



Role of nanoparticles in phase separation and final morphology of superhydrophobic polypropylene/zinc oxide nanocomposite surfaces

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ABSTRACT

In this work, phase separation process was revisited to study the effect of nanoparticles in acceleration of phase separation and formation of hierarchical structures. Superhydrophobic surfaces were prepared using polypropylene (PP) and the corresponding nanocomposites containing zinc oxide (ZnO) nanoparticles through a typical solution casting method. The wettability and morphological behavior of the surfaces were investigated via water contact angle (WCA) measurements and scanning electron microscopy (SEM), respectively. It was found that upon introduction of ZnO nanoparticles into the pure PP, the obtained surfaces have become superhydrophobic with WCAs above 150° and sliding angles below 10°. Calcination of ZnO nanoparticles was exploited to explicate the unexpected significant loss in superhydrophobicity of the sample loaded with high ZnO content. Crystallization behavior of the samples were also investigated via differential scanning calorimetry and correlated to superhydrophobicity of the surfaces. X-ray photoelectron and Fourier transform infrared spectroscopies were also utilized to further characterize the samples. An attempt was also made to present a more clear mechanism for formation of hierarchical structures which are responsible for superhydrophobicity. Likewise, the so far proposed mechanisms for formation of micro/nano roughness on the superhydrophobic surfaces were reviewed as well.

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1. Introduction

For over a decade, superhydrophobic surfaces have gained significant attention within the scientific community as well as different industries because of their unique water-repellent, self-cleaning, anti-fouling, anti-icing, anti-sticking, anti-contamination properties, and their promising potential for practical applications [1–7]. Based on the general definition, surfaces with water contact angles (WCA) above 150° are considered as superhydrophobic. However, a more exact definition of a superhydrophobic surface must include the extent of hysteresis (difference between advancing and receding angles) which must be lower than 10° [8]. In nature, from which the superhydrophobicity phenomenon has been inspired, the lotus leaves are the most famous examples of a superhydrophobic surface with WCA larger than 150° and a sliding

angle less than 10°, on which water droplets roll off effortlessly. It is now well-established that this phenomenon is originated from the combination of micro/nano hierarchical structures and low surface energy materials on the leaves' surfaces [9]. There have been reported numerous methods for preparation of superhydrophobic surfaces as evidenced by several review articles on the fabrication methods of such surfaces [10–13]. These methods are mainly divided into two approaches: either make a rough surface from a low surface energy material or modify a rough surface with a material of low surface energy [14].

Polypropylene (PP) is an intrinsically hydrophobic polymer with a melting temperature about 170 °C and shows various morphologies based on the employed processing conditions. PP is one of the most used polymers in many commodity as well as industrial applications, which offers a combination of outstanding physical, chemical, mechanical, and thermal properties not found in any other thermoplastic. However, its application in the coating industry has been limited because PP has a thermoplastic nature and the final film could only be formed via the evaporation of the

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solvent. On the other hand, for polymers to reach the properties needed for performance coatings, they must achieve a very high molecular weight and consequently a very high viscosity. Therefore, only low concentrations are possible for most of the application techniques leading to a limited working window for PP coatings [15]. One of the important applications has been studied in detail [7] where a superhydrophobic PP coating was fabricated with improved anticoagulation properties at the blood-polymer interface. Several researches have been done on superhydrophobic PP surfaces within the recent years. Rasilainen et al. [16] have prepared micro pit PP surfaces by injection molding. In another work, they have prepared PP surfaces with anisotropic hierarchical structures by template method [17]. Puukilainen et al. [18] have also fabricated superhydrophobic polyolefin surfaces by injection molding where microstructured aluminum and dual-structured anodized aluminum oxide (AAO) mold inserts were used to pattern surfaces. All the methods mentioned above have been successfully employed to fabricate stable superhydrophobic surfaces made of PP; however, the experimental could be rather costly and also complicated as compared to some other methods such as those based on phase separation. In 2003, Erbil et al. [19] have reported a simple and inexpensive but effective way to fabricate superhydrophobic surfaces through a suitable selection of solvents, non-solvents, and drying temperature. They have chosen PP because of its intrinsic hydrophobic nature and the fact that PP is a simple and common plastic. Since then, numerous researchers have used this method for a wide range of polymers [20], polymer blends [21], and nanocomposites [22]. For instance, Lv et al. [23] reported the successful modification of PP hollow fibers into superhydrophobic membranes by using a proper combination of non-solvents via phase separation method presented by Erbil. PP has also been used, by Rioboo et al. [24], as a model coating for a quantitative analysis on the drop impact behavior in superhydrophobic surfaces. In another research [25], various PPs, with different tacticities and molecular weights, have been used in fabrication of superhydrophobic coatings via simple solution casting and dip coating methods. The conclusion was that, in all cases, superhydrophobic surfaces were obtained even for atactic PP, however through variation of the quantity of the coated material. In a recent study by Liu et al. [22], superhydrophobic PP surfaces were fabricated via phase separation method using halloysite nanotubes (HNTs), instead of non-solvents, which can accelerate the phase separation process by playing the role of heterogeneous nuclei for PP.

