

Synthesis of Sio2 Nanoparticles via Sol-Gel Technique for the Fabrication of Anti-Corrosive and Self-Cleaning Superhydrophobic Surface Coatings to Be Utilized At Variable Thermal Conditions

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ABSTRACT

Superhydrophobic surfaces are being widelyanalyzed, engineered, and employed for a variety of engineering applications ever since last decade because of some exceptional self-cleaning characteristics and anti-wetting behavior. Herein westate the synthesis of SiO₂ nanoparticles by using sol-gel technique utilizing theliquid solution of sodiumsilicate as precursor. surface functionalization of synthesized SiO₂ nanoparticles is accomplished by treating it with TMCS at roomtemperature. Surface functionalized SiO2 nanoparticles were coated with spray gun overglass and metallic surfaces after sonication with acetonefor 30 minutes. A prominent contact angle was attained between surfaces and water droplet that averaged about 154° for metallicsurface, and 151° for glass surfaces.

Keywords: Superhydrophobic; Thin-film; Silica nanoparticles; Sol-gel; Surface Modification.

I. INTRODUCTION:

Superhydrophobicity is available in atmosphere all around us. Lotus leave is the best illustration of regular superhydrophobic surface in our nature. Superhydrophobic surfaces are

portrayed by having a contact angle more noteworthy than 150°[1]. Having an extensive water contact angle of almost 161°, when a water droplet interacts with the outer layer of lotus leave it beads up like an spherical object[2]. Superhydrophobic surfaces are acquiring interests because of their magnificent applications such as self-cleaning windows, water repellant textures and so on[3]. Specialists have inferred that this magnificent property of super hydrophobicity is brought about by the mix of miniature or surface roughness at nano level, and hydro repulsing materials like waxy substances covered on outer uneven surfaces. In late examinations nano materials or nano particles are generally utilized in instigating the roughness characteristics on ordinary surfaces, these particles incorporate TiO₂, ZnO and SiO_2 , and synthetic substances, for example, fluorinated silanes and alkylated silanes are utilized as surface functionalizing agents to change over the surfaces from hydrophilic to hydrophobic[4]. Nonetheless, particles like TiO₂, ZnO and synthetics are too costly to possibly be utilized in bulk amounts furthermore, they are dangerous to human wellbeing too.





Several research articles suggest that utilization of SiO₂have considerably increased due to the optimum outcomes achieved by this particular type of nanoparticles[5]. It is being suggested through many studies that super hydrophobicity for any surface is generally achieved by two different method, either by chemical treatment of the surfaces or by surface rendering[6]. Roughness on the surface created through rendering the surface texture up to nano level is being considered very efficient, easy, and optimum way of obtaining the property of super hydrophobicity on any substrate[7]. For this surface rendering SiO₂nanoparticles havefound a vital role, due to their exceptional properties, and cost efficiency. studies Past suggest that SiO₂nanoparticle surface coatings have exhibited excellent water contact angles of up to 157° on various substrates, including metal, glass, and fabrics. Beside such excellent behavior of superhydrophobic surfaces fabricated through various techniques, durability of such coatings has always been compromised[8][9]. Herein this research focuses on enhanced durability of SiO₂ nanoparticle surface coating fabricated through solgel method.

II. MATERIALS AND EXPERIMENTAL WORK

2.1 Materials:

Sodium Silicate Solution SSS (Sigma)Ethyl Alcohol (Sigma), Methyl Alcohol (Sigma) Acetone(sigma), Acetone (Sigma), Trimethylchlorosilane (TMCS) (Sigma), glass slides and metal pieces were purchased from local market.

2.2 Experimental Work

Sodium silicate solution was utilized as precursor for the process, at first 25 ml of sodium silicate solution was added into 300 ml of deionized water to dilute it at the room temperature. Dilution was done in order to make the reaction quick and easy, as greater surface area was provided with this dilution. Solution was kept on magnetic stirrer for 1 hour at ambient temperature, in order to completely mix it, and get homogenous solution[5]. At second stage 200 ml of ethyl alcohol was introduced to the solution at the rate of 0.8 ml per minute, the solution was kept on continuous stirring during this ethyl alcohol addition process. Slow suspension of ethyl alcohol into the solution resulted in homogenous and minimum size of the silica nanoparticles that are required for the coating purpose. Hence the silica nano particles are achieved.After the synthesis of silica nano particles, surface functionalization of the particles is carried out to obtain and optimize maximum water contact angle, for this 30 ml of TMCS trimethylchlorosilane is added into the solution, and then solution is kept for aging at room temperature for 2 hours. After aging the same solution is kept on stirring on magnetic stirrer for 2 hours. Finally, the solution possessing silica nanoparticles is centrifuged at 2500 rpm at room temperature for 40 minutes to segregate the Hence surface modified particles. silica nanoparticles are achieved.03 wt.% of surface modifiedSiO₂ nanoparticles were suspended in 100 ml acetone in a glass beaker to be kept for sonicationon ultrasonic bath equipment for20 mins, Besides this, metallic pieces and glass slides are rinsed and washed with D.I water and then further with methyl alcohol, and acetone rinsed respectively to eliminate any dust, oil or grease from the substrate. The sonicated solution containing SiO₂nanoparticles is then shiftedinto a spray bottleto be spray coated on the metallic pieces and glass slides. The metallic pieces and glass slides coated with SiO₂nanoparticles are then put in furnace at the temperature of 150 °c for achieving a good dry and durable superhydrophobic surface.

III. RESULTS

3.1 SEM Analysis

TMCS modified silica nanoparticles were analyzed for surface morphology using scanning electron microscope (SEM) as shown in Fig. 1





Fig.1 SEM micrographs of the synthesized particle at(a) 2000x, (b) 5000x, and (C) 10000x magnification

Surface topography was analyzed through scanning electron microscopy (SEM) at different magnifications; (a) 2000x, (b) 5000x, and (C) 10000x. Analysis of SEM micrographs confirmed the amorphous structure of particles having considerable tendency to agglomerate in sizable clusters, that would further enhance the hydrophobic property of particles.

3.2 Fourier Transform Infrared Spectroscopy (FTIR):

The Fourier transformed infrared spectroscopic (FTIR) spectra of functionally modified SiO_2 nanoparticles is shown in Fig. 2





Fig. 2FTIR Analysis TMCS modified silica nanoparticles.

Obtained FTIR spectrum clarifies that the peak at around 3000 cm⁻¹ can be linked to the presence of C-H group due to the functional modification of SiO₂nanoparticles by TMCS. the peak at around and between 1600-1625cm⁻¹ is linked with the bending vibrations of the said molecules [10]. The sharp bend 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 provide rise to the peak observed at around 800cm⁻¹ and at around 1260 cm⁻¹where presence of $Si-CH_3$ confirmed because of replacement of O_2 molecule due to the TMCS surface modification, that is the major factor in the enhancement of WCA of the prepared surface[11].

3.3 Particle Size Distribution:

For the particle size distribution, Zeta Sizer Nano ZS was used, that provided the results that average size of the particles is 120 nm, whereas maximum particles were obtained of the size 95 nm.



Fig. 3 Particle size distribution of synthesized silica nano particles

3.4 X-Ray Diffraction:

Surface modified silica particle were characterized through XRD, The analysