



Investigation of wettability and optical properties of superhydrophobic polystyrene-SiO₂ composite surfaces



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ABSTRACT

Polystyrene coatings with varying SiO₂ nanoparticle contents were prepared by spin coating technique and their wettability and optical properties were investigated. Superhydrophobic surfaces were achieved with water contact angle values higher than 168° and optimum nanoparticle content was indicated for the highest light propagation and water repellency. Higher CAH values were calculated for hydrophobic surfaces due to their rough surface morphology, however in the case of superhydrophobic surfaces, CAH values were calculated lower. Herein absorption occurred below 300 nm, beyond that all samples displayed wide range of transparency as their nanoparticle content change. In general, the transmittance and reflectance values of the films increased with the decrease in the silica content for the almost same coating thicknesses. Due to the aggregation of the nanoparticles, spectral transmittance values of films that have silica content larger than 85% SiO₂ are lower than the others.

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1. Introduction

Preparation of superhydrophobic surfaces which demonstrate high water contact angles more than 150° and contact angle hysteresis (CAH) values lower than 10° attracts high attention from both scientific community and industry due to their highly desired advantages such as; anti-fouling, oil-water separation, anti-bacterial, anti-icing, self-cleaning or flow enhancement [1–11]. Superhydrophobic behavior depends on surface roughness as well as surface chemical composition. It is well known that hydrophobic surfaces with roughness at micro/nanometer scale display superhydrophobic behaviors [12–14].

Nanoparticles, especially silicon dioxide (SiO₂), are being researched comprehensively [11,15–20]. Yilgor et al. [12] developed a simple and general method for the preparation of superhydrophobic surfaces which is applicable to a large number of polymers. In their study, multi-step spin coating process was applied where hydrophobic silica was coated onto a polymer surface from dispersion in THF. They demonstrated in all cases that superhydrophobicity can be obtainable. SiO₂ is also used for surfaces which demonstrate multiple properties. Zhang et al. [19]

combined superhydrophobicity and the magnetic property of γ -Fe₂O₃. They prepared superparamagnetic-superhydrophobic films and characterized thoroughly which indicated these nanocomposites can be easily magnetized and demagnetized. He et al. [16] demonstrated that the formation of networks of nanoparticle aggregates is more important than root-mean-square (RMS). Cengiz et al. [20] investigated water and oil repellencies of flat and rough perfluoro-styrene composite surfaces with varying silica contents. They demonstrated the contact angle increase with the increase of silica content due to the trapped air which minimizes the contact area of the droplet on solid surface. Water contact angle values were measured around 170° on their composite surfaces, however, in the case of hexadecane drop, contact angle values of composite surfaces showed a decrease with the increase of silica concentration. This result indicated that hexadecane drop can penetrate between the roughnesses and no trapped air could be present. Milionis et al. [21] presented a simple, water-based and cost effective spray-coating method to produce superhydrophobic and oleophobic coatings by using nanopowder fumed silica dispersed in an acrylic fluorochemical. They reported that wettability properties could be controlled by changing nanoparticle ratio in solution and increase in the surface roughness lead to a wetting transition to Cassie-Baxter state where liquid droplets were restrained against penetrating between the roughnesses on surface. Milionis et al. [22] also reported another

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research on 'non-stick' superhydrophobic surfaces by utilizing biodegradable biopolymers to overcome 'persistence' problem where wetting properties can be controlled by changing the ratio of biopolymer to nanoparticle. Resistance of these coatings against abrasion was another problem which should not be treated lightly. In order to solve this problem several approaches were presented. Davis et al. [23] reported an electrospun composite nano-fiber mats which show a new mechanism to be exploited for wear damage. It was mentioned that for a variety of the liquids, droplets were observed to peel away the composite fibers revealing similar fibrous network. This method can be manipulated for new approaches to create more durable repellent surfaces.

In order to obtain superhydrophobicity, surface chemical composition alone is not enough and surface roughness must be introduced which leads a decrease of light propagation. Therefore preparation of an applicable superhydrophobic coating requires an optical investigation. In our previous study [24], various flat polymer coatings were used to study their optical and wettability properties. We found that light propagation can fluctuate even with changing only polymer types and this indicated that choosing polymer type is another important factor not just in the terms of liquid-solid interaction but also optical properties. Yang et al. [25] prepared transparent superhydrophobic nanocomposites films using polystyrene functionalized carbon nanotubes. Coated samples demonstrated water contact angles around 160° and also with high transparency at visible region. It was stated that this technology is applicable for developing self-cleaning coatings optoelectronic or electromagnetic interference shielding. Cao and Gao [26] reported a facile process to fabricate transparent superhydrophobic surfaces using polystyrene and silica nanoparticles. They demonstrated highly transparent, with approximately 90% transparency, superhydrophobic surfaces using layer by layer assembly technique to create porous network as coating method. Ebert et al. [27] prepared transparent superhydrophobic glass substrates using SiO_2 , ZnO and ITO nanoparticles. They found that transparent superhydrophobic surfaces with wear resistance can be fabricated with a broad range of materials.

Herein, superhydrophobic PS- SiO_2 nano particle composite surfaces were prepared with varying silica contents to investigate their wettability and optical properties. Depending on silica contents surface wettability was changed; increase in silica content also caused an increase in water contact angle (CA) values. Surfaces that have silica content larger than 50% SiO_2 exhibited superhydrophobic behavior with water CA values larger than 168° and these surfaces have low CAH values. Such composite surfaces are widely used in various industrial applications. It is necessary not to compromise the optical properties while improving the wettability of the surfaces. The average transmittance values of PS-silica composite films ranged from ~40% to ~90% in the visible range. It can be concluded that the increase in the SiO_2 concentration decreased the transmittance values of the coatings. PS-silica composite films can be switched between opaque to transparent states by changing the SiO_2 concentration, especially in the visible range, since absorption edges of the films are shifted to lower wavelength with the decrease in SiO_2 concentration. Surfaces demonstrated high transparency, however transparency of superhydrophobic surfaces decreased, as expected, with the increase of surface roughness.

It is important to prepare water repellent surfaces with high transparencies and these type of surfaces can be utilized for numerous applications. In this work, we prepared superhydrophobic PS- SiO_2 composite films with high transparencies however, transmittance of composite films slightly deteriorated with the increase of nanoparticle content, due to the increased SiO_2 nanoparticle aggregates.

2. Materials and methods

2.1. Materials

Polystyrene (PS) polymer with a M_w of 234.893 and PDI of 2.12, (both values were found from GPC analysis) was purchased from Styrolution; HDK H18 hydrophobic pyrogenic silica nanoparticles, with BET surfaces area of approximately 120 m^2/g , were purchased from WACKER. Chemicals and toluene was purchased from MERCK and all chemicals were directly used as received.

2.2. Preparation of polymer films

Glass slides were cleaned in chromic acid solution at room temperature, than rinsed with distilled water and dried at 100°C . Cookson Electronics G3P-8 Spin coater was used for the preparation of the thin films. Hydrophobic fumed silica particles were agglomerated in polymer solution, to overcome this problem, SiO_2 nanoparticles were firstly dispersed in toluene solvent by using Bandelin HD2200 ultrasonic homogenizer. PS polymer was dissolved in toluene with a concentration of 50 mg/ml . Nanoparticles dispersed in toluene were mixed with the PS solution with same volume and then the mixture was dispersed again by applying 2 cycles and 90% amplitude for 10 s. Nanoparticle concentration in PS solution was varied between 2.5 mg/ml to 50 mg/ml . PS + SiO_2 nanoparticle solution was spin coated on glass slides at 3000 rpm and 2 μl solutions were poured on each glass slides during the coating process. Coated samples were dried at room temperature overnight.

2.3. Contact angle measurements

KSV Attension Theta Optical Tensiometer was used to measure advancing (θ_a), receding (θ_r) and equilibrium (θ_e) water contact angles under air. Computer controlled needle was used to adjust the drop volume. 5 μl drop volume was used for θ_e measurements and drop volume was increased from 3 μl to 7 μl during θ_a measurements, afterwards drop volume was decreased to 3 μl to measure θ_r . The needle was kept within the droplet during both advancing and receding contact angle measurements. Three different contact angle measurements were performed on each sample surface and average CA values were reported with standard deviations of $\pm 2^\circ$.

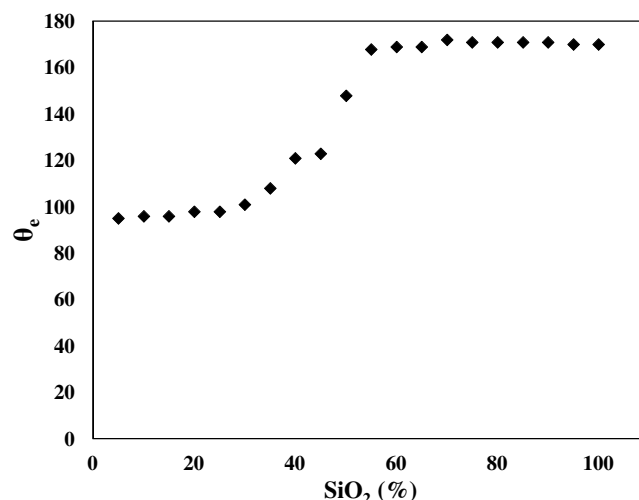


Fig. 1. θ_e values of PS composite surfaces with varying SiO_2 nanoparticle content.

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