment results in increase in surface energy of Polypropylene and confirming the presence of polar groups on the surface with decrease in contact angle (Su,2004). (He - O_2) plasma, makes the surface rough, rippled and only slightly affected. When plasma treatment was given at low temperature for few minutes with CO_2 gas and CO_2 – O_2 mixture, makes the Polypropylene webs wettable (Tsai, 1997). [N_2 + H_2 + He] significantly improves the dyeability of Polypropylene spun bonded non-wovens webs (Zhang,2000).

Surface modification using Dielectric Barrier Discharge [DBD] plasma changes the hydrophobic character into an increasingly hydrophilic nature which was confirmed by the decreased contact angle from 90° to 55°. Atmospheric glow plasma treatment with reactive gases like He, Ar, Air, CO_2 of Polypropylene melt blown non-woven also increases the wettability.

The functionalization with the bonds such as C-H, C-C, C-N, C-O-C and C-O-H on the surface of the Polypropylene along with increased surface energy was observed in the atmospheric pressure plasma treated with gasses such as N_2, H_2, NH_3 and mixture on Polypropylene plates (Bourbigot,1999). Plasma treatment with polymerisable monomers causes significant surface modification in Polypropylene. When such treatment was given with O_2 gas and Acrylic acid monomer, it was found that surface etching followed by polymer coating takes place and enhances the electrochemical properties of Polypropylene (Basarir,2005). Generation of Carbonyl,

ester and OH groups with increased oxygen content has also been confirmed by Denes. et al (1997).

Plasma polymerization / grafting on Polypropylene fabrics with Acrylonitrile (Sarmadi, 1993), Hexamethyldisoloxane (HMDSO) (Sarmadi, 1995). Melamine and Urea (Ganapathy, 1996), have been studied by various researchers. The structural changes and functionalities generated has resulted in achieving certain desirable properties in Polypropylene.

Grafting with acrylonitrile cold plasma leads to improved water absorption and dyeing properties, with HMDSO using RF cold plasma, lowers the water uptake value with increased contact angle. Several methods for changing the wetability of Polypropylene using plasma technology has been reported in literature. Corona discharge plasma is reported to incorporate oxygen containing functional groups such as Hydroperoxide, ROOH, ions and neutral functional groups such as RCOOR, R_3COR, R_2CO, which helps in improving wettability and adhesion (Tsuhiya,1998).Besides improving the wettability, corona treatment also improves the antistatic and friction properties of spin finished Polypropylene (Havtojarvi,2000). Polypropylene non-woven filter webs were used for Oxidative plasma treatment and the treated samples were tested for wetability using liquids with a range of surface tensions and also the filtration time for fixed amounts of water determined. Reference materials without plasma treatment can only be wetted with liquids with surface tension <35 m N/m without treatment, no water can pass through the Polypropylene-web without applying a high pressure Polypropylene oxidative plasma treatments can be very beneficial in order to improve wetability and increase the surface tensions. However the treatment conditions should be selected with utmost care especially since selecting conditions that are too aggressive will result in a lowering of the positive effects (1997).

Atmospheric plasma with fluorocarbon gas at RF-5 KHz given on non-woven fabric makes the fabric more hydrophobic due to considerably large amount of

pressure in the water column (388.87 mm). It provides good barrier against blood and water and can even provide a barrier against microbes, which makes it a better finish for surgical gowns than commercially available fabric (Ralkowski, 1997).

Yousefi et al. (2003) have studied the surface modification of biaxially-oriented polypropylene (BOPP) films by low temperature, low pressure oxygen plasma treatment. SEM shows CH_2 groups of Polypropylene were reduced by hydrogen loss and polymer degradation due to oxygen plasma etching. The spectra represented new functional groups assigned –CO and –OH absorption bands. Creation of polar groups such as hydroxyl and carboxyl on the surface increased the surface energy and wetability. Marian McCord et al.(2002) treated atmospheric plasma on Polypropylene, which alters the surface chemistry and morphology of Polypropylene . Yoon J. Hwang et al. (2005) studied from the spectroscopy analysis, reveals that the surface oxidation by the formation of hydrophilic groups enhances the surface wetability of the Polypropylene non-woven fabrics with He atmospheric pressure glow discharge plasma treatment. Plasma treatment increase fibre to fibre friction, playing an important role in enhancing a tensile strength, low stress mechanical properties and air permeability.

Hocker (2002) observed that the barrier discharge or corona treatment of Polypropylene fibres significantly increases the hydrophilicity of the surface, the contact angle of water being decreased from 90° to 55°. Even after two weeks, a sustained effect was observed, the contact angle of water being 60°.Plasma treatment of Polypropylene and indulin kraft lignin can also be carried out in specially designed electrode-less plasma reactor aiming at improvement in strength of the composites made from these materials and efficient surface modification, thus avoiding long reaction times and use of large volume reactants. This ensured effective implantations of reactive functional groups on lignin surface (Toriz, 2004) Plasma treatments showed promise for producing hygroscopicity in fibres, altering degradation rates of biomedical materials (such as sutures) and depositing anti real

coatings. Plasmas are used to provide clean hydrophilic surfaces and to change the state characteristics and sterilize medical components (Kan, 1998). Tyan, et al.(2002) have utilized low temperature, high density microwave plasma to activate PP nonwoven structures followed by coupling agent inclusion and collagen immobilization for biomedical applications.

Plasma anti-microbial treatment of polypropylene non-woven fabrics for surgical gowns using fluorocarbon gas was found to have effect such as it does not alter weight, thickness, stiffness, air-permeability, breaking strength and elongation. The fabric also becomes water repellent with high blood and water resistant with inhibition for microbes growth. Oxygenated helium plasma was used to enhance initiation of graft copolymerization of glycidyel metha acrylate (Gawish,2006) in Polypropylene non-woven to synthesize biocidal fabrics proved to be antistatic, antimicrobial and insect repellent.

Qu fu wei et al.(2006) have found plasma treated Polypropylene non-woven treated with oxygen gas plasma improving the dynamic water absorption properties in which fibre fineness place an important role. Surface dielectric barrier discharge which find wide spread applications in ozone production, were used to hydrophilise light weight Polypropylene non-woven fabric the samples were treated with nitrogen plasma and the method was found to be used potentially in laminating, printing, metal plating lines etc. Oxygen and nitrogen functionalizing gasses were mixed to give low temperature atmospheric plasma treatment with a roll-to-roll system showed direct improvement in wettability tested by capillary tests (Rombola, 2006).

A book entitled "Plasma Technologies for Textiles" has just been published by Woodhead publishing, Cambridge, UK (Shishoo, 2007). Its publication is timely. It is by far the most comprehensive review of the subject to date and highlights the huge potential of gas plasma treatments for textile manufacturing and processing.

2.10 EXPERIMENTAL DESIGN

Taguchi's method replicates each experiment with the aid of an outer array that deliberately include the sources of variation that a product would come across while in service. Such a design is called a minimum sensitivity design or a robust design and the Robust Design method is called Taguchi method (Bagchi, 1993). To achieve the optimum design factor setting, Taguchi advocated a combination of two stage process in which the first step is related to the selection of robustness seeking factors and the second step with the selection of adjustment factors to achieve the desired target performance. The various stages in the experimental design has been dealt by many authors in the past (Istiaque, 2006; Kumar, 2006). In other experimental designs, uncontrollable factors are kept under observation during experimentation, whereas Taguchi methods include those factors in the experimentation to make the design a robust one in the form of S/N ratios. In robust design, one minimizes sensitivity to noise by seeking combinations of DP settings. The most appropriate S/N ratio can be selected depending upon the properties of interest (Table 2.3), for both scaling factors and adjusting factors.

Table 2.3 Signal-to-Noise ratio and its significance

S. No.	*Case*	*S/N ratio*
1	Target is the best	$S/N\ (\theta) = 10 \log_{10} (\bar{r}^2 / s^2)$
2	Small-the-better	$S/N\ (\theta) = -10 \log_{10} (y_i^2 / n)$
3	Larger-the-better	$S/N\ (\theta) = 10 \log_{10} [(1/yi^2) / n]$
4	Binary scale	$S/N\ (\theta) = 10 \log_{10} (p/1-p)$ p= proportion of good products

CHAPTER 3

MATERIALS AND METHODS

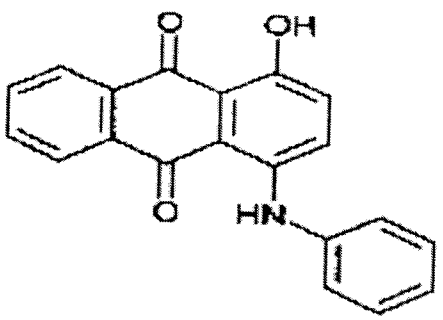
3.1 MATERIALS

3.1.1 Substrate

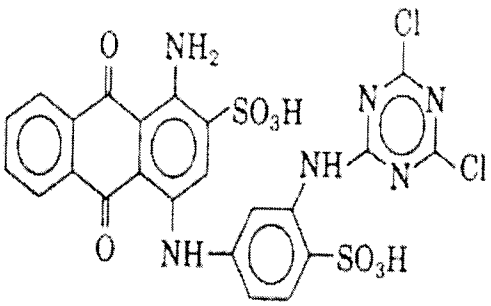
Spun bonded Polypropylene non-woven fabrics which had areal densities (GSM) of 80 and 100 supplied by M/s Tex source India, Coimbatore, were used in this study.

3.1.2 Dyes, Chemicals & Auxillaries

Cibacet Violet 2RI (C.I. Disperse Violet 27), Cold brand, Dichloro triazine Reactive dye-Procion Blue MR (C.I. Reactive Blue 4), dispersing agent, glacial acetic acid, non-ionic detergent, caustic soda, soda ash, wetting agent, Glabaurs salt were used in dyeing. Formic acid was used in surface energy measurements. Air, Oxygen and Nitrogen were the gases used for plasma treatment. The Chemical structures of the dyes used are given below.



C.I.Disperse Violet 27



C.I.Reactive Blue 4

Fig. 3.1 Chemical Structures of the Dyes

3.1.3 Plasma Reactor

A capacitively coupled atmospheric pressure DC plasma system with provision up to 3 kV and 500 mA 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. Aluminium and Copper electrodes were used independently in the present study. The schematic line diagram of the reactor is given below.

