IN SITU ATMOSPHERIC PRESSURE PLASMA TREATMENT OF TEXTILES FOR ENHANCED PROPERTIES

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IN SITU ATMOSPHERIC PRESSURE PLASMA TREATMENT OF TEXTILES FOR ENHANCED PROPERTIES

by

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in fulfillment of the requirements of the degree of

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DEDICATED TO MY PARENTS, WIFE, DAUGHTER AND

ALL MY FAMILY MEMBERS

CERTIFICATE

This is to certify that the thesis titled '**In Situ Atmospheric Pressure Plasma Treatment of Textiles For Enhanced Properties',** being submitted by **Prasanta Kumar Panda** to the Indian Institute of Technology Delhi, for the award of degree **Doctor of Philosophy**, is a record of bonafide research work carried out by him. He has worked under our guidance and supervision and fulfilled the requirements for the submission of thesis which has attained the standard required for a PhD. Degree of this Institute.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

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ABSTRACT

Plasma process is an environmentally friendly and low temperature process. This process can be used as an alternate of wet chemical process. Unlike conventional wet chemical process, plasma process does not require either water or hazardous solvents. Further the plasma process does not affect the bulk properties and comfort properties of textiles. Recently, interest is growing in the area of atmospheric pressure plasma process for the industrial application in order to integrate the plasma with continuous process and avoid the costly pumping system involved with vacuum plasma. Etching of the textile surface using atmospheric pressure plasma to alter its behavior is a simple process. On the other hand, carrying out reaction of the precursor with the textile to impart durable functionality using atmospheric pressure plasma is a challenge. It is a usually a difficult task to create a favourable environment for the interaction of generated radicals with the surface of a fabric.

In the first part of this study, wool fabrics were treated with atmospheric pressure helium glow discharge plasma in an attempt to improve felting and dyeing behaviour with cold brand reactive dyes using cold pad-batch method at neutral pH. On glow plasma treatment, the hydrophilicity of wool surface and its resistance towards felting was greatly improved without any significant damage to the cuticle layer. The colour strength of the plasma treated dyed wool on the surface (in terms of K/S) was found to be nearly double of the colour strength of dyed untreated wool fabric, however, the corresponding total dye uptake of the treated wool increased by a much lower value of 40-50%. The reason behind this altered dyeing behavior was investigated by studying the dye uptake using infinite bath and surface characteristics using SEM and SIMS. It was found that the glow plasma treatment greatly

transformed the chemical surface of the wool fibers. It resulted in uniform removal of hydrophobic cuticular layer, which resulted in better diffusion of the dye molecules into the fiber, and formation of hydrophilic $–NH_2$ groups near the surface, which helped in anchoring the dye molecules close to the surface giving higher colour strength than expected.

In the second part, in situ reaction of various long chain hydrocarbon precursors with cellulosic substrate inside plasma zone were investigated. Each of the selected precursor compounds had C12 alkyl chain as the hydrophobic moiety to impart hydrophobicity to the substrate and a functional group to aid reaction with the active sites of the substrate. The selected compounds were dodecyl acrylate (DA, with acrylate group), lauryl alcohol (LA, with hydroxyl group), dodecanoic acid (DODAC, with carboxylic acid) and dodecane (DD, without any functional group). Treatment was carried out in situ with these compounds using helium gas as a carrier. After the treatment, durable hydrophobicity was obtained in all cases with water absorbency time of substrate increasing to >60 min in the case of dodecyl acrylate (DA) and lauryl alcohol (LA), up to 19 min in the case of dodecane (DD) and only up to 9 min in the case of dodecanoic acid (DODAC). The functional groups were found to have profound effect on the extent of functionality obtained and the plasma parameters required to obtain effective reaction with the substrate. Both plasma zone and the functionalized surfaces were characterized to understand the chemistry involved with the different precursors. Plasma zone was characterized by using oscilloscope, optical emission spectroscopy (OES), and gas chromatography-mass spectrometry (GC-MS). Functionalized surfaces were characterized by using ATR-FTIR, Raman and secondary ion mass spectroscopies. The study of the surface morphology by FE-SEM and the durability of treatment by solvent and soap washing revealed the formation of a thin layer covalently reacted on the individual fibres of the fabric.

In the third part, crease resistant functionalization of 100% cotton fabric was carried out using combination of acrylic acid (AA) and dodecanoic acid (DODAC). The treatment was carried out in situ using He / (AA+ DODAC) plasma at low frequency. After the treatment, improvement in the crease recovery angle of the cotton was found to be 40%. The functional property developed after the treatment was durable to both solvent and soap washing. Effect of various parameters such as concentration of precursors, power density and treatment time on functionalization was investigated. Plasma was characterized using oscilloscope and optical emission spectroscopy (OES). Modification of chemical nature of the fabric surface was characterized by ATR-FTIR spectroscopy. The scanning of the surface morphology by FE-SEM revealed the formation of a uniform thin layer on individual fibres. The fabric was found to loss only 28% its tearing strength while showing a small increase in tensile strength after the plasma treatment. Both the precursors, when used alone could not exhibit significant improvement in the crease recovery angle. On the other hand their combination was found to be helpful in imparting the functionality.

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List of Symbols and Abbreviations

Symbol/Abbreviation	**Expanded Form/Term**
T_e	electron temperature
T_h	heavy particle temperature
n_n	density of neutral particles
n_i	density of ions
n_e	density of electrons
n	density of plasma
m	meter
g	gram
L	liter
cm	centimeter
mm	millimeter
nm	nanometer
cc	cubic centimeter
h	hour
min	minute
He	helium

M	molar
W	watt
kV	kilovolt
kHz	kilohertz
CTE	completely thermodynamic equilibrium
LTE	local thermodynamic equilibrium
CCP	capacitive coupled plasma
ICP	inductively coupled plasma
DBD	dielectric barrier discharge
DC	direct current
AC	alternate current
I-V	current-voltage
OSHA	occupational safety and health administration
APGP	atmospheric pressure glow plasma
SLPM	standard liter per minute
HTHP	high temperature high pressure
FE-SEM	field emission scanning electron microscopy
SIMS	secondary ion mass spectrometer

DA	dodecyl acrylate
RMS	root mean square
OES	optical emission spectroscopy
GC-MS	gas chromatography mass spectrometry
ATR-FTIR	attenuated total reflectance-Fourier transform infrared
LA	lauryl alcohol
DODAC	dodecanoic acid
DD	dodecane
AA	acrylic acid