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Research Article

Superhydrophobic Coating using Silica Nano Particles Extracted from Agricultural Waste

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Abstract

Superhydrophobic surfaces having water contact angle greater than 150° are very interesting and can be very useful for applications such as anti-fouling, de-icing, anti-corrosion, water proofing and anti-fog in many use cases such as transport, daily uses around homes. hydrophobic surfaces which are present naturally fascinated scientists and engineers for many years, researchers found out that the hydrophobicity can be replicated by having micro or nano surface roughness, the problem then for many years was that it was not cost effective to achieve superhydrophobic surfaces. Present study focuses on a cost effective method for achieving superhydrophobic surfaces using SiO_2 nano particles which are extracted from agricultural waste i-e Rice Husk, the contact angle was calculated using photos taken with a smartphone and using image analysis software, average static contact angles of water drops were found to be in the range of 151°

Keywords: *Superhydrophobic, Nano surface roughness, Rice Husk, SiO_2*

1. Introduction

Superhydrophobicity is present in nature around us the best example of natural superhydrophobic surface is the lotus leave. Superhydrophobic surfaces are characterized by having a contact angle greater than 150°. The lotus leave has water contact angle of approximately 164° so when a water drop comes in contact with the surface of lotus leave it beads up(D. Ebert *et al*, 2012). Superhydrophobic surfaces are gaining interests due to their excellent applications like self-cleaning windows, water repellant fabrics etc. Researchers have concluded that the super hydrophobicity is caused by the combination of micro or nano surface roughness and water repelling waxy materials coated on top of the rough surfaces(G. Y. Bae *et al*, 2009). In recent studies nano particles are widely being used to induce the roughness property on normal surfaces, these particles include TiO2, ZnO and SiO2, and chemicals such as fluorinated silanes and alkylated silanes are used as surface modifiers to convert the surfaces from hydrophilic to hydrophobic. However, particles like SiO2, TiO2, ZnO and chemicals are too expansive to be used in abundant quantities and are hazardous to human health also.

Rice Husk Ash on the other hand can be used as a potential source for the SiO2 because rice husk is considered as agriculture waste and is burned to produce energy in most rice mills, it is worth to mention here that the rice husk ash contains approximately 90% to 95% Silica and rest are metallic impurities and the silica can be extracted by many techniques like sol-gel method, alkali extraction and precipitation etc.(V. H. Le *et al*, 2013). The silica present in RHA is in the amorphous state which has a host of applications due to its high surface area and high reactivity so it can be used in other applications besides the superhydrophobic surfaces as well. This study however is focused on extraction of amorphous silica nano particles from rice husk using simple method of sol-gel using common inexpensive chemicals which are also environment friendly, and using the extracted silica nano particles to produce superhydrophic surfaces on different substrates like glass, metal, paper etc.

2. Materials and Experimental Work

2.1 Materials

Rice Husk, Hydrochloric Acid (sigma), Ethanol (sigma), Methanol (Sigma) Acetone (sigma) n-Hexane (sigma), Trimethylchlorosilane (TMCS) (sigma), glass slides were purchased from local market.

2.2 Extraction of silica nano particles

Rice Husk as received was washed with de-ionized water three times to remove the bulk of dust and

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debris particles, the washed rice husk was then dried for 24hrs at room temperature. After the rice husk was dried the washed rice husk was then treated with diluted HCL solution at 60 °C under continuous stirring to remove the metallic impurities which cause the silica to be crystalline at temperatures above 700°C(W. Wang *et al*, 2011). The acid treated rice husk is then again washed with deionized water to neutralize and remove any traces of acid solution and dried overnight in an oven at 60°C. Finally, the dried husk is then heated to 650-700°C in a muffle furnace to burn the organic mass. The ash now obtained is rich in silica which is evident from its pure white color(W. Wang *et al*, 2011), although the white ash is rich in silica and can be used as is, but for our application we need tighter control over particle size and purity of 99.8%, to achieve this the white ash is boiled with 3M NaOH for 5 hours to produce sodium silicate. The obtained solution is then filtered and 1M HCL is then added dropwise under constant stirring to neutralize the solution, as the pH of the solution reaches near 8.0 a hydrogel starts to form, continuous stirring prevents the gel to form quickly(U. Zulfiqar *et al*, 2015) and this results in smaller overall particle size, the stirring is then stopped and the hydrogel is then dried overnight in an oven at 60°C. A white powder is obtained after all the water is dried and the powder is then subjected to washing with warm deionized water to remove the traces of sodium chloride. The washed silica nano particles are then dried in an oven at 60°C for 3 hours.