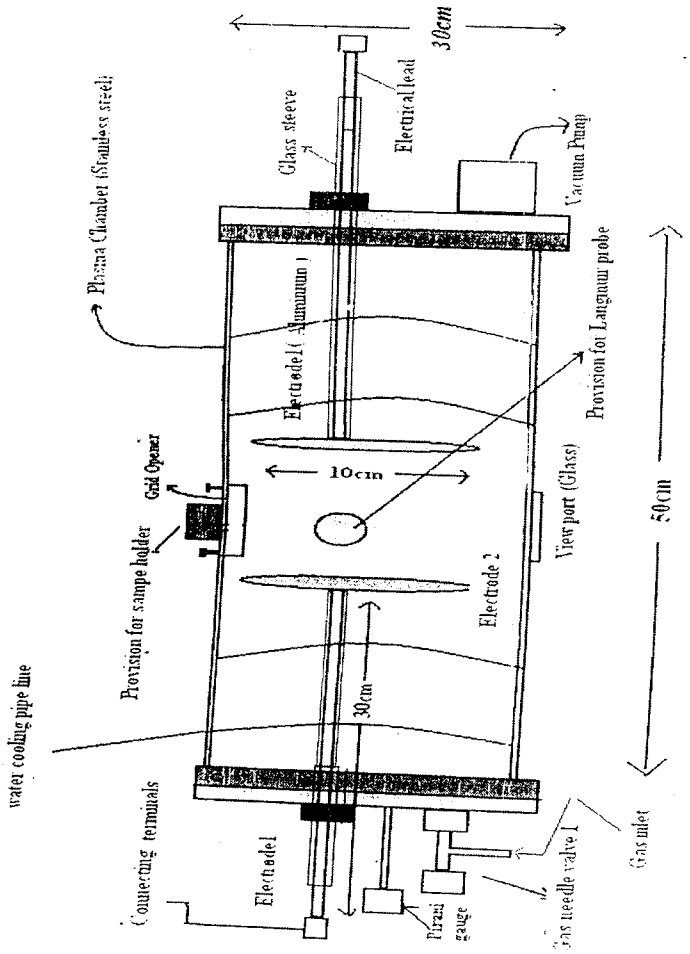


Fig. 3.2 Plasma Chamber

3.2 METHODS

3.2.1 Removal of Spin Finish

Fabric samples were washed with 1% non ionic detergent (Swanic 6L) solution at 70° C for 15 min., then rinsed with cold water and dried at room temperature.

3.2.2 Plasma Treatment

Samples of 20 x 20 cm placed in the middle of two parallel electrode plates, were treated with Air, Oxygen and Nitrogen plasma with electrodes such as Aluminum and Copper with spacing between electrodes of 3 cm, 5 cm, and 7 cm for various durations. Fabric samples were placed between the electrodes, the working pressure was adjusted with a rotary vacuum pump. Evacuation was carried out for a duration of 10 min. and then the power supply was switched on. The plate current was increased until glow discharge 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 sample 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. Gas flow rate and output power were maintained at 10 cc/min. and 300V respectively through out the study. Treatment time was varied from 30, 60 and 90 secs. and pressure was varied from 100, 200 and 300 mtorr.

3.2.3 Dyeing

3.2.3.1 Dyeing with disperse dyes

The treated and untreated fabric samples were dyed at boil for 30 minutes with material to liquor ratio of 1:40 using 0.5% on weight of fabric dye, 1 gpl dispersing agent, 0.5 gpl wetting agent and acetic acid to adjust pH to 5.5. The dyed samples were washed with 0.5% non-ionic detergent at 60°C for 15 min.; then rinsed

in cold water and dried at room temperature; then given reduction clearing treatment with 1gpl Hydros and 3 ml/l (30 % solution) of caustic soda in order to remove the unfixed dye present in the surface of the substrate.

3.2.3.2 Dying with Reactive dyes

The treated and untreated samples were dyed with cold brand reactive dye (dichloro triazine) at room temperature for 45 min. with material to liquor ratio of 1:40 using 30 gpl glabaur's salt 2 gpl soda ash and 0.5 gpl wetting agent. The dyed samples were soaped with 0.5% non-ionic detergent at boil for 10 min, rinsed and dried at room temperature.

3.2.4 Hydrophilicity

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

3.2.4.1 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 to disappear is measured and recorded as wetting time. Average of 5 readings is reported.

3.2.4.2 Wickability

The procedure used to measure the fabric wickability was based on the standard BS: 3432. A sample of 20 x 5 cm was vertically suspended 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. As the density of polypropylene is 0.91 g/cc which is less than the density of water (1gm/cc), a standard weight of 2 gm was attached at the tip of the sample with the help of a hook. 5 tests each for treated and

untreated fabrics were tested, and the average was reported. Higher the height of water climbed, better is the wicking property.

3.2.5 Etching Loss

Etching loss was calculated using analytical weighting balance with accuracy of 0.001 g. 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.

$$\%EtchingLoss = \frac{W_1 - W_2}{W_1} \times 100$$

where W_1 and W_2 are the weights of the untreated and plasma treated samples respectively.

3.2.6 Surface Energy

Surface energy measurement was carried out using the Critical Wetting Surface Energy method (Peter,1997; Kartick Samanta,2006). Surface energy of the fabrics was measured by using formic acid solutions of different concentrations. A drop of formic acid solution of a particular concentration was placed on the fabric and if the drop was absorbed by the fabric within 5 seconds, then surface energy of the fabric was considered equivalent to the surface tension of the solution. Formic acid and water were taken in different proportions and mixed together in order to get test solutions of different surface energies. The surface tension of formic acid is 37.67 dynes/cm and it is 72.8 dynes/cm for water.

3.2.7 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.

$$\%\text{MoistureContent} = \frac{W_C - W_D}{W_C} \times 100$$

where W_C and W_D are the Weights of conditioned sample and dry sample respectively.

After plasma treatment the PP fabric specimens were exposed to air at ambient temperature and weighed at different storage times for decay study.

3.2.8 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 scan after 20 scans to reduce noise effects in measurements

3.2.9 Yellowing Index

In order to measure the yellowing tendency of fabrics caused by the plasma treatment, treated and untreated fabrics were measured using ASTM E313 for Yellowness Index in Gretag Magbeth Computer Colour Matching quality control software.

3.2.10 Whiteness Index

In order to measure the change in whiteness of the fabrics caused by the plasma treatment, the treated and untreated fabrics were measured using ASTM E313 Whiteness Index (CIE) in the Gretag Magbeth Computer Colour Matching quality control software.

3.2.11 Fastness of Dyed Fabrics

3.2.11.1 Wash fastness

The dyed samples were tested for ISO 2 wash fastness following the AATCC Test method 61-2003. in a Launder-O-meter. The specimen size was 5 x 10cm and

was sandwiched between same size of white fabrics (for assessing the staining on white). The washing was done at 50°C for 45min using 5gpl non-ionic soap with material to liquor ratio of 1:50.

3.2.11.2 Rubbing fastness

The dyed samples were tested for wet and dry rubbing fastness following the AATCC Test method 8-2005 in a crock meter. The specimen size was 5x13 cm and crocked for 10 cycles at rate of 1 turn per second to slide the white fabric covered finger, back and forth 20 times. The assessment of grading was done by referring to grey scales (change in colour and staining on white) and also confirmed using the compute colour matching quality control software

3.2.12 Exhaustion

Measurement of absorbance at λ_{max} for the initial dye bath and residual dye bath after dyeing was measured in a Perkin-Elmer visible spectrophotometer. Measurements of dye solutions were conducted at room temperature and the percentage exhaustion was calculated as given below.

$$\%\text{Exhaustion} = \frac{A_i - A_r}{A_i} \times 100$$

where A_i and A_r are the absorbance values of the initial and residual dye bath solutions respectively.

3.2.13 Colour strength

Disperse and Reactive dyed samples were measured for the colour strength (K/S) at λ_{max} (wavelength of maximum absorbance) using Kubelka-Munk function, in Gretag Magbeth computer colour matching system.

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

Where R is the Reflectance factor, S is the Scattering co-efficient and K is the Absorption co-efficient.

3.2.14 Decay Study

In order to assess the stability of the plasma treatment the absorbency by drop test and moisture content were measured immediately after plasma treatment and after different periods of ageing (intervals) such as 2, 4, 6, 8, 18, 24 and 48 hours.

3.2.15 SEM Micrograph

Fibres 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 5.0 kV.

3.2.16 Tensile Strength

Tensile strengths 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 15 x 10 cm and the average of 5 test results for each samples was reported.

3.2.17 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.

3.2.18 Experimental Design

The effectiveness of plasma treatment is influenced by the process conditions such as treatment time, electrode material, pressure, distance between the electrodes, frequency of power, power source (DC/AC), voltage and the type of gas plasma, gas flow rate etc. The combination of parameters often decides the efficiency of the process. Inclusion of the noise factors related to fabric construction and process condition to find out optimum design parameters, analysis of variance (ANOVA) followed by the confirmation tests using the optimum values obtained in the study, provide robust design parameters that gives stable output performances.

The control factors (4 factors, 3 levels) and the noise factors (2 factors, 2 levels) selected for the study and L9 array template for experiments are given in Table 3.1, 3.2 & 3.3.

Table 3.1 Control Factors

Control Factors	*Levels*		
	1	2	3
Time (secs)	30	60	90
Pressure (mtorr)	100	200	300
Distance between the electrodes (cms)	3	5	7
Type of Plasma	Air	Oxygen	Nitrogen

Table 3.2 Noise Factors

Noise factors		*Levels*	
		1	2
1	GSM of fabric	80	100
2	Electrode	Copper	Aluminium

Table 3.3 L9 Array Template for Experiments

Noise Factor Repetitions					1	2	3	4
Expt. No.	Time (secs)	Pressure (mtorr)	Distance between the Electrodes (cm)	GSM	100	80	100	80
				Type of electrode	Cu	Cu	Al	Al
				Type of gas plasma	1	2	3	4
1	30	100	3	Air				
2	30	200	5	Oxygen				
3	30	300	7	Nitrogen				
4	60	100	5	Nitrogen				
5	60	200	7	Air				
6	60	300	3	Oxygen				
7	90	100	7	Oxygen				
8	90	200	3	Nitrogen				
9	90	300	5	Air				

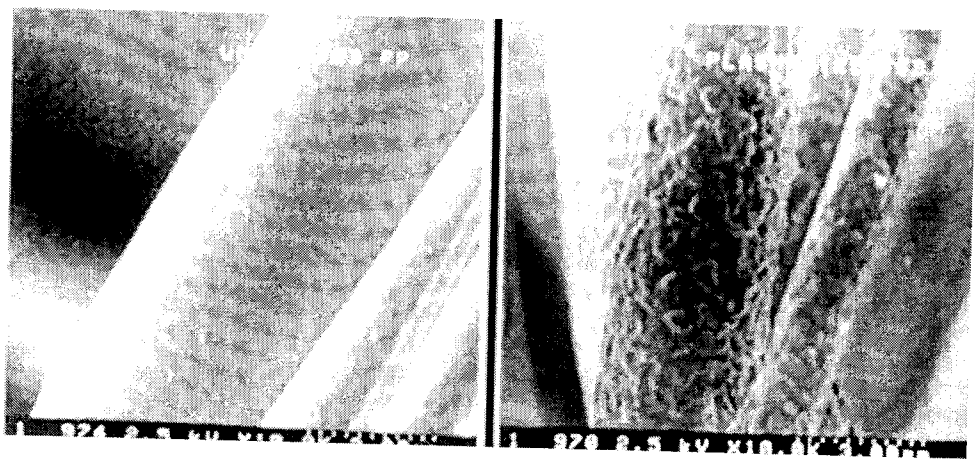
The Taguchi Excel sheet prepared by Dr. Apte, IIT, Bombay, Powai. Mumbai available in webpage http://www.ee.iitb.ac.in/-apte was used to get the factor effect plot & ANOVA tables and analysis was done.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 SURFACE MORPHOLOGY ANALYSIS

The effects of plasma treatment on morphological changes in the untreated and treated samples are shown in the form of SEM Micrograph in Fig 4.1 & 4.2. The untreated polypropylene fibre had a smooth surface, while all plasma treated samples exhibited surface morphological changes. Increased surface roughness can be produced by the etching effect of plasma active species bombardment on the polypropylene surface.



