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Preparation and characterization of superhydrophobic coatings

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To obtain a surface with water-repelling and self-cleaning abilities attracts much interest in the nanotechnology age. In this study, a perfluroalkyl surfactant (perflurodecanoic acid, PFDA) was coated on nano-sized boehmite surfaces to obtain hydrophobic nano-particles via surface modification. Then the hydrophobic boehmite was coated on a transparent plastic film using spin-coating to obtain a hydrophobic film. The effects of various amounts of hydrophobic polymer on the adsorption behavior and contact angle for hydrophobic properties were investigated using FTIR, and a contact-angle meter. The adsorption of PFDA onto the surface of boehmite takes place through ionic bonding. The contact angles of the thin film prepared by spin coating PFDA-modified-boehmite on glass substrates were above 150^o as the adsorption of PFDA onto the surface of boehmite reached saturation.

Key words: superhydrophobicity, spin coating, boehmite, contact angle.

Introduction

Superhydrophobic surfaces that have a water contact angle > 150^o have attracted much interest from both industry and in fundamental research. Such surfaces have been widely used in practical applications such as windshields of automobiles, eye glasses, glass covers for solar cells, satellite dishes, and roofing.

The superhydrophobicity is obtained by a combination of an extremely hydrophobic surface and a high surface roughness [1-2]. It has been reported that perfluroalkyl polymer can provide a surface with a very low surface energy and display very good hydrophobicity and oleophobicity [3]. However, the maximum water contact angle a highly effective fluorinated surface can attain is approximately 120^o. To increase the water contact angle above 150^o, a substantial surface roughness is required. The surface roughness can be precisely controlled by adding different amounts of nano-sized particles to the coating [4-5].

In this study, a perfluroalkyl surfactant was coated on the nano-sized boehmite surfaces to obtain hydrophobic nano-particles via surface modification. Then the hydrophobic boehmite was coated on a transparent plastic film using spin-coating to obtain a hydrophobic film. The bonding mechanisms between the PFDA and boehmite were studied using diffuse reflectance Fourier transformation infrared spectroscopy to explain the effects of various amounts of hydrophobic polymer on the adsorption behavior and contact angle for the hydrophobic properties.

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Experiment

The boehmite powders (DISPAL 18N4, Condea Chemie, Hamburg, Germany) with the addition of PFDA (97%, Lancaster) and ethanol (99.5%) were mixed for 3 h. The PFDA concentrations varied from 1-100% of the solid weight. After mixing, the surface modified boehmite powders were subsequently washed 2 times with ethanol and once with hexane to remove the non-adsorbed PFDA. Then the slurry was dried at 80 ^{o}C for 12 h. The boehmite powders modified with different PFDA concentrations are given as Table 1. The chemical characteristics of boehmite powders with and without PFDA treatment were determined using Fourier transform infrared spectroscopy (EQUINO 55, Brucker, Germany). All the films were made by spin-coating (900 rpm) boehmite sols (15 wt% ethanol solution) on glass substrates. The glass substrates were rinsed with a solution of 0.1 M H_2SO_4 and H_2O_2 (1 : 1) for 60 minutes at 50 ^{o}C and rinsed with deionized water before coating. After coating, the samples were dried under ambient conditions for 5 minutes and then heat treated at 80 ^{o}C in air for 12 h to remove the residual solvent. Contact angles with water were measured with a contact angle meter (OCA 15, Dataphysics, German). The water droplet size used for the measurements was ~10 μl. The contact angles were measured at five different points.

Table 1. The notation of the boehmite powders modified with different PFDA concentrations

Samples	B0	B1	B5	B10	B20	B50	B100
PFDA concentration (wt%)	0	1	5	10	20	50	100

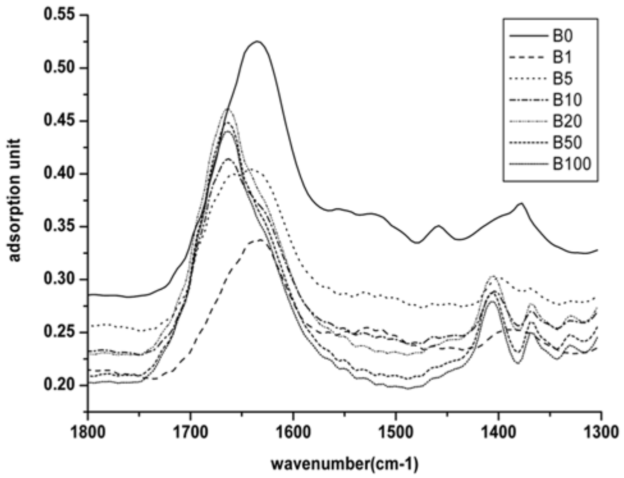

Fig. 1. FTIR spectra of the boehmite samples in the region 1300-1800 cm^{-1} before and after the addition of various amounts of PFDA.

