Dynamic Wetting of Plasma-Treated Polypropylene Nonwovens

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Received 1 January 2006; accepted 2 March 2006
DOI 10.1002/app.24459
Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Various techniques have been employed to improve the wettability of polypropylene materials for a wide range of applications. In this study, polypropylene nonwovens were treated in oxygen plasma for improving water adsorption properties. The effects of plasma treatment on wetting and water adsorption behavior were characterized using dynamic contact angle measurements and dynamic sorption measurements. The introduction of hydrophilic groups was detected by attenuated total reflection–Fourier transform infrared spectroscopy. The plasma treatment roughened the fiber surface revealed by atomic force microscopy. The roughened and hydrophilic surface resulted in the change in advancing and receding contact angles. The dynamic sorption measurements also examined the water adsorption behavior of the materials. The investigation revealed that plasma treatment could significantly improve the water adsorption properties of polypropylene nonwovens. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 2157–2160, 2007

Key words: surface; adsorption; polypropylene; plasma treatment; AFM; contact angle

INTRODUCTION

Nonwoven materials are consolidated fibrous materials, which are different from the conventional textile fabrics. Nonwovens are based on webs of individual fibers. These fibrous webs, which contain small pores, facilitate the transport of liquids into the materials and retain the liquid after sorption.[1] Polypropylene nonwovens are increasingly being used in many industries associated with wetting and adsorption because of the excellent physical and chemical properties of polypropylene fibers compared to other fibers.[2]

For the applications of polypropylene nonwovens in sorption-related industries, polypropylene materials have to be modified to improve the wettability of the materials. Surface modification by plasma treatment has opened up new possibilities in relation to wettability and adsorption of nonwoven materials.[3] Plasma is a low-temperature glow discharge or a low-pressure partially ionized gas, consisting of large concentrations of excited atomic, molecular ionic, and free radical species. Plasma surface treatment causes changes to a limited depth; bulk properties of even the most delicate materials remain unchanged.[4]

In this study, polypropylene nonwovens were treated in oxygen plasma for improving the water adsorption properties. The effect of plasma treatment on wetting and water adsorption behavior was characterized using dynamic contact angle measurements and dynamic sorption measurements.

EXPERIMENTAL

Preparation of materials

Polypropylene nonwovens used in this study were polypropylene needlepunched materials. The details are listed in Table I. The samples were washed in ethanol followed by rinsing twice in distilled water, and then the samples were dried at 50°C for plasma treatment.

Plasma treatment

The polypropylene nonwoven samples were treated with oxygen in Europlasma CD 400M/PC laboratory system. The treatment parameters are listed in Table II.

Surface characterization

AFM observation

Scanning probe microscope (SPM), particularly in the form of atomic force microscopy (AFM), has been increasingly applied in polymer research.[5] The AFM

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Contract grant sponsors: Chinese Ministry of Education (No. 106089), Southern Yangtze University; contract grant number: 2005LYY014.

Journal of Applied Polymer Science, Vol. 104, 2157–2160 (2007)

TABLE I
Polypropylene Nonwovens

Materials	Processing	Mass per square meter (g/m^2)	Thickness (mm)	Fiber diameter (μm)
NP1	Needlepunching	220	2.25	16
NP2	Needlepunching	220	2.27	32

used in this work was a Digital Instrument Nanoscope III. Scanning was carried out in tapping-mode AFM, and all samples were scanned at room temperature.

ATR-FTIR spectroscopy

Fourier transform infrared (FTIR) analyses were conducted on a Perkin–Elmer 1720X attenuated total reflection–Fourier transform infrared (ATR-FTIR) spectrometer. The spectra were recorded with Harrick ATR attachment, using KRS-5 crystal ($50 \times 10 \times 2$ mm^3), at an incident angle of 45° with 25 reflections.

Dynamic contact angles

The dynamic contact angle measurement of individual fiber was performed using a CDCA-100F produced by Camtel in UK. The dynamic contact angles were determined by Wilhelmy technique,[6] where a solid sample was immersed and withdrawn into and out from a liquid, while simultaneously measuring the force acting on the solid sample at 20°C. The advancing and receding contact angles could then be determined from the obtained force curve.

Water adsorption

All tests were performed at (20 ± 1)°C. All samples were conditioned at (20 ± 1)°C and at (65 ± 2)% relative humidity. Each sample was cut into the size of 1 cm × 4 cm with sharp scissors. The specimens were placed a CDCA-100F adsorption apparatus. When the specimen was immersed into water at 20°C, the water adsorption was detected and recorded. The water adsorption was plotted as a function of time.

RESULTS AND DISCUSSION

Surface morphology and chemistry

The series of images obtained by AFM show the change in surface morphology of the PP fibers in the nonwovens before and after plasma treatment. The fibril structures of the untreated PP fibers in NP1 material is observed by AFM examination, as illustrated in Figure 1(a). It can also be seen from the image that the fibrils are oriented in the direction of the fiber axis. The fibril structures are believed to be formed by fiber drawing during fiber processing.[7] Figure 1(b, c) reveals the effects on the PP fiber surface of oxygen plasma treatments for 30 and 60 s, respectively. The surface of the fiber surface in NP1 nonwoven is altered by plasma

TABLE II
Plasma-Treatment Parameters

Gas	Oxygen
Power (W)	100
Time (s)	30 s, 60 s
Gas flow (L/min)	0.3

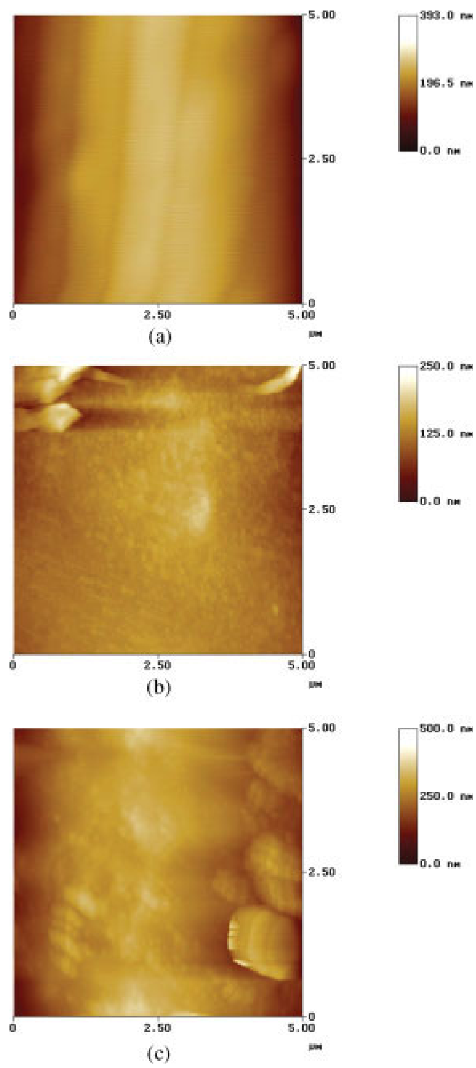

Figure 1 SPM images of PP fibers (NP1): (a) untreated; (b) plasma treated for 30 s; (c) plasma treated for 60 s. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

treatment. Aggregate structures with various sizes can be seen on the fiber surface after plasma treatment for 30 s, as displayed in Figure 1(b). The fibril structures are not visible any more. Oxygen plasma treatment for 60 s further roughens the fiber surface, forming pit-like structures, as revealed in Figure 1(c). The presence of pit structures can be attributed to an etching effect of the plasma treatment. The similar phenomenon is also observed on the fibers in NP2 nonwoven.

Oxygen plasma treatment significantly affects the surface chemistry of polypropylene nonwovens. Polypropylene has only C—C and C—H functional groups, and the FTIR adsorption spectrum contains sharp, well-defined adsorption bands,[8] as presented in Figure 2. The plasma treatment leads to the appearance of new adsorption peaks at 1715 cm^{-1}, which can be attributed to C=O stretching band in COOH carboxyl groups.[9] It is also observed that the concentration of carbonyl functional group is increased with the increase in treatment time, as exhibited in Figure 2.

Dynamic contact angles and water adsorption

The effect of plasma treatment on surface wettability is revealed by dynamic contact angle measurement. It can be seen from Figure 3(a) that the untreated fibers have advancing contact angles over 95° and receding contact angles about 85°. The untreated fibers have an obvious hysteresis of about 10°, because of the surface roughness, as illustrated in Figure 1(a). It can also been seen from the curves that the fibers obtained from NP1 and NP2 have very similar contact angles, even they have a big difference in fiber diameter. From the results in Figure 3(b, c), it can be concluded that the advancing and receding contact angles are considerably reduced after treatment by oxygen plasma. The advancing contact angles are reduced to about 65°, and the receding contact angles are lowered to about 45° after plasma treatment for 30 s. It is observed that the hysteresis is increased from about 10° for the untreated fibers to 20° for the treated fibers. This phenomenon is attributed to roughening of the fiber surface by plasma treatment.[10] It can also be seen from Figure 3(b) that the fibers with smaller diameter show lower contact angles than do thicker fibers. This phenomenon is even more obvious when the treatment is extended to 60 s, as revealed in Figure 3(c). The plasma treatment for 60 s causes a further decrease of contact angle of the fibers, as presented in Figure 3(c). The introduction of hydrophilic groups onto the fiber surface and the roughening of the surface are believed to be the main reason for the decrease of advancing and receding contact angles.

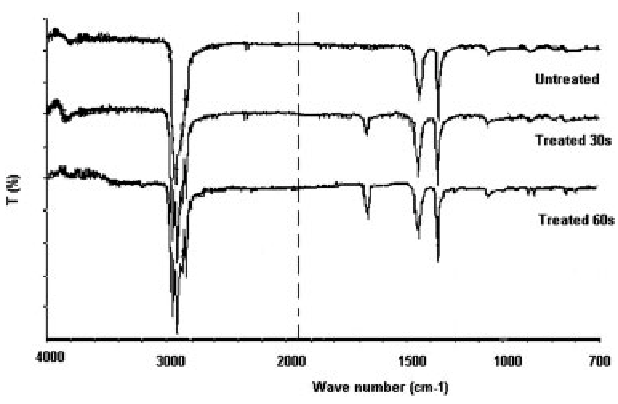

Figure 2 FTIR spectra of PP fibers.