Control sample ***Sample without spin finish removal***

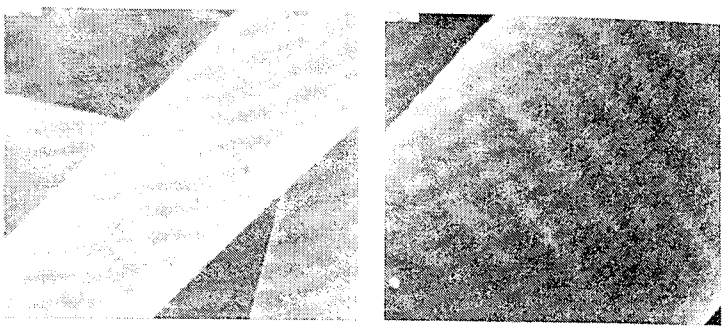
(Electrode-Copper, Distance between the Electrodes-3 cm. Treatment Time - 60sec, Pressure- 300 mtorr , Power-300 V, GSM of fabric- 80, Plasma – Oxygen, Magnification-2500X and 6000X)

Fig. 4.1 SEM Micrograph of untreated and plasma treated polypropylene fibres with spin finish on the surface.

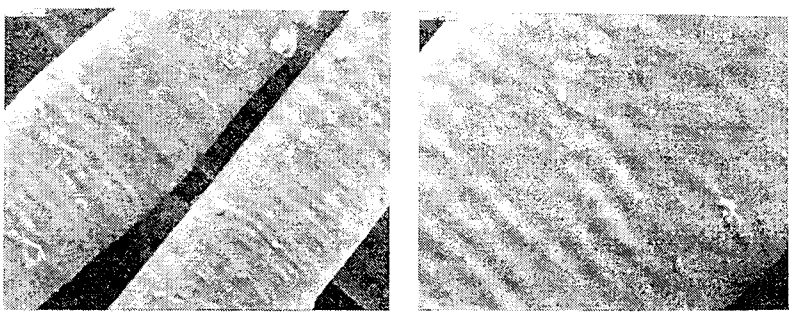
At shorter exposure times (30 and 60 sec), the surface morphological changes do not seem significant with plasma treatment. However, the surface morphological changes appear more pronounced with further exposure times, resulting in increased surface rippling and waviness developed in the direction

perpendicular to fibre axis. Therefore, the surface of polypropylene can reveal more etching effect due to the longer duration of plasma-substrate interaction.

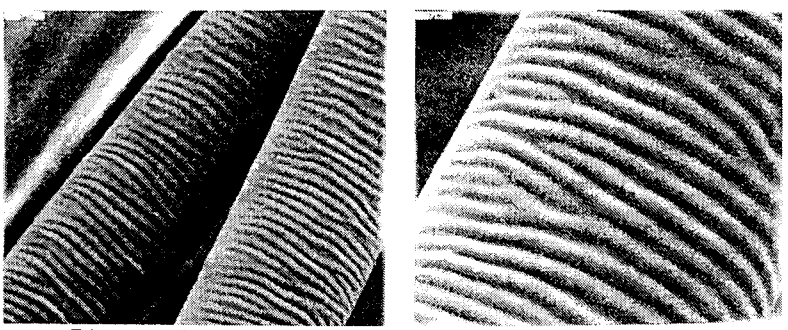
In case of fibres with spin finish on its surface, voids like microdeformation (roughness) were observed homogeneously throughout the surface (Fig.4.1). This could be possibly due to the rupture of the thin film coating of spin finish on the fibre surface and penetration of plasma to only few micron depth levels



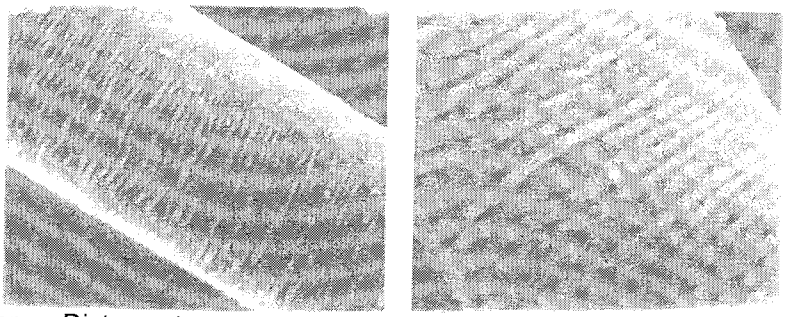
(Electrode–Copper, Distance between the Electrodes-3 cm. Treatment Time - 30sec, Pressure-300 mtorr, Power-300 V, GSM of fabric-80, Plasma-Oxygen, Magnification-2500X and 6000X)



(Electrode-Copper, Distance between the Electrodes-3 cm, Treatment Time - 60sec, Pressure-300 mtorr , Power-300 V , GSM of fabric-80, Plasma- Oxygen, Magnification-2500X and 6000X)



(Electrode–Copper, Distance between the Electrodes-3 cm., Treatment Time - 90sec, Pressure-300 mtorr, Power -300 V, GSM of fabric-80, Plasma – Oxygen, Magnification-2500X and 6000X))



(Electrode–Copper, Distance between the Electrodes-3 cm. Treatment Time - 90sec, Pressure – 300 mtorr, Power –300 V, GSM of fabric – 80, Plasma- Air, Magnification-2500X and 6000X)

Fig. 4.2 SEM Micrograph of polypropylene fibres treated with Air & Oxygen plasmas for different durations.

4.2 FTIR SPECTRA ANALYSIS

The FTIR finger prints obtained for untreated and plasma treated samples are given in Annexure. Breaking of chemical bonds, implantation of polar groups, exchanging of polymer structure & elemental composition within a few molecular layers and exchanging carbon–hydrogen bondings by functional groups are the various happenings in the surface of the fibre during plasma treatment. The type of introduced functional groups depends on the gas plasma used for treatment. These groups are responsible to provide better wettability and dyeability.

The spectra obtained for the oxygen plasma treated samples showed alteration from the untreated samples in the area of 1728 cm^{-1} that represents C=O groups implanted in the fibre. The height of the peaks also appears to have changed. The changes in the peak height and shift in the wave number from the untreated samples, in the wave numbers 2925-2853 cm^{-1}, 1270-1150 cm^{-1} and 3700-3480 cm^{-1} confirms the incorporation of C=O, COO and OH groups respectively. The shift in the region of 1100 cm^{-1} is also due to hydroxyl groups while the changes in 1784 cm^{-1} and 1284 cm^{-1} is also responsible for C=O and C-O groups respectively. Plasma treatment thus found to be effective in modifying the hydrophilic properties by including C=O, COOH and OH groups. The COOH component increases dramatically on the fibre surface after being treated with oxygen plasma.

4.3 EFFECT OF PLASMA TREATMENT ON ETCHING LOSS

Etching or cleaning to remove the surface layer basically with gas plasmas cause a cold burning process which transforms the surface in to typical burning products like water, carbon dioxide nitrous oxide and the likes. The results are shown in Table 4.1 & 4.2 and Fig. 4.3. Though insignificant weight loss in the range of 0.010-0.126% was obtained, the rippling and waviness effect seen in SEM micrograph was predominant. The highest weight loss occurs with oxygen and nitrogen-plasmas followed by air plasma. Weight loss was found to be increasing with exposure time. The difference in weight loss with respect to gas plasmas confirms that the physical etching nature and rate are different for different gases. This is also reflecting in the SEM micrographs.

Table 4.1 Effect of Process Parameters on Signal-to-Noise Ratio of Etching Loss

Noise Factor Repetitions					*1*	*2*	*3*	*4*	
Expt. No.	*Time (secs)*	*Pressure (mtorr)*	*Distance between the Electrodes (cm)*	*GSM*	*100*	*80*	*100*	*80*	*S/N ratio*
				Type of electrode	*Cu*	*Cu*	*Al*	*Al*	
				Type of gas plasma	*1*	*2*	*3*	*4*	
1	30	100	3	Air	0.089	0.098	0.078	0.081	-21.47
2	30	200	5	Oxygen	0.069	0.076	0.060	0.069	-23.38
3	30	300	7	Nitrogen	0.029	0.038	0.022	0.026	-31.32
4	60	100	5	Nitrogen	0.062	0.078	0.054	0.061	-24.13
5	60	200	7	Air	0.034	0.041	0.026	0.031	-29.98
6	60	300	3	Oxygen	0.100	0.112	0.089	0.097	-20.13
7	90	100	7	Oxygen	0.018	0.022	0.010	0.016	-36.78
8	90	200	3	Nitrogen	0.109	0.126	0.088	0.097	-19.81
9	90	300	5	Air	0.062	0.079	0.050	0.059	-24.42

(values in percentage)

The distance between electrodes plays dominant role while other variables play least role as for as weight loss due to plasma treatment is concerned. As the whole, the weight loss seen was to a very insignificant level.

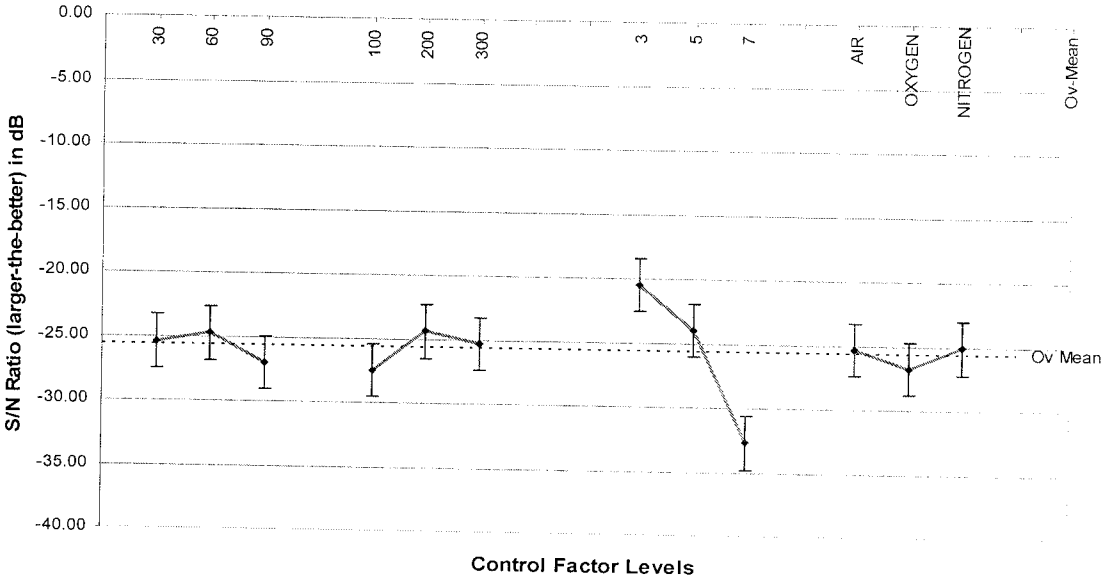


Fig.4.3 Factor effect plot for Etching loss

Table 4.2 ANOVA for Etching Loss

Control factors \ levels	*Levels*			*Degree of freedom*	*Mean Square*	*Factor Effect (percent)*	*Factor After pooling*
	1	*2*	*3*				
Time (secs)	-25.39	-24.75	-27.00	2	4	3	-
Pressure (mtorr)	-27.46	-24.39	-25.29	2	7	6	2
Distance between the electrodes(cm)	-20.47	-23.98	-32.69	2	119	89	36
Type of gas plasma	-25.29	-26.76	-25.09	2	3	2	-

4.4 EFFECT OF PLASMA TREATMENT ON WETTABLITY

The wettability / hyrophilicity of treated and untreated PP samples were assessed by two methods viz. Absorbency by drop test (time of wetting) and wicking height. The results (Table 4.3 & 4.4: Fig. 4.4) of the tests showed significant reduction in wetting time i.e, improved wettability. As against 88-296 seconds of wetting time observed in different plasma treated samples, untreated PP does not absorb water drop at all, even after 10 mins. The results of different wetting time for different gas plasmas confirm the difference in chemical modifications that has taken place in the surface of the substrate. The type of gas used during plasma treatment had definite effect on the fabrics. The time of treatment and distance between electrodes had significant effect on the absorbency. With higher treatment time and shorter distance between electrodes, the absorbency has improved significantly. This is in agreement with the surface energy values obtained for the PP samples treated with different gas plasmas. The vertical upward wicking test results (Table 4.5 &4.6; Fig. 4.5) also showed similar results.