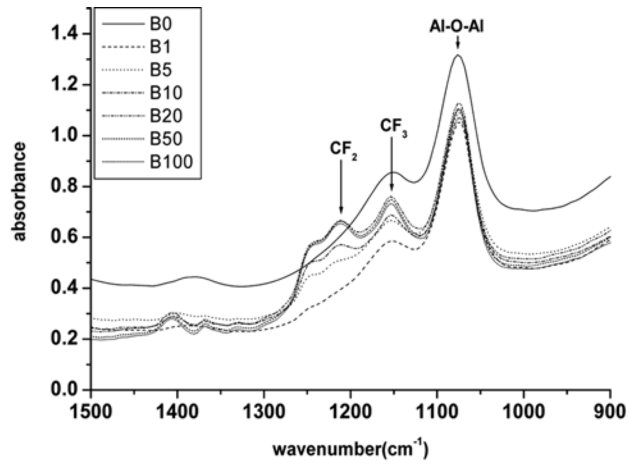

Fig. 2. FTIR spectra of the boehmite samples in the region 900-1500 cm^{-1} before and after the addition of various amounts of PFDA.

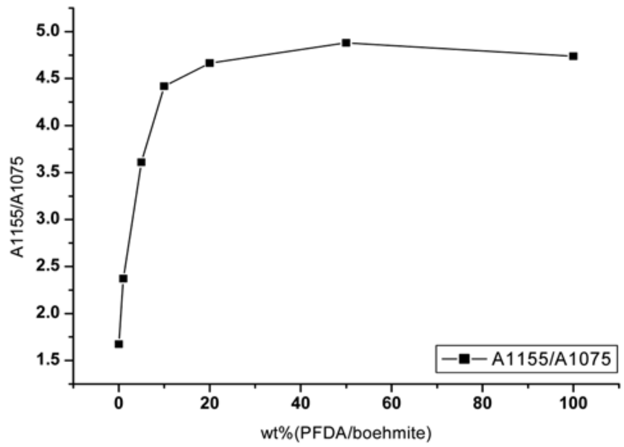

Fig. 3. Relative integrated intensities of CF_3 bonding around 1150 cm^{-1} with respect to the absorption peak (Al-O-Al) belonging to boehmite at 1075 cm^{-1} (A_{1155}/A_{1075}) obtained from the PFDA-treated boehmite samples are plotted against the amount of PFDA.

Results and Discussion

Adsorption of PFDA

The FTIR spectra of the boehmite in the region 1300-1800 cm^{-1} before and after the addition of various amounts of PFDA are shown in Fig. 1. It is observed that the intensity of the band at 1378 cm^{-1} which relates to the O-H group of boehmite decreases and new peaks at 1405, 1367 cm^{-1} due to COO- stretching and 1330 cm^{-1} due to C-F stretching are observed. Moreover, the bands at 1765 and 1705 cm^{-1} belonging to C = O stretching in PFDA shift to around 1670-1650 cm^{-1}, which may be caused by the interaction of the -COOH group of PFDA with cation sites at the surface of boehmite as reported by Ha *et al*. [6]. The above results confirm that the adsorption of PFDA onto the boehmite surface takes place through the ionic bonding between the COO- group in PFDA and the Al sites of boehmite.

The FTIR spectra of the boehmite in the region 900-1500 cm^{-1} before and after the addition of various amounts of PFDA are shown in Fig. 2. New bands at 1150 and 1215 cm^{-1} due to CF_3 and CF_2 stretching from the PFDA are observed after addition of PFDA. The relative integrated intensities of CF_3 bonding around 1150 cm^{-1} with respect to the absorption peak (Al-O-Al) belonging to boehmite at 1075 cm^{-1} (A_{1155}/A_{1075}) obtained from the PFDA-treated boehmite are plotted against the amount of PFDA in Fig. 3. The A_{1155}/A_{1075} value in Fig. 3 can be used as an indicator of PFDA adsorption onto the boehmite surface. The amount of PFDA adsorbed onto the surface of boehmite reached saturation as the addition amount of PFDA added was about 15 wt%.