2.3 Functionalization of silica nano particles

The silica nano particles are then slowly stirred in a 10% solution of Trimethylchlorosilane with n-hexane for 24 hours. The particles are then washed with n-hexane followed by ethanol to remove the excess TMCS from the particles surface(A. Venkateswara Rao *et al*, 2009).

2.4 Preparation of superhydrophobic surfaces

0.6g of surface functionalized silica nano particles are then suspended in 20 ml acetone in a glass beaker and sonicated for 30 minutes, simultaneously glass slides are washed with deionized water and then rinsed with acetone to remove any dust, oil or grease from the surface. The solution is then transferred into a spray bottle and sprayed on the glass slides. The slides are then left to dry at room temperature for 20 minutes. Acetone is slowly evaporated during drying and only Functionalized nano particles are left on the surface.

3. Results

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of synthesized silica nano particles is shown in Fig. 1

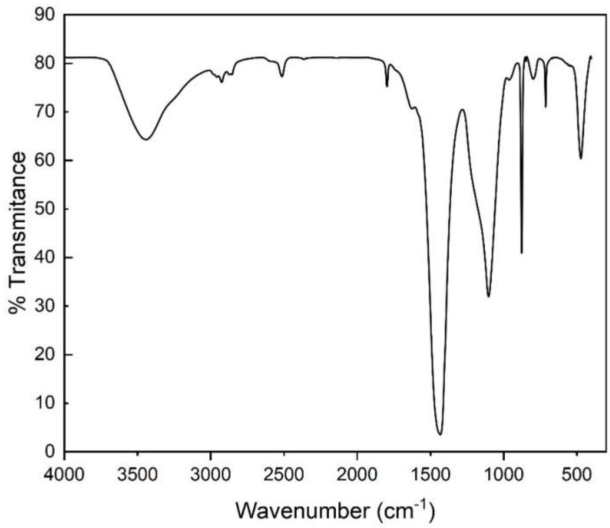

Fig. 1 FTIR spectra of synthesized silica nano particles

The above FTIR spectra elaborates that the bend at approximately 3450 cm^{-1} can be associated to -OH group stretching vibrations of either silanol or water molecules adsorbed on the surface of the particles(D. B. Mahadik *et al*, 2011), (S. Azat*et al*, 2019), the bend at approximately 1600-1625cm^{-1} is associated with the bending vibrations of the said molecules. The sharp peak at around 1100cm^{-1} is given by the asymmetric vibrations of Si-O-Si bonds while the stretching vibrations of the same Si-O-Si bonds give rise to the peak observed at around 800 cm^{-1} and at around 472 cm^{-1} bending vibrations of the same bonds are observed(A. Venkateswara Rao *et al*, 2009), (S. Azat*et al*, 2019), (Faouzi. H *et al*, 2014).

3.2 Particle Size Analysis

The particle size of the obtained silica nano particles was measured using Malvern Zeta Sizer Nano ZS, the results show that the average particle size is 300 nm.