Table 4.3 Effect of Process Parameters on Signal-to-Noise Ratio of Absorbency

Noise Factor Repetitions					*1*	*2*	*3*	*4*	
Expt. No.	*Time (secs)*	*Pressure (mtorr)*	*Distance between the Electrodes (cm)*	*GSM*	*100*	*80*	*100*	*80*	*S/N ratio*
				Type of electrode	*Cu*	*Cu*	*Al*	*Al*	
				Type of gas plasma	*1*	*2*	*3*	*4*	
1	30	100	3	Air	132	109	166	143	-42.86
2	30	200	5	Oxygen	158	136	192	172	-44.39
3	30	300	7	Nitrogen	260	238	296	272	-48.54
4	60	100	5	Nitrogen	187	163	155	135	-44.14
5	60	200	7	Air	155	135	189	170	-44.27
6	60	300	3	Oxygen	88	66	120	101	-39.63
7	90	100	7	Oxygen	165	140	193	175	-44.57
8	90	200	3	Nitrogen	139	115	85	150	-41.92
9	90	300	5	Air	105	80	130	110	-40.65

(values in seconds)

Wetting and wicking are two related processes; a liquid that does not wet fibres can not wick in to fabric, so fibre wettablity is a prerequisite of wicking. The higher

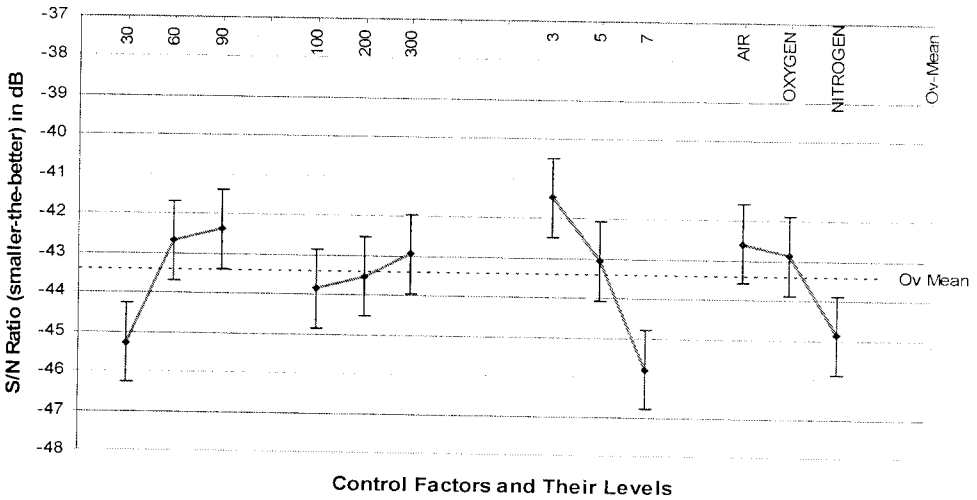


Fig.4.4 Factor effect plot for Absorbency

Table 4.4 ANOVA for Absorbency

Control factors \ levels	*Levels*			*Degree of Freedom*	*Mean Square*	*Factor Effect (percent)*	*Factor After pooling*
	1	*2*	*3*				
Time (secs)	-45.26	-42.68	-42.38	2	8	28	12
Pressure (mtorr)	-43.86	-43.53	-42.94	2	1	2	-
Distance between the electrodes(cm)	-41.47	-43.06	-45.79	2	14	53	22
Type of gas plasma	-42.59	-42.86	-44.87	2	5	17	7

wicking by plasma treated samples can be 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 fibres. Thus the shape and size distribution of the inter fibre capillary spaces will be modified.

Table 4.5 Effect of Process Parameters on Signal-to-Noise Ratio of Wickability

Noise Factor Repetitions					*1*	*2*	*3*	*4*	*S/N ratio*
Expt. No.	*Time (secs)*	*Pressure (mtorr)*	*Distance between the Electrodes (cm)*	*GSM*	*100*	*80*	*100*	*80*	
				Type of electrode	*Cu*	*Cu*	*Al*	*Al*	
				Type of gas plasma	*1*	*2*	*3*	*4*	
1	30	100	3	Air	46	51	39	44	32.94
2	30	200	5	Oxygen	41	47	36	41	32.19
3	30	300	7	Nitrogen	30	37	25	30	29.44
4	60	100	5	Nitrogen	22	27	17	20	26.29
5	60	200	7	Air	38	42	30	34	30.92
6	60	300	3	Oxygen	57	61	52	55	34.96
7	90	100	7	Oxygen	28	33	23	27	28.65
8	90	200	3	Nitrogen	46	53	38	43	32.80
9	90	300	5	Air	55	61	47	52	34.49

(Values in mm)

Table 4.6 ANOVA for Wickability

Control factors \ levels	***Levels***			***Degree of Freedom***	***Mean Square***	***Factor Effect (percent)***	***Factor After pooling***
	1	***2***	***3***				
Time (secs)	31.52	30.72	31.98	2	1	4	-
Pressure (mtorr)	29.30	31.97	32.96	2	11	33	9
Distance between the electrodes(cm)	33.57	30.99	29.67	2	12	36	10
Type of gas plasma	32.79	31.93	29.51	2	9	27	7

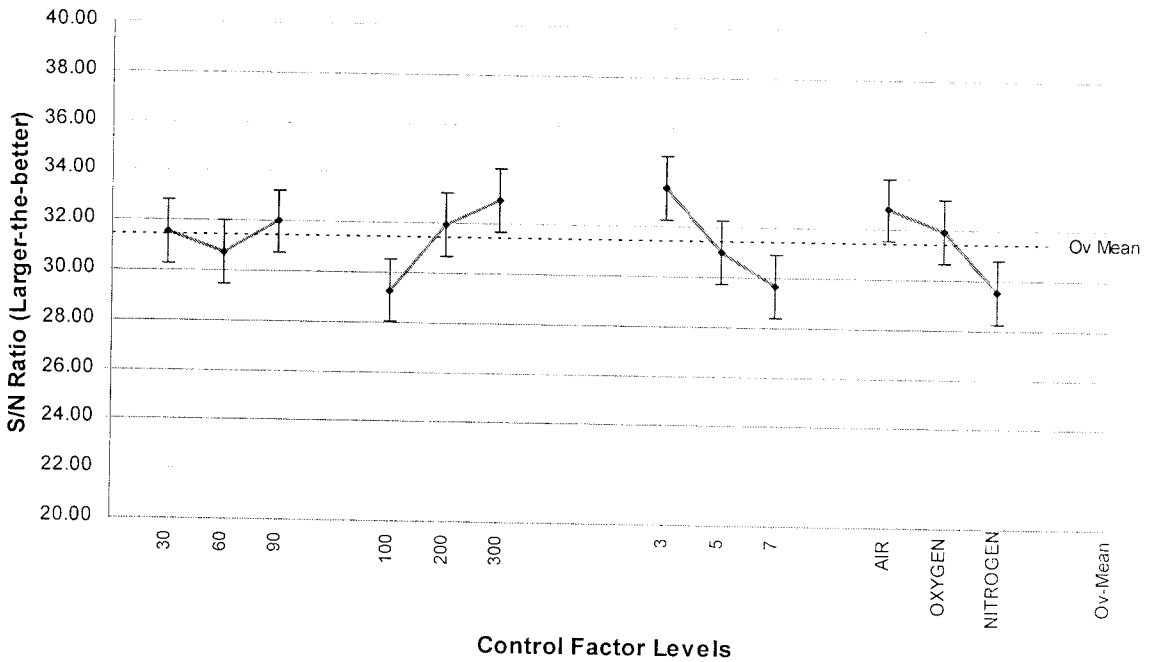


Fig.4.5 Factor effect plot for Wicking

This may also lead to the unblocking of surface capillaries and hence promote more rapid wetting. While in drop test, distance between electrodes playing dominant role followed by time of treatment and type of gas plasma; in wickablity results, pressure, distance between electrodes and type of gas plasma play more or less equal role with time of treatment being the least. This is something we could not explain.

4.5 EFFECT OF PLASMA TREATMENT ON SURFACE ENERGY

The change in surface energy of polypropylene fabrics upon plasma treatment is shown in Fig. 4.6 and Table 4.7 & 4.8. Surface energy of polypropylene is 29 dynes/cm [~~52~~ 21, ~~67~~ 26, 105]. Upon plasma treatment, it can be increased significantly to a value greater than 71 dynes/cm. The method followed for measuring surface energy could not be used for determining values greater than 71 dynes/cm. The surface energy was measurable in the range of 38-71 dynes/cm using formic acid and water combination method. As the values of surface energy obtained in the study were very close to 71 dynes/cm and not exceeded, this method was considered adequate for

our study. Pressure and type of gas plasma play dominant roles while other variables play comparatively much less role as for as improving surface energy of the substrate by plasma treatment is concerned.