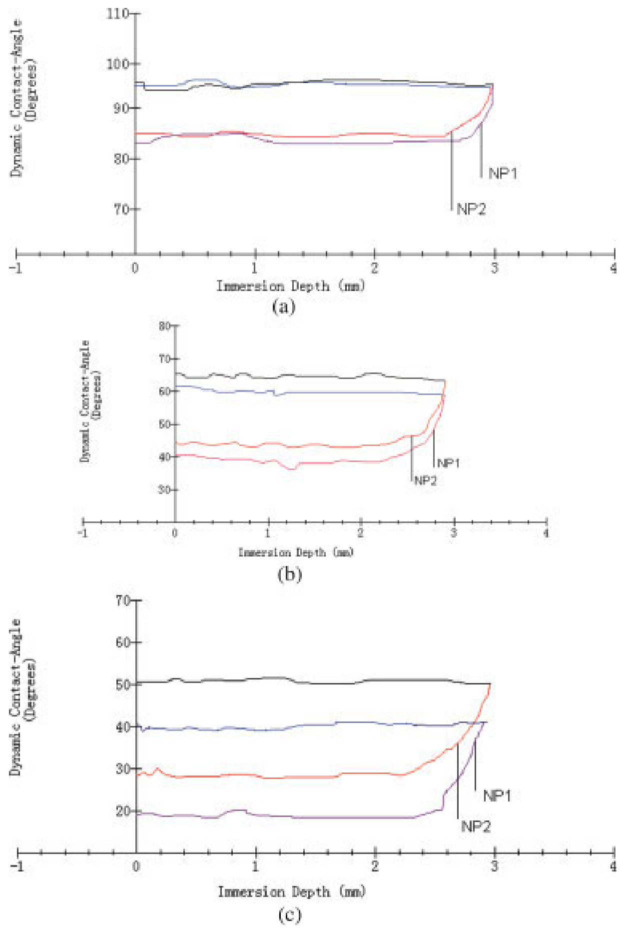

Figure 3 Dynamic contact angles of PP fibers: (a) untreated; (b) plasma treated for 30 s; (c) plasma treated for 60 s. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Before plasma treatment, the polypropylene nonwovens do not show any water adsorption, as presented in Figure 4(a). The minus adsorption indicates the effect of the buoyancy of water, which pushes the materials upwards.

Plasma treatment significantly improves the water sorption properties of polypropylene needlepunched nonwoven, as illustrated in Figure 4(b). It can be seen from the results that the water adsorption process follows the similar pattern. At the initial stage of adsorption, amount of water absorbed increase very fast. It can

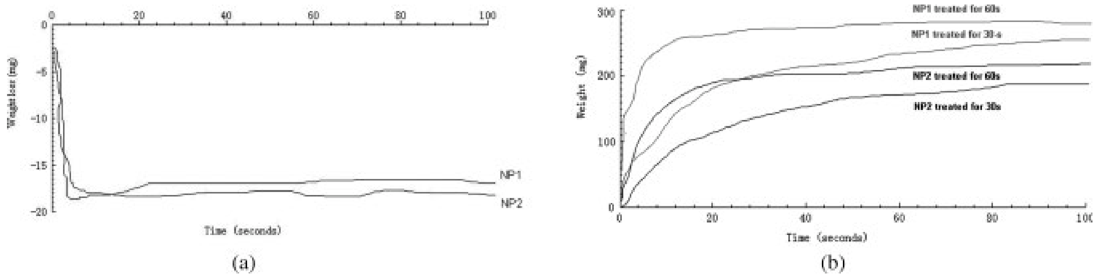

Figure 4 Water adsorption of needlepunched nonwoven: (a) untreated; (b) plasma treated.

also be seen from Figure 4(b) that the initial adsorption rate is increased as the treatment time is increased from 30 to 60 s. This is attributed to the decrease in advancing contact angle with the increase in treatment time.

The adsorption curves reveal that the adsorption rate is gradually decreased as sorption time is extended. The increase in treatment time results in an increase in the total amount of water absorbed. When the treatment time is extended from 30 to 60 s, the adsorption is increased to about 280 mg from 240 mg for NP1 and to 220 mg from 190 mg for NP2. After the plasma treatment, NP1 nonwoven shows better adsorption properties than those of NP2 material. The smaller diameters of fibers in NP1 contribute to the increase in water adsorption. The finer fibers provide more capillary channels for water adsorption.

CONCLUSIONS

The oxygen plasma treatments significantly alter the wettability of the polypropylene fibers. Oxygen plasma treatments introduce the polar groups on fiber surfaces, and so change the wetting behavior of the fibers. Plasma treatments also change the dynamic contact angles of the fibers and adsorption behavior of the nonwoven materials. Plasma treatment is, therefore, likely to become increasingly valuable in textile industries.

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