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Modification of surface properties of polypropylene (PP) film using DC glow discharge air plasma

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ABSTRACT

The industrial use of polypropylene (PP) films is limited because of undesirable properties such as poor adhesion and printability. In the present study, a DC glow discharge plasma has been used to improve the surface properties of PP films and make it useful for technical applications. The change in hydrophilicity of modified PP film surface was investigated by contact angle (CA) and surface energy measurements as a function of exposure time. In addition, plasma-treated PP films have been subjected to an ageing process to determine the durability of the plasma treatment. Changes in morphological and chemical composition of PP films were analyzed by atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS). The improvement in adhesion was studied by measuring T-peel and lap shear strength. The results show that the surface hydrophilicity has been improved due to the increase in the roughness and the introduction of oxygen-containing polar groups. The AFM observation on PP film shows that the roughness of the surface increased due to plasma treatment. Analysis of chemical binding states and surface chemical composition by XPS showed an increase in the formation of polar functional groups and the concentration of oxygen content on the plasma-processed PP film surfaces. T-peel and lap shear test for adhesion strength measurement showed that the adhesion strength of the plasma-modified PP films increased compared with untreated films surface.

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1. Introduction

Polypropylene (PP) shows excellent mechanical properties, light weight and a high adaptability to complicated structures and is therefore qualified for a broad range of applications *e.g.* in the fields of coating, bonding, printing and metallization, require good adhesion between the polymer and a surface layer such as a lacquer or a metal film [1–3]. However, good adhesion between the two substrates is a vital presence for giving high-strength laminates. Generally, the presence of polar groups and the surface topography of the substrate play a very important role for obtaining good adhesion and hence good mechanical performance of the laminates. The polypropylene films have limitation to their adhesion properties due to their non-polar nature and low surface energy [4,5]. For this reason PP films need, some additional treatment to raise surface activity, thus enhancing wettability and

consequently, the adhesive properties. Various attempts have been made to improve the surface properties of the PP films e.g. corona discharge, plasma treatment and chemical etching. Corona discharge has usually uneven surface modification, atmospheric pressure discharge causes surface etching by all types of species such as ions, electrons, etc. In contrast to this DC discharge has advantage that it is well controlled and we can select the type of ions for surface treatment. The DC glow discharge plasma has advantage that it is more convenient and efficient for processing of polymeric materials [6,7]. Therefore, in the present work, PP films were treated in DC discharge. We have chosen air as a precursor because it is cheaper and easy to work with. Recently, research on the use of glow discharge plasma treatment has grown in interest since they are environmentally efficient [7–9]. Moreover, it is a dry treatment method, which is better suited for industrial applications. Depending on the gas used for plasma generation and the general conditions, it is possible to activate a polymeric surface by inserting active species, surface etching, cross linking process or in combination of the process. The use of low-pressure conditions allows the plasma treatment at low or moderate temperatures; in

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this way, the aggressiveness of the plasma treatment is considerably reduced and consequently degradation is reduced. It is possible to generate a glow discharge plasma by either direct or RF current. A glow discharge plasma comprises a complex mixture of particles (ground-state and excited neutrals and ions, molecules and fragments and electrons) and a broad spectrum of electromagnetic radiation. These are all chemically active and lead to a variety of plasma chemical reactions and form new types of compounds. The different species present in plasma induce the formation of free radicals in the polymeric chain and in this way it is possible to insert or interact with certain functional groups on the polymer surface which will enhance the surface properties of the polymeric films [10-13]. In the present work polypropylene films were processed in DC glow discharge air plasma under different exposure times, with an aim of improving the intrinsic low surface properties. The change in hydrophilicity of plasmamodified PP films was characterized by measuring the contact angle (CA) as a function of exposure time. In addition, the plasmatreated samples have been subjected to an ageing process to determine durability of the plasma treatment. The surface morphology of the modified PP films were analyzed using atomic force microscopy (AFM). The functionalization of the plasmatreated PP film surfaces were characterized by X-ray photoelectron spectroscopy (XPS). The adhesion of PP film before and after treatment was investigated by T-peel and lap shear tests.

2. Experimental setup and methodology

2.1. Material

Polypropylene films of 80 μ m thickness were supplied by Reliance, India. The PP films were cut into 5 cm \times 5 cm sections for plasma treatment. Before the plasma treatment, the films were washed ultrasonically in acetone and distilled water for 15 min and then dried at room temperature.

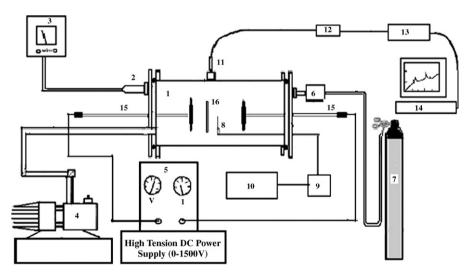
2.2. Glow discharge plasma treatment

Schematic diagram of the DC glow discharge setup, used for surface modification of the polymer films, is shown in Fig. 1. It consists of a cylindrical chamber which is made of glass tube of length 29 cm and internal diameter 10 cm and closed by end plates made of stainless steel. Two aluminium circular electrodes of diameter 5 cm are fixed inside the chamber parallel to each other and perpendicular to the axis separated by a distance of 3 cm and it is connected to the high-tension DC power supply (1.5 kV). Air was used as a processing gas. Before the plasma treatment, the chamber was first thoroughly cleaned and air tightened. Initially the chamber was evacuated to a pressure of 10^{-3} mbar using a vacuum pump (EDWARDS: E2M5). Through an air inlet (fine control gas needle valve) the required low pressure was maintained and measured by a pirani gauge. The polymer film was inserted with its surface perpendicular to the discharge axis between the parallel disc electrodes through a sample holder. The discharge potential and base pressure was kept constant at 400 V and 0.2 mbar, respectively. Typical operating parameters are listed in Table 1.The weight of the sample was measured before and after the plasma treatment, to estimate etching effects on the surface layers of PP films. A microbalance (Make: Mettler, model: AE 240) having accuracy 10^{-5} g was used for this purpose. The plasma etching effect described by weight loss was calculated using the following expression [14]:

Weight loss(%) =
$$\left(\frac{W_{\rm ut} - W_{\rm pt}}{W_{\rm ut}}\right) \times 100$$
 (1)

where $W_{\rm ut}$ and $W_{\rm pt}$ are the weight of untreated and plasma-treated samples, respectively.

The surface energy was measured by measuring the angle of contact using the sessile drop method. The liquids used for calculating surface energy of the PP films are water and glycerol of known γ^p (polar component) and γ^d (disperse component). The



1)Vacuum chamber 2) Pirani gauge head 3) Pirani Gauge 4) Rotary Pump 5)High tension DC Power supply 6)Fine control gas needle valve 7) Plasma gas cylinder 8) Langmuir Probe 9) Commutator 10) Bipolar Power supply 11) Fiber optics 12) 1/4 Monochromator 13) PMT Power supply14) Computer with X_T Recorder 15) Electrodes 16) polymer film

Fig. 1. Experimental setup of DC glow discharge plasma system.

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