Table 4.7 Effect of Process Parameters on Signal-to-Noise Ratio of Surface Energy

Noise Factor Repetitions					*1*	*2*	*3*	*4*	*S/N ratio*
Expt. No.	*Time (secs)*	*Pressure (mtorr)*	*Distance between the Electrodes (cm)*	*GSM*	*100*	*80*	*100*	*80*	
				Type of electrode	*Cu*	*Cu*	*Al*	*Al*	
				Type of gas plasma	*1*	*2*	*3*	*4*	
1	30	100	3	Air	48	50	38	41	32.84
2	30	200	5	Oxygen	48	52	38	41	32.84
3	30	300	7	Nitrogen	39	42	38	38	31.37
4	60	100	5	Nitrogen	38	41	38	38	30.44
5	60	200	7	Air	53	57	45	48	34.01
6	60	300	3	Oxygen	68	72	55	59	35.92
7	90	100	7	Oxygen	43	46	38	41	32.30
8	90	200	3	Nitrogen	46	50	38	41	32.70
9	90	300	5	Air	70	71	72	70	36.44

(values in dynes/cm)

Table 4.8 ANOVA for Surface Energy

Control factors \ levels	***Levels***			***Degree of Freedom***	***Mean Square***	***Factor Effect (percent)***	***Factor After pooling***
	1	***2***	***3***				
Time (secs)	32.48	33.89	34.02	2	2	16	2203
Pressure (mtorr)	32.30	33.16	34.92	2	5	40	5327
Distance between the electrodes(cm)	33.78	33.85	32.75	2	1	8	1134
Type of gas plasma	34.58	33.71	32.09	2	5	36	4782

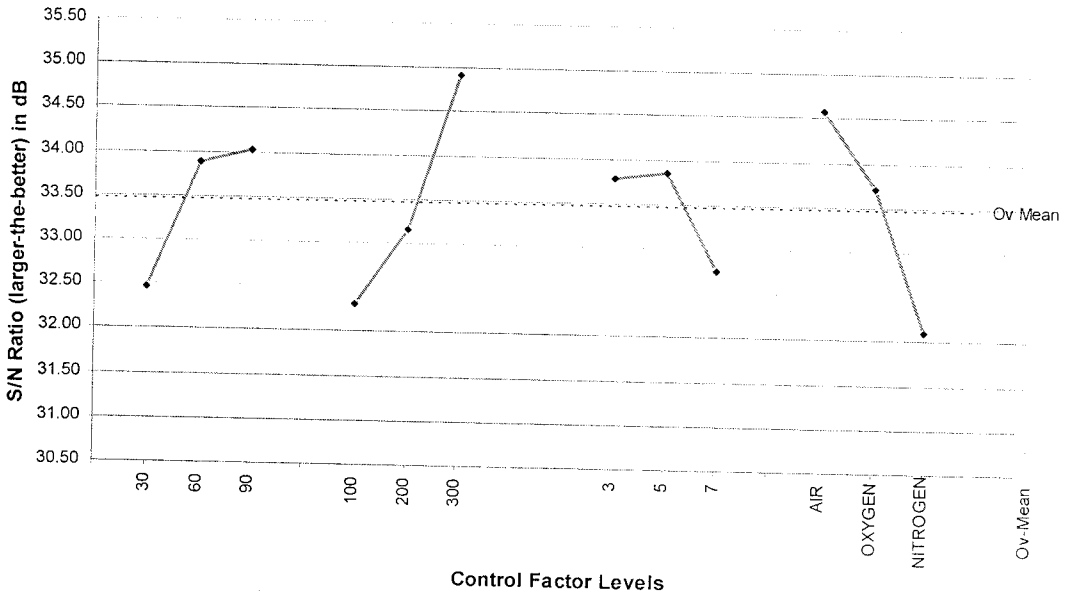


Fig.4.6 Factor Effect Plot for Surface Energy

The improvement in surface energy is also reflecting in improved wettability results as, always higher the surface energy of the substrate better will be the wettablity

4.6 EFFECT OF PLASMA TREATMENT ON YELLOWING AND WHITENESS

It has been observed that some degree of yellowing and loss in whiteness is something inevitable in plasma treatment. The results of yellowness index and whiteness index measured for untreated and various plasma treated samples using computer colour measuring software is presented in Table 4.9 & 4.11 and Fig. 4.7 & 4.8. The ANOVA results of the same are shown in Table 4.10 and 4.12. The type of gas used during plasma treatment had an effect on the yellowing of fabrics. The time of treatment and distance between electrodes had significant effect on the loss in whiteness. With higher treatment time and shorter distance between electrodes, the whiteness has reduced and the degree of yellowing has increased. This effect was

seen on invariably all plasma treated samples with different gases. The reason for this observed yellowing could be possibly due to the presence of oxidized substances (burning products) like carbon dioxide, nitrous oxide and the likes that are developed, forming some compounds during plasma treatment on fabric surface.

The distance between electrodes followed by the type of plasma plays dominant role than the time of treatment and pressure in deciding level of loss in whiteness and increase in yellowness of fabric. The maximum loss in whiteness was observed in air plasma treatment for 30 seconds at 100 mtorr pressure with distance between electrode of 3 cm. and minimum being, with nitrogen plasma treatment for 30 seconds at 300 mtorr pressure with distance between electrode of 7 cm. Under the same gas plasmas the minimum and maximum yellowing was noticed.

Table 4.9 Effect of Process Parameters on Signal-to-Noise Ratio of Whiteness Index

Noise Factor Repetitions					*1*	*2*	*3*	*4*	*S/N ratio*
Expt. No.	*Time (secs)*	*Pressure (mtorr)*	*Distance between the Electrodes (cm)*	*GSM*	*100*	*80*	*100*	*80*	
				Type of electrode	*Cu*	*Cu*	*Al*	*Al*	
				Type of gas plasma	*1*	*2*	*3*	*4*	
1	30	100	3	Air	69.3	66.2	78.1	74.1	37.09
2	30	200	5	Oxygen	75.1	70	80.6	79.6	37.62
3	30	300	7	Nitrogen	80.6	81.3	82.5	83.0	38.28
4	60	100	5	Nitrogen	82.1	79.0	78.7	71.9	37.80
5	60	200	7	Air	78.0	72.1	81.5	78.0	37.75
6	60	300	3	Oxygen	66.4	68.9	71.3	68.2	36.73
7	90	100	7	Oxygen	76.9	79.9	77.0	80.0	37.89
8	90	200	3	Nitrogen	75.1	72.1	77.1	77.9	37.55
9	90	300	5	Air	67.9	62.3	75.2	73.1	36.78

(values in whiteness index)

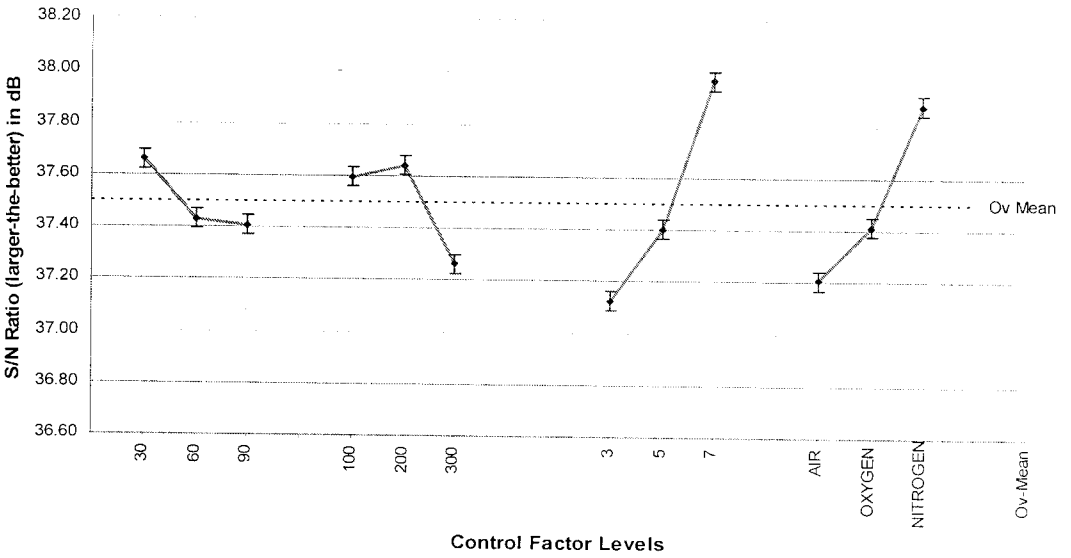


Fig.4.7 Factor Effect Plot for Whiteness Index

Table 4.10 ANOVA for Whiteness Index

Control factors \ levels	*Levels*			*Degree of Freedom*	*Mean Square*	*Factor Effect (percent)*	*Factor After pooling*
	1	*2*	*3*				
Time (secs)	37.66	37.43	37.41	2	0	5	59
Pressure (mtorr)	37.59	37.64	37.26	2	0	12	127
Distance between the electrodes (cm)	37.12	37.40	37.97	2	1	51	559
Type of gas plasma	37.21	37.41	37.88	2	0	32	355

Table 4.11 Effect of Process Parameters on Signal-to-Noise Ratio of Yellowness Index

Noise Factor Repetitions					1	2	3	4	
Expt. No.	Time (secs)	Pressure (mtorr)	Distance between the Electrodes (cm)	GSM	100	80	100	80	S/N ratio
				Type of electrode	Cu	Cu	Al	Al	
				Type of gas plasma	1	2	3	4	
1	30	100	3	Air	9.62	10.42	6.38	8.09	-18.85
2	30	200	5	Oxygen	7.80	9.12	4.92	5.19	-16.88
3	30	300	7	Nitrogen	4.98	4.08	3.92	3.76	-12.49
4	60	100	5	Nitrogen	3.64	5.54	6.23	8.33	-17.07
5	60	200	7	Air	6.28	8.08	3.96	5.54	-17.05
6	60	300	3	Oxygen	11.52	9.15	8.62	9.84	-21.44
7	90	100	7	Oxygen	6.65	5.12	6.54	5.01	-12.98
8	90	200	3	Nitrogen	6.82	8.08	5.78	5.12	-15.87
9	90	300	5	Air	9.92	11.54	7.12	8.09	-19.39

(values in yellowness index)

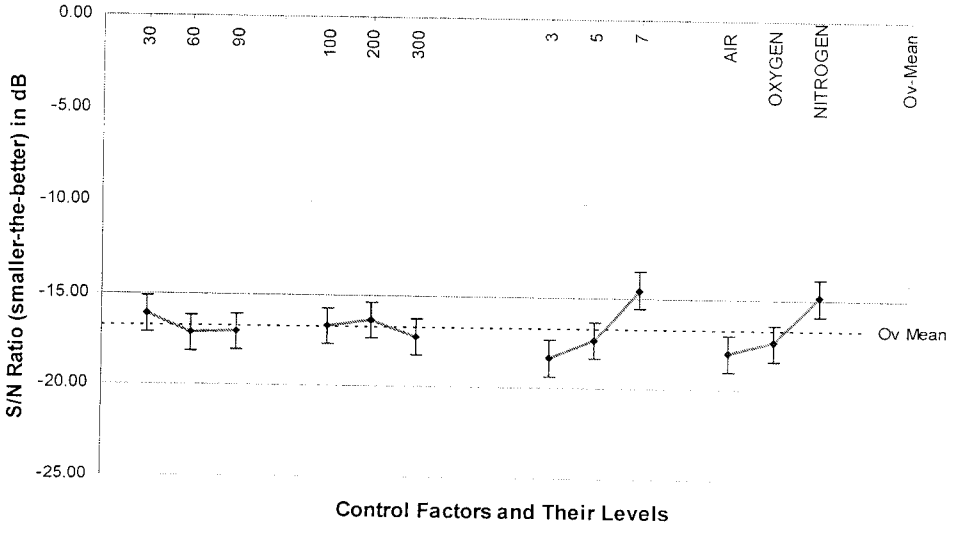


Fig.4.8 Factor Effect Plot for Yellowness Index

Table 4.12 ANOVA for Yellowness Index

Control factors \ levels	*Levels*			*Degree of Freedom*	*Mean Square*	*Factor Effect (percent)*	*Factor After pooling*
	1	*2*	*3*				
Time (secs)	-16.08	-17.14	-17.03	2	1	5	-
Pressure (mtorr)	-16.68	-16.33	-17.24	2	1	3	-
Distance between the electrodes (cm)	-18.34	-17.36	-14.55	2	12	54	14
Type of gas plasma	-18.01	-17.37	-14.87	2	8	38	10

4.7 EXHAUSTION AND COLOUR STRENGTH (K/S) IN REACTIVE AND DISPERSE DYEINGS

To evaluate the effect of plasma treatment on dyeability characteristics of PP, they were dyed with disperse and reactive dyes. While the reactive dye using cold brand was carried out according to the standard procedure, the dyeing with disperse dye was carried out at boil. Dyeing at higher temperatures (130-135^0 C) similar to dyeing of polyester is not possible as PP fibre cannot withstand this high temperature for the dyeing duration.