Contact angle

The profiles of water drops on the coating prepared by spin coating boehmite sol modified with different amounts of PFDA on a glass slide are shown in Fig. 4. The water contact angles for the coatings on a glass slide depend on the adsorption of PFDA onto the surface of the boehmite. The contact angles of the thin film prepared by spin coating PFDA-modified-boehmite on a glass substrate increased with an increase in the addition of PFDA and were above 150° as the adsorption of PFDA onto the surface of boehmite reached saturation as shown in Fig. 5.

Conclusions

1. The adsorption of PFDA onto the surface of boehmite takes place through ionic bonding.

2. The adsorption of PFDA onto the surface of boehmite reached saturation as the addition amount of PFDA added was about 15 wt%.

3. The contact angles of the thin film prepared by spin coating PFDA-modified-boehmite on glass substrates were above 150° as the adsorption of PFDA onto the surface of boehmite reached saturation.

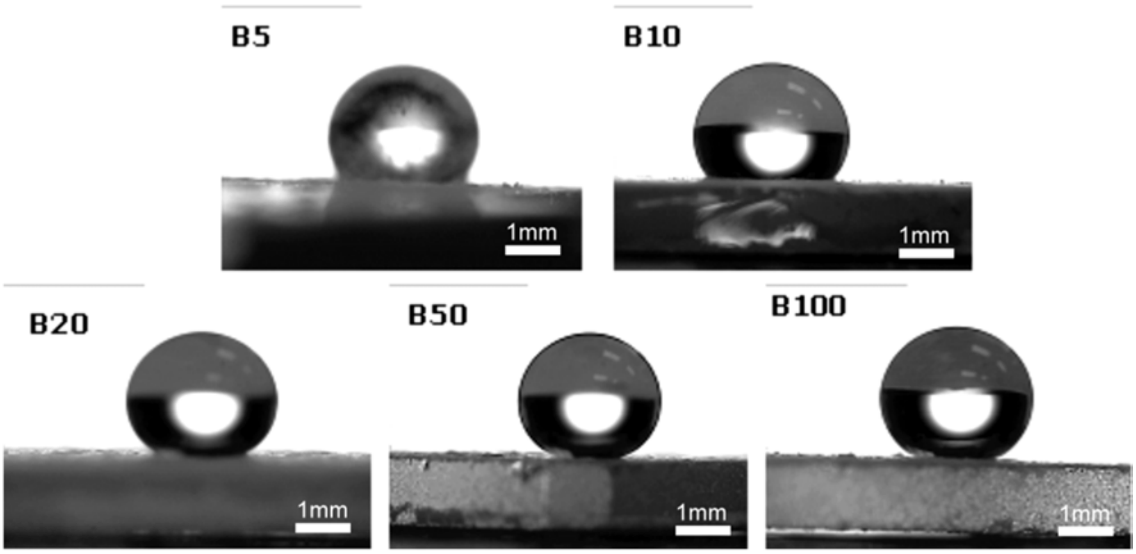

Fig. 4. Profiles of water drops on the coating prepared by spin coating boehmite sol modified with different amounts of PFDA on a glass slide.

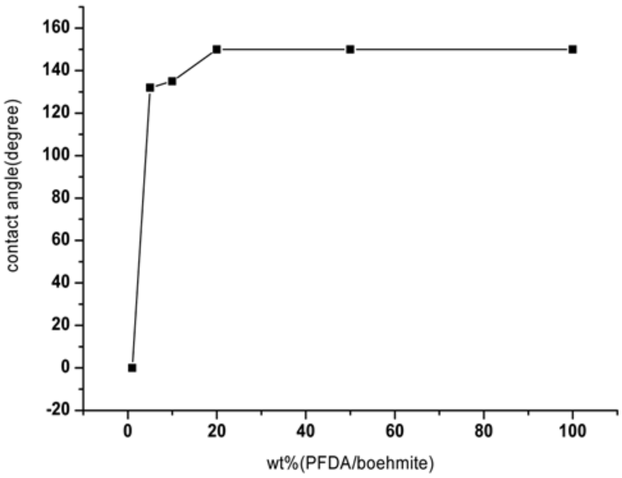

Fig. 5. Variation of contact angles of the thin film prepared by spin coating PFDA-modified-boehmite on a glass substrate with the addition of PFDA.

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