Plasma surface treatment of HDPE powder in a fluidized bed reactor

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Summary

A plasma surface treatment of HDPE has been carried out in a fluidized bed. The effects of operation parameters on the surface composition and hydrophilicity have been determined. The oxygen functionalities are formed at the outermost layer of HDPE powder. The hydrophobic surface of HDPE powder has been transformed to hydrophilic surface by the oxygen-plasma treatment in a fluidized bed. The contact angle of the plasma treated HDPE powder decreases linearly with radio frequency (rf) power but increases with O₂ flow rate. Also, the angle decreases with increasing the composite parameter as the total plasma energy down to 6,000 [(W/FM)t]. The contact angle has been correlated with the composite parameter as: \( \theta = 90 - 6.64 \times 10^3 [(W/FM)t]. \)

Introduction

The plasma treatment is a useful tool to modify the surface properties of the polymeric materials from hydrophobic to hydrophilic or inversely from hydrophilic to hydrophobic(1, 2). Low-temperature plasma has been utilized to improve polymer surface properties by introducing new functional groups without change of bulk property. A pure oxygen plasma is known to contain both positive and negative ions, atoms, ozone, and metastables of atomic and molecular oxygen, as well as electrons and a broad electromagnetic spectrum(3). Essentials of the plasma surface treatment technique are electrons, ions, atoms, and radicals in the plasma attack the surface of the polymeric materials, then abstract hydrogen atoms from the polymeric surface to form radical sites at the surface, and finally oxygen atoms react with the radical to form oxygen functionalities including OH, C=O, and COOH groups(4). Therefore, good contact of the polymeric surface with plasma is an important factor for surface modification.

In case of the plasma surface treatment of flat solid materials, such as polymer films, the intimate contact between plasma and solid surface are not critical and conventional plasma reactors, barrel- or jar-type, is good enough for practical use. However, such a conventional reactor cannot be used for powder materials due to the lack of solid mixing. Plasma fluidized bed reactor can provide intimate mixing between the powders and the reactive gas to improve both the reaction rate and the uniformity of the treated surface(5-7).

Experimental variables such as rf power, flow rate of gas and treatment time are known to influence the surface treatment of polymer films(8,9). Usually an external parameter, W/FM in Joules per unit mass of gas is used to express the plasma energy density, where W, F, and M are the rf power, the flow rate of gas, and the molecular weight of the gas. In polymer-forming gas, W/FM may represent properties of plasma polymer(10-12). In this study, the

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effects of oxygen flow rate, discharge power of plasma, and treatment time on hydrophilicity of plasma treated HDPE powders in terms of contact angle have been investigated in a fluidized bed reactor. Also, the parameter \((W/FM)t\) is used to represent the total energy per unit mass of gas which can interpret surface properties of HDPE powder since most of the energy input is consumed by the reaction between oxygen and HDPE\((13,14)\).

**Experimental**

Experiments were carried out in a fluidized bed of 34 mm-ID x 0.8 m-high Pyrex glass column as shown in Fig. 1. High density polyethylene (HDPE-231 μm) powder was fluidized by oxygen gas. The gas flow rate was regulated and measured with a mass flow controller. The particles were supported on a sintered plate distributor which is situated between the main column and a distributor box. The reduced pressure in the bed was maintained by means of a vacuum pump. Entrained particles were captured by a particle trap.

An inductively coupled electrode (4.8 mm-OD, 6 turns) for glow discharge at 13.56 MHz (rf) frequency was placed at a distance of 90 mm from the gas distributor and was connected to an auto matching network and an rf power generator. To prevent overheating of the electrode, cooling water was supplied to the electrode.

Initially, HDPE powders were dried in vacuum oven at 60°C overnight. This dried HDPE powder of 14.2 g (apparent density; 956 kg/m³; size; 60/70 mesh - 0.250/0.212 mm) loaded into the reactor and gas in the reactor was evacuated by a mechanical pump to 1.33 Pa. Oxygen gas was injected into the distributor box and the desired gas flow rate, pressure, and power were adjusted to start glow discharge. After the desired treatment time was over, plasma was cut-off, and the samples were kept in the reactor for more than 10 min at the given gas flow rate.

The high density polyethylene powders were treated by plasma in the fluidized bed at rf power of 50 to 200 W, oxygen flow rate of 15.2 to 28.4 sccm, and treatment time of 1 to 13 h at a pressure of 133 Pa.

IR spectra of the plasma-treated HDPE powders were recorded on a Bomem FTIR spectrometer with a diffuse reflector. The spectral resolution was 2 cm⁻¹, and 100 scans were