It is important to note that, because of the weight loss of the plasma treated samples due to the etching effect, which occurs in the plasma chamber, the weight loss leads to the total surface area of the samples being effectively changed.

The improvement in wettability improves the dyeing characteristics and the functional groups generated in the fibre surface either by breaking of chemical bonds or by oxidation, such as C–OH, C=O, COOH, C-O-C helps the dye to interact with the fibre and bound chemically which is confirmed by the reasonably good exhaustion in dyeing & colour strength values and fastness properties 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 4.14).

Table 4.13 Exhaustion of Reactive and Disperse Dyes on Untreated and Plasma Treated fabrics

Dye	*Sample*	*% Exhaustion*
C.I Disperse violet 27 (λ_{max} = 416 nm)	Untreated O_2 plasma Air plasma N_2 plasma	5.8 56.2 68.5 49.3
C.I Reactive Blue 4 (λ_{max} =518 nm)	Untreated O_2 plasma Air plasma N_2 plasma	5.0 35.5 41.2 29.4

Table 4.14 Colour Strength of Reactive and Disperse Dyed Untreated and Plasma Treated fabrics

Dye	*Sample/Treatment*	*K/S at λ_{max}*
C.I Disperse violet 27 (λ_{max}=416 nm)	Untreated O_2 plasma Air plasma N_2 plasma	0.142 1.22 1.82 0.97
C.I Reactive Blue 4 (λ_{max}=518 nm)	Untreated O_2 plasma Air plasma N_2 plasma	0.09 1.64 2.02 1.38

(Distance between electrodes-3 cm, Treatment time-60 secs, Pressure-300 mtorr, Power-300V, Electrode - Cu., Illuminant- D65, Observer- 2^0)

From the Table 4.13 it can be observed that in both disperse and reactive dyeing, considerable amount of dye got exhausted on to the plasma treated fibre.

While the untreated PP has shown only tinting, with an exhaustion of only 5 - 6 % in both dyeings, plasma treated PP has shown significant improvement, with an exhaustion of the order of 49-68% and 29-41% respectively.

Compared to very high exhaustion in the range of 90-95% that normally takes place and possible in polyester, only less exhaustion has taken place in plasma treated PP in disperse dyeing. The reason for this could be possibly because of the high crystalline nature of PP than polyester. The possibility of dyeing plasma treated PP with disperse dye is achieved mainly because of the functional groups generated on the surface and the interaction of the dye by forming hydrogen bonds with these groups, whereas, the polyester-disperse dye dyeing system follows solid solution theory. The other possible reason could be the rupture of chemical bonds on the surface and the physical changes (etching) that has taken place in the morphology of the fibre which enabled dye reaching more surface area.

While 60-80% exhaustion is possible with cold brand reactive dye on cotton, only 29-41% exhaustion was observed on plasma treated polypropylene. As the reactive dye can form covalent bond only with OH groups out of the various functional groups generated due to plasma treatment and the less quantity of OH groups available now, only low levels of exhaustion was obtained. In both reactive and disperse dyeings, better results were obtained in air plasma treated samples followed by oxygen and nitrogen plasmas.

4.8 WASH AND RUBBING FASTNESS OF DYED FABRICS

The wash fastness and rubbing fastness results of disperse and reactive dyed plasma treated samples are presented in Table 4.15. Satisfactory to excellent wash fastness and moderate rubbing fastness were obtained .This confirms that the dye taken up by the fibre is permanently fixed (chemically bonded) with the fibre.The wet rubbing fastness was found to be slightly inferior to dry rubbing fastness. Though low levels of exhaustion was achieved in the dyeing, whatever dye exhausted on to the

fibre got fixed with the fibre by forming covalent bonds with OH groups generated on the fibre surface in case of reactive dyeing and by hydrogen bonds with hydrophobic interactions with the fibre in case of disperse dyeing.

Table 4.15 Wash and Rubbing Fastness of Dyed fabrics

Dye/ Plasma Treatment	*Wash Fastness*		*Rubbing Fastness*			
	CC	*SW*	*Dry*		*Wet*	
			CC	*SW*	*CC*	*SW*
Disperse Dye						
Oxygen	4	4	3	3	2-3	2-3
Nitrogen	3-4	3	2-3	2-3	2	2-3
Air	4-5	4-5	3-4	3-4	3	3
Reactive Dye						
Oxygen	4-5	4	3-4	3-4	3	3
Nitrogen	4	3-4	3	3	2-3	2-3
Air	4-5	4-5	4	4	3-4	3-4

CC : Change in colour SW : Staining on white

4.9 EFFECT OF PLASMA TREATMENT ON TENSILE STRENGTH

Tensile strength measurement of the untreated and plasma treated samples are presented in Table 4.16. Significant increase in tensile strength was observed with plasma treatment in the samples..

Table 4.16 Effect of Plasma Treatment on Tensile Strength of the Fabric

Treatment	*Tensile Strength (N)*		*% Increase in Tensile Strength*
	Mean	*SD*	
Control	92.4	3.7	-----
Oxygen Plasma	96.5	3.2	4.4
Nitrogen Plasma	94.0	4.0	1.7
Air Plasma	101.6	3.8	9.9

(Electrode-Copper, Distance between the electrodes-3 cm, Treatment time-60 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric-80)

The effect of increased fibre-to-fibre friction due to the etching that takes place during plasma treatment could be the reason for this. The structure of Polypropylene non-woven fabric consists of bonds between fibres, which are relatively weaker than those of woven or knitted fabrics consisting of yarns. Therefore, the increased tensile properties might be affected mainly by fibre-to-fibre friction when the fabric structure is deformed due to tensile force. Surface roughness increases the surface area thus enhancing the fibre-to-fibre friction. Among the different gas plasmas studied, air plasma showed the maximum increase in the tensile strength i.e. 9.9% as compared to Hydrogen and Oxygen plasmas in which the increase was, in the order of 1.7 and 4.4% respectively. This confirms that, while all other conditions of treatment remaining the same, the air plasma causes more effective surface impinging and changes in the fibre as compared to the other two plasmas.

4.10 EFFECT OF PLASMA TREATMENT ON MOISTURE CONTENT

While the untreated polypropylene had 0 % moisture content, an increase up to 0.11 % was demonstrated by the plasma treatment which is considered as a significant improvement in the moisture content (Table 4.17).

Moisture content of plasma treated PP will depend on two main factors: the changes in surface morphology due to the etching action of the plasma and the formation of polar groups on the surface, which will help moisture penetration and binding on the surface. These could be the possible reasons for the improved moisture content observed. Moisture content will also depend on the nature of polar groups generated.

4.11 AGEING / DECAY STUDY

In order to establish the presence and to study the stability of functional groups or free radicals created on fibre during the plasma treatment, it was considered important to investigate the effect of an ageing period on samples after being subjected to plasma treatment. The moisture content measurement and absorbency

by drop test were carried out immediately after plasma treatment and after different periods of ageing (storage) and the results are reported in Table 4.17 & 4.18.

Table 4.17 Moisture Content of Plasma Treated Fabric after Different Storage Periods.

Storage Time (Hrs)	*0 **	*2*	*4*	*6*	*8*	*18*	*24*	*48*
Moisture content (%)	0.17	0.15	0.19	0.12	0.11	0.11	0.11	0.11

(Untreated-0 % , Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-60 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80, Plasma-Oxygen, * Immediately after plasma treatment)

Table 4.18 Absorbency of Plasma Treated Fabric after Different Storage Periods

Storage time (Hrs)	0*	2	4	6	8	18	24	48
Absorbency (secs)	66	78	88	95	105	105	105	105

(Untreated - >10 mins. Electrode-Copper, Distance between the electrodes – 3 cm., Treatment time-60 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric-80, Plasma-Oxygen., * immediately after plasma treatment)

From the results, it is clear that as the ageing period increases the moisture content decreases markedly up to 4 hours period of ageing, but then the effect tends to slow down and nearly levels off. Similar trend was also observed in the absorbency results by drop test which confirms the decaying effect and stabilization after a period of ageing of 8 hrs. The stabilized effect obtained after 8 hrs. does not change even after 2 days which confirms the permanency of hydrophilisation / wettability obtained by plasma treatment. Surface roughness is not expected to change with ageing time while the hydrophilicity of the plasma treated PP was found to decrease with time. The surface hydrophobicity partly recovered at a rather fast

rate in the first few hours after plasma treatments becoming progressively slower afterwards before stabilising at a constant level.

4.12 SUMMARY

The summary of the findings through various tests results obtained are given in Table 4.19 & 4.20. Out of the two electrodes used, copper showed better results than aluminium. Though all gas plasmas showed improved hydrophilicity, air plasma appears to be better and beneficial, besides being available freely. Fabric with higher areal density was found to be effectively treated by plasma which is confirmed by the better results obtained in terms of hydrophilicity. Distance between electrodes plays a dominant role than the other factors, confirmed by absorbency, wickability and all other tests except surface energy in which pressure had an important role. Shorter distance between electrodes, longer treatment time and higher pressure of gas give better results of hydrophilicity but with significant loss in whiteness. Plasma treatment makes polypropylene dyeable with disperse and cold brand reactive dyes with very good fastness results. Air plasma showed better dyeing results than the other plasmas.

Table 4.19 Summary of Effect of Process Parameters on Signal-to-Noise Ratio

No.	Design Parameters				Signal-to-Noise ratio					
					Smaller the Better	Larger the Better	Larger the Better	Larger the Better	Smaller the Better	Larger the Better
	Time (Secs.)	Pressure (mtorr)	D.B.E (cms)	Plasma	A	W	SE	WI	YI	EL
1	30	100	3	Air	-42.86	32.94	32.84	37.09	-18.85	-21.47
2	30	200	5	O_2	-44.39	32.19	32.84	37.62	-16.88	-23.38
3	30	300	7	N_2	-48.54	29.44	31.37	38.28	-12.49	-31.32
4	60	100	5	N_2	-44.14	26.29	30.44	37.80	-17.07	-24.13
5	60	200	7	Air	-44.27	30.92	34.01	37.75	-17.05	-29.98
6	60	300	3	O_2	-39.63	34.96	35.92	36.73	-21.44	-20.13
7	90	100	7	O_2	-44.57	28.65	32.30	37.89	-12.98	-36.78
8	90	200	3	N_2	-41.92	32.80	32.70	37.55	-15.87	-19.82
9	90	300	5	Air	-40.65	34.49	36.44	36.78	-19.39	-24.42

DBE- Distance between the Electrodes, A- Absorbency, W- Wickability, SE- Surface Energy, WI- Whiteness Index, YI- Yellowness Index, EL- Etching Loss

Table 4.20 Summary of ANOVA Results

Design Parameters	Degree Freedom	Absorbency		Wickability		Surface Energy		Whiteness Index		Yellowness Index		Etching Loss	
		Factor Effect (%)	F Value	Factor Effect (%)	F Value	Factor Effect (%)	F Value	Factor Effect (%)	F Value	Factor Effect (%)	F Value	Factor Effect (%)	F Value
Time (Secs.)	2	28	12	4	-	16	2203	5	59	5	-	3	-
Pressure(mtorr)	2	2	-	33	9	40	5327	12	127	3	-	6	2
D.B.E(cms)*	2	53	22	36	10	8	1134	51	559	54	14	89	36
Plasma	2	17	7	27	7	36	4782	32	355	38	10	2	-

* DBE- Distance between the Electrodes

Plasma treatment results in significant improvement in moisture content with insignificant loss in weight. Though plasma treatment effect was found to be decaying with ageing period, it stabilises after a period of eight hours with significantly improved hydrophilicity in the fibre.