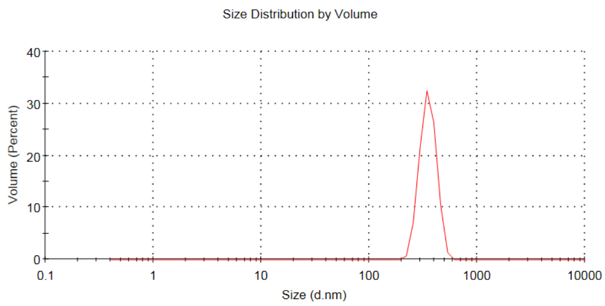

Fig. 2 Particle size graph of silica nano particles

3.3 X-Ray Diffraction

The obtained silica nano particles were analyzed using X-ray Diffraction technique, Fig. 3 shows the XRD pattern, it can be seen in the pattern the characteristic broad hump centered at around 22° is obtained which indicates that the silica obtained from rice husk is amorphous in nature(W. Wang*et al*, 2011), (U. Zulfiqar *et al*, 2015), (J. H. Lee *et al*, 2017) and no evidence was found for presence of any crystalline phases, which is in line with the previous studies.

3.4 Contact Angle

Static water contact angle was calculated by capturing a macro photograph of water droplet on the coated surface, the drop shape was then analyzed using image analysis software to calculate the contact angle of water to the coated surface. As shown in Fig.4, The average contact angle was found to be 151° which is considered the contact angle of water on a superhydrophobic surface.(D. A. Schaeffer *et al*, 2015) (H. Chen *et al*, 2012)

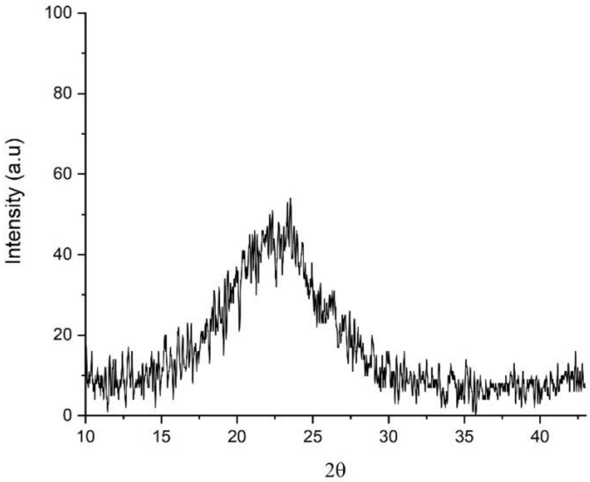

Fig. 3 XRD pattern of silica nano particles

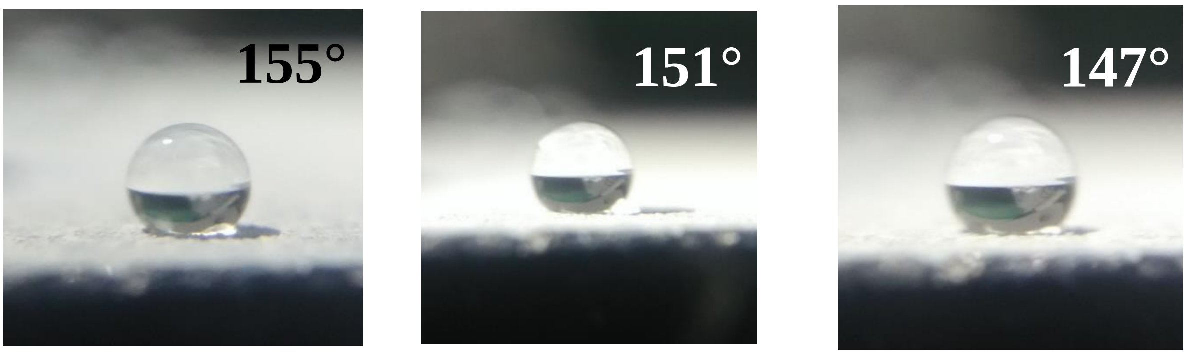

Fig. 4 Macro photographs of water drop on coated surface showing respective contact angles

Conclusion

SiO_2 based superhydrophobic surfaces were successfully prepared by using low cost spray method, the resulting contact angle of water was also found to be in the range of superhydrophobic i-e > 150°, this study also suggests that Rice Husk can be used as a viable and very low cost source of Amorphous Silica Nano particles because Rice husk is treated as agricultural waste and is mostly burned as a fuel, but the ash is rich in silica.

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