Zinc oxide (ZnO) is an important semiconductive material and has drawn much attention because of its characteristics in optics, photonics, electronics, and biological applications [26]. In addition, ZnO nanoparticles have been demonstrated to improve mechanical, optical, and thermal properties and crystallization extent in polymers. Recently, ZnO nanoparticles have been incorporated into polydimethylsiloxane in order to fabricate superhydrophobic and superoleophilic coatings [2,27]. In another work, Zhu et al. [28] have prepared intelligent superhydrophobic ZnO surfaces possessing water adhesive reversibility.

In this study, the phase separation method is employed for achieving superhydrophobicity on PP surfaces. According to the literature, there have been many reports on superhydrophobic surfaces using a vast range of polymers via the phase separation method; however, to the best of our knowledge, there is very limited literature on polymer/ZnO superhydrophobic coatings and no report has been published on PP/ZnO superhydrophobic coatings with self-cleaning behavior via phase separation method. Additionally, an attempt was made to present a more clear mechanism of the phase separation process and formation of a micro/nanostructure on the surface.

2. Material and methods

2.1. Materials

The materials used in this work were of commercial grades. PP (SEETEC H5300, density = 0.9 g/cm³) was obtained from LG corporation with melt flow rate of 3.5 g/10 min which was measured under a load of total mass of 2.16 kg at a temperature of 230 °C. Xylene (Merck) was selected as the solvent to dissolve the granular PP. Zinc oxide (ZnO) nanoparticles, particle average size of 20–30 nm and specific surface area of 35–50 m²/g, were kindly supplied by TECNAN.

2.2. Preparation of coatings

The superhydrophobic PP/ZnO nanocomposite surfaces were prepared through solution casting method. A typical procedure was as follows: for the pure PP coating, 0.5 g PP granules were dissolved completely in 30 mL xylene at around 120 °C. Whereas for nanocomposite samples, different contents of dried ZnO nanoparticles (5, 50, 100, 200, and 300 mg) were first vigorously stirred for 60 min in 30 mL xylene, and then ultrasonicated for 45 min. Subsequently, 0.5 g PP granules were introduced into the solution and temperature was raised to 120 °C. After that, these PP/ZnO solutions were magnetically stirred with a rotation speed of 800 rpm for 60 min. Finally, the solutions were casted on the previously cleaned glass slides using a spin coater and dried at different temperatures. For simplicity, the prepared samples are named PP5, PP50, PP100, PP200, and PP300 in which the number accounts for the mass of ZnO nanoparticles in the coatings.

2.3. Characterization

Morphologies of the coating surfaces were evaluated on a scanning electron microscope (KYKY-EM3200) operated at 25 kV. To increase the resolution for SEM observation, the samples were plated with gold coating in order to render them electrically conductive. A video-based contact angle measurement system (OCA 15, DataPhysics Instruments GmbH, Germany) was employed to determine the WCAs of the samples. The WCA measurement of each sample was carried out at least three times across the sample surface using the sessile drop method by dispensing 4 μL drops of de-ionized water on the sample surfaces. All WCA measurements were taken under ambient laboratory conditions at a temperature of about 25 °C. X-ray photoelectron spectroscopy (XPS) analysis was performed by using XPS spectroscopy with a monochromatic AlK_α X-ray source (1486.6 eV photons), operated at 180 W (12 kV and 15 mA) and under ultra-high vacuum conditions. The as-received and calcined ZnO nanoparticles were also characterized using FT-IR (Bomem, Canada). About 1 mg of the samples were milled with KBr and formed into tablets. The data were recorded in a band range from 400 to 4000 cm⁻¹. Non-isothermal crystallization of the samples was carried out on an Elmer Diamond differential scanning calorimeter (DSC). All the samples were first heated to 200 °C for 3 min to remove any thermal history, and then cooled to 55 °C at 10 °C/min (cooling scan). Crystallization peak temperatures were obtained from the cooling scan.

3. Results and discussion

3.1. Wettability of the coatings

All the fabricated polymer coatings exhibited white porous appearances and very strong hydrophobic characteristics. As mentioned earlier, isotactic PP is a hydrophobic polymer and WCA of

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