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 polypropylene surface

2. Plasma treatment causes implantation of functional groups and the type of introduced functiona1 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 polypropylene which was confirmed by absorbency evaluation through drop test, vertical wicking height and surface energy measurements.

5. The type of gas used during plasma treatment , the time of treatment and distance between electrodes, had definite and significant effect on the absorbency.

6. With higher treatment time and shorter distance between electrodes, the absorbency has improved significantly and this is in agreement with the higher surface energy values obtained.

7. While in drop test, distance between electrodes playing dominant role followed by time of treatment and type of gas plasma ; in wickablity results, pressure, distance

between electrodes and type of gas plasma play more or less equal role with time of treatment being the least

8. From a surface energy value of 29 dynes/cm in polypropylene, it is possible to improve to the order of 71 dynes/cm by plasma treatment.

9. Plasma treatment makes polypropylene dyeable with Disperse and Reactive dyes with very good fastness properties.

10. Plasma treatment causes some degree of yellowing and loss in whiteness which is something inevitable.

11. While the untreated polypropylene has 0 % moisture content, an increase up to 0.11 % was demonstrated by the plasma treatment which is a significant improvement in the moisture content.

12. Though plasma treatment effect was found to be decaying with ageing period, it stabilises after a period of eight hours with significantly improved hydrophilicity in the fibre.

13. Shorter distance between electrodes, longer treatment time and higher pressure of gas give better results of hydrophilicity.

14. Out of the two electrodes used, copper shows better results than aluminium.

15. Fabric with higher areal density was found to be effectively treated by plasma.

16. Though all gas plasmas improve hydrophilicity, air plasma appears to be better and beneficial, besides being available freely.

SCOPE FOR FUTURE WORK

- The effective plasma treatment method from Radio Frequency plasma, Dielectric barrier discharge plasma, Corona etc., can be investigated and identified in order to get the best results in terms efficacy of desirable properties on the substrate and cost economics.

- 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.

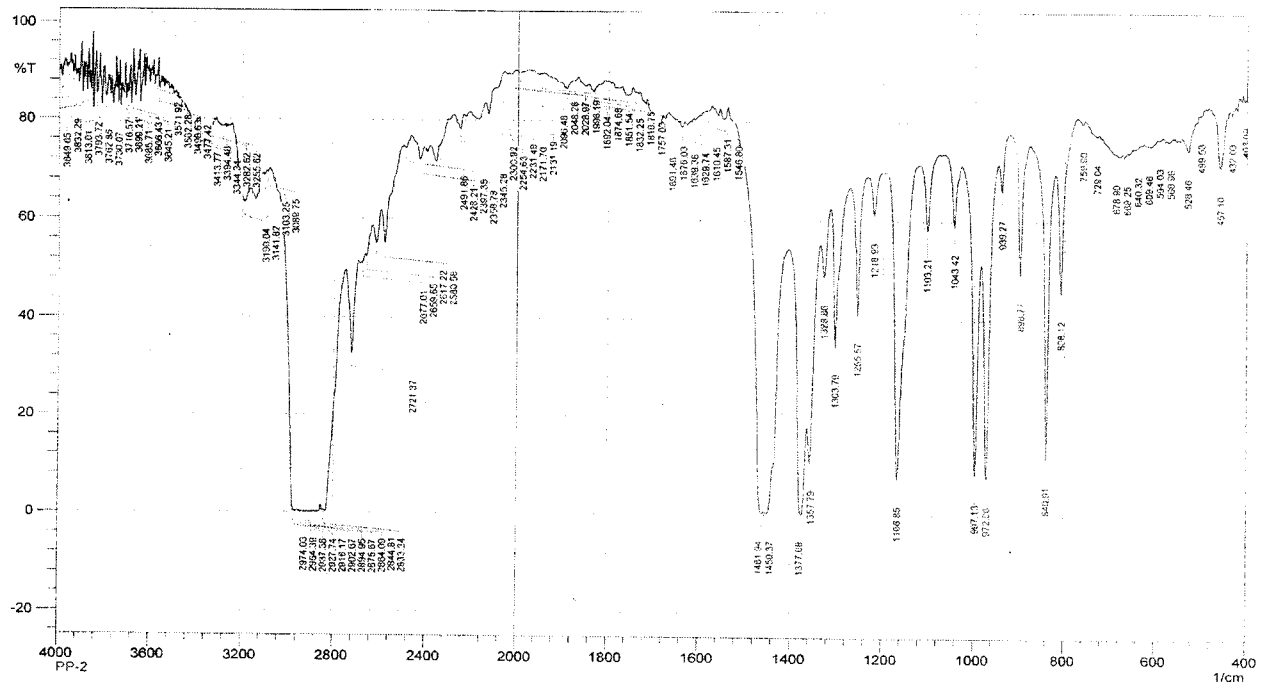
- Various plasmas with inert gases such as nitrogen, argon, helium and reactive gases such as ammonia, fluorocarbons, carbon dioxide, etc. can be studied independently and in combinations to obtain different surface changes and applications.

- Surface modifications, in order to impart soil repellency, flame retardency and anti-microbial kinds of effect, can be studied with the help of plasma polymerisable gases such as acrylic acids, organo silaxanes, etc.,

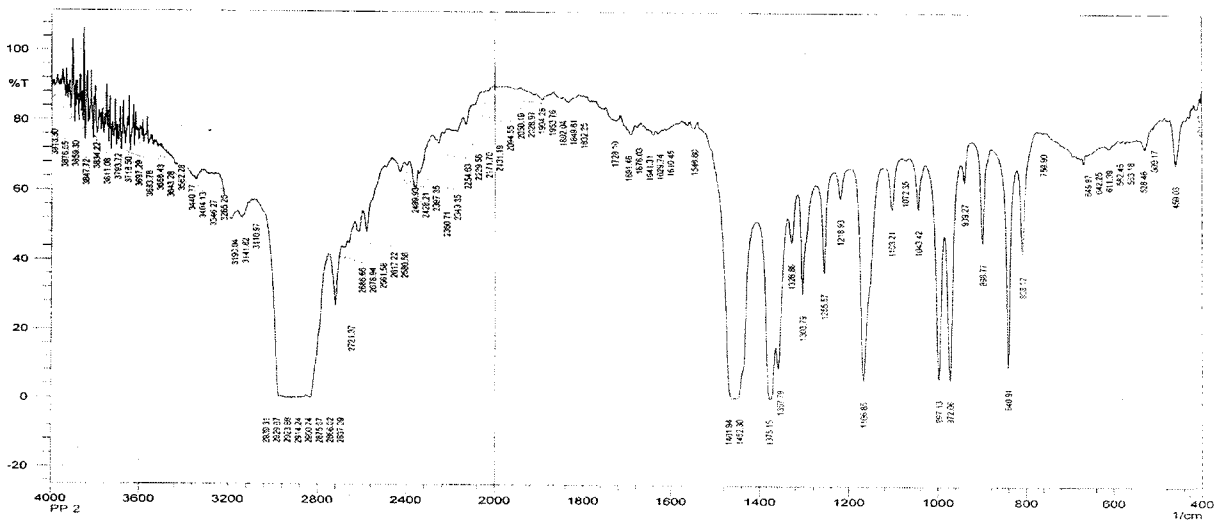
- Effect of Flow rate of gas, break down voltage for gases in different plasma methods can be investigated.

ANNEXURE

FTIR Spectra of untreated and plasma treated samples

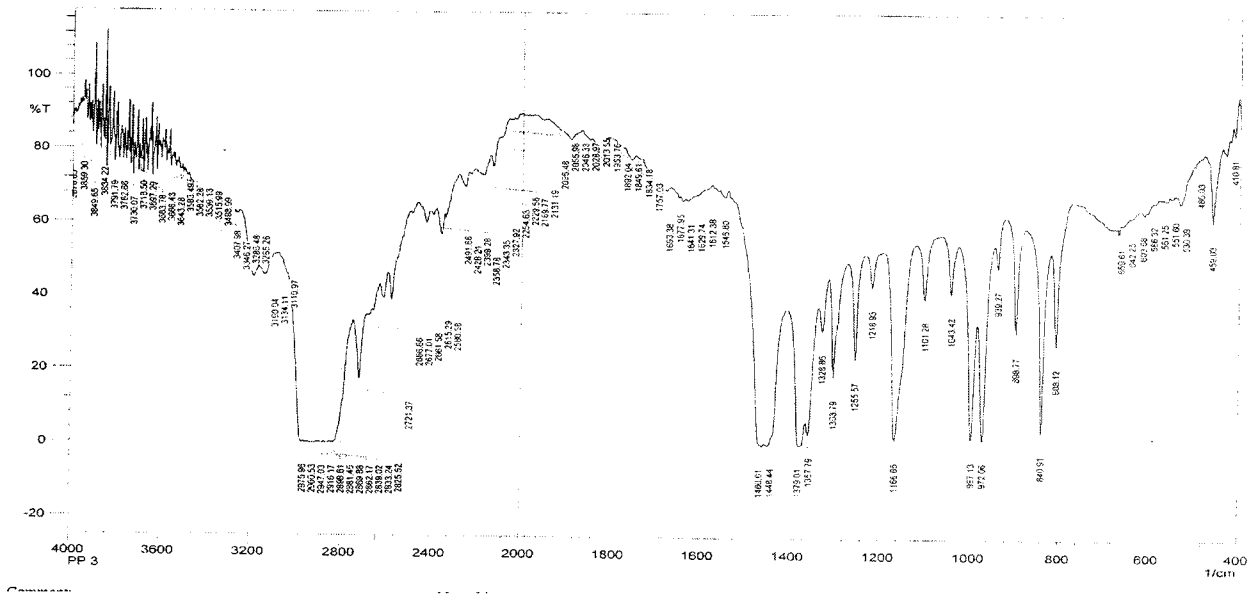


FTIR Spectra of untreated Sample



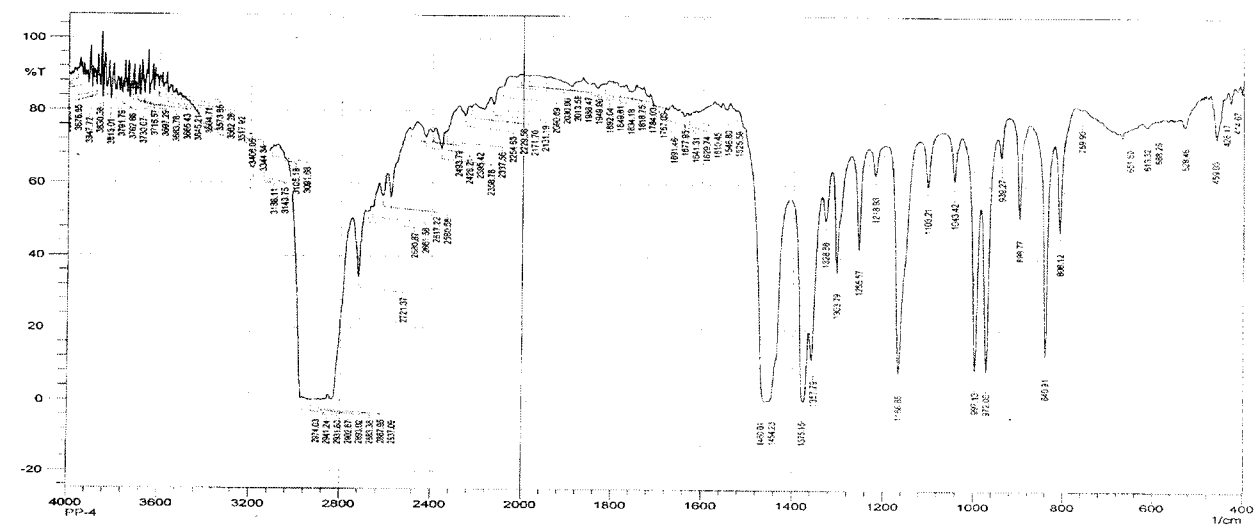
FTIR Spectra of Oxygen Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-30 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80,



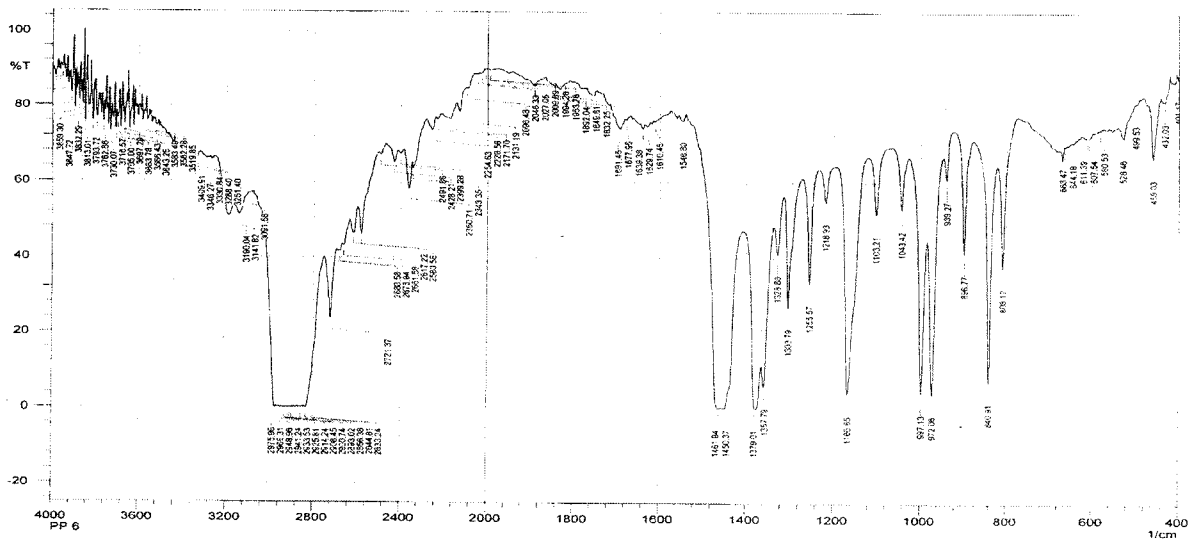
FTIR Spectra of Nitrogen Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-60 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80,



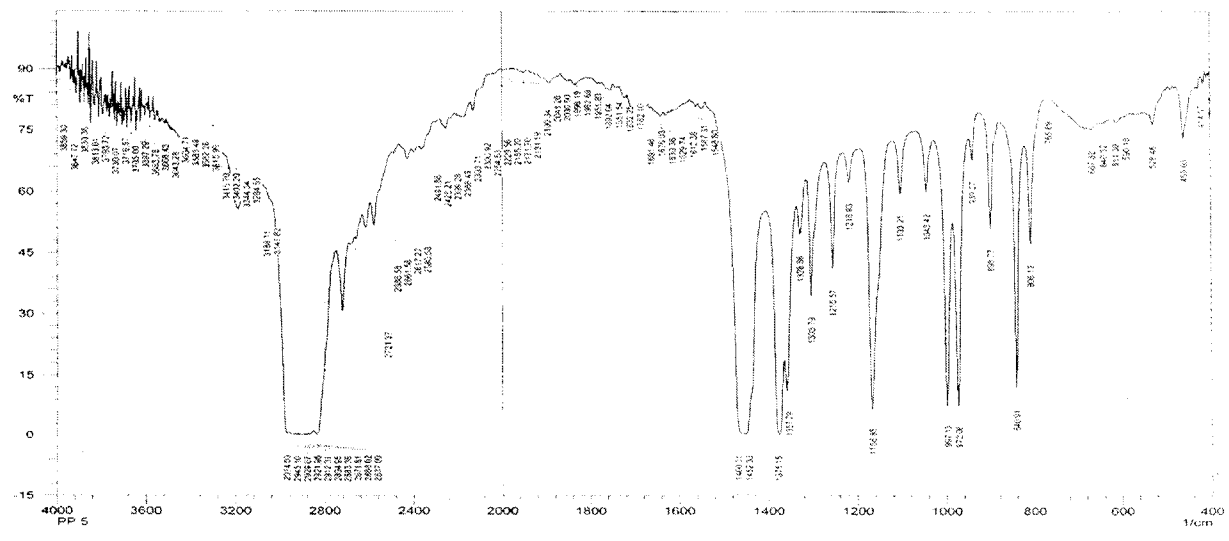
FTIR Spectra of Oxygen Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-60 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80, Plasma-Oxygen



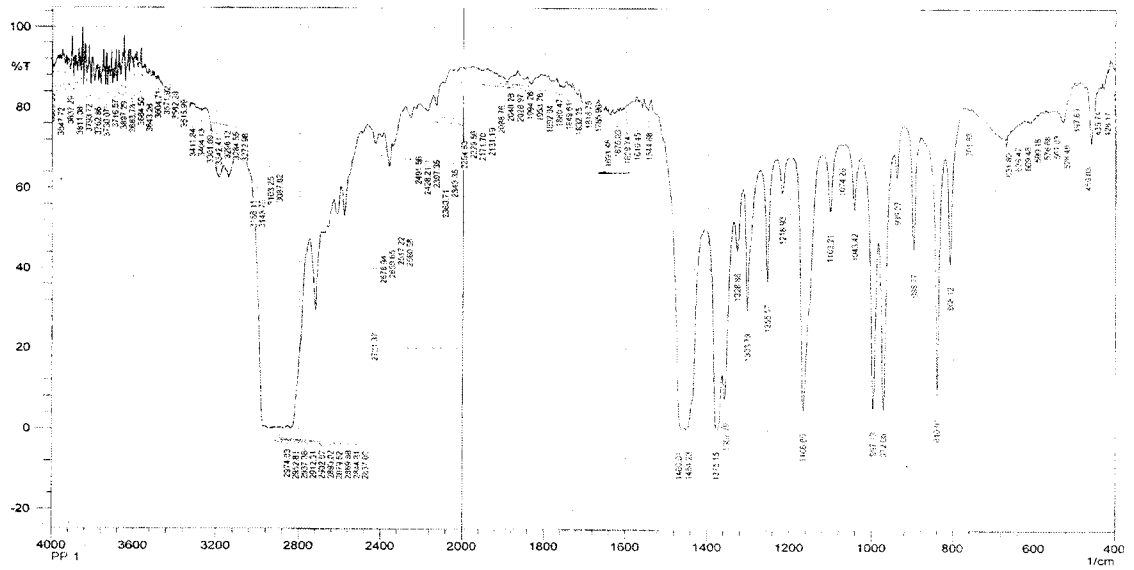
FTIR Spectra of Air Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-7 cm., Treatment Time-30 sec, Pressure-100 mtorr, Power-300 V, GSM of fabric – 80,



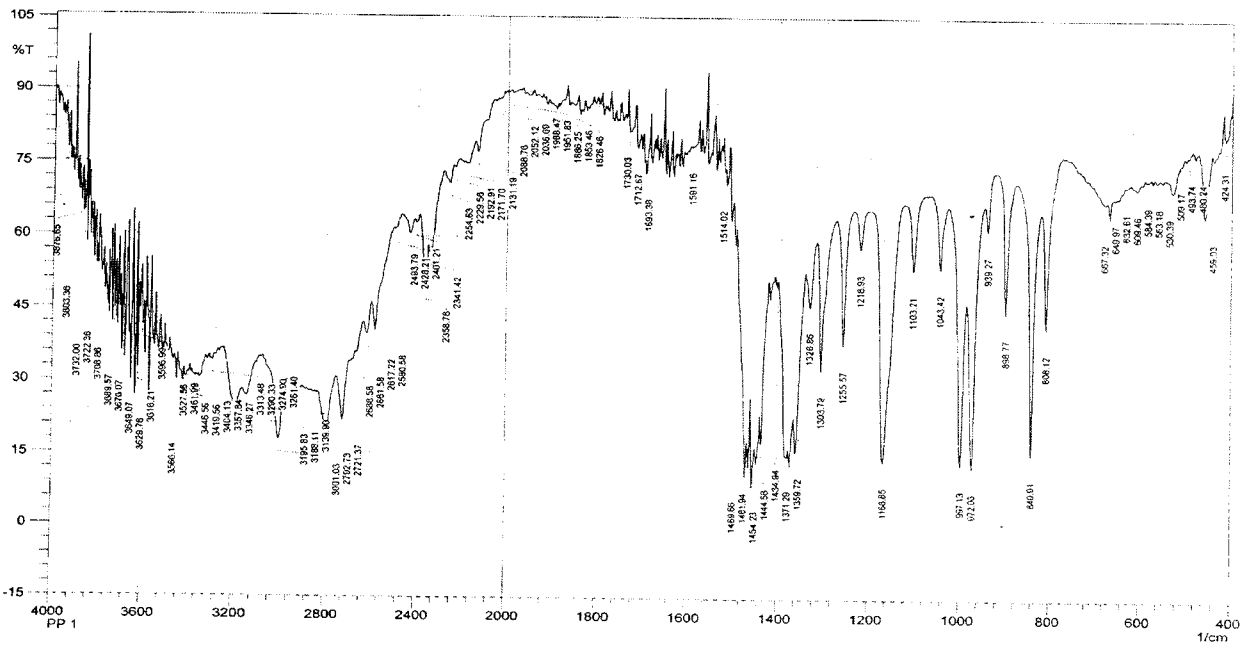
FTIR Spectra of Oxygen Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-5 cm., Treatment Time-30 sec, Pressure-100 mtorr, Power-300 V, GSM of fabric – 80,



FTIR Spectra of Nitrogen Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-90 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80,



FTIR Spectra of Air Plasma treated Sample

Electrode-Copper, Distance between the Electrodes-3 cm., Treatment Time-30 sec, Pressure-300 mtorr, Power-300 V, GSM of fabric – 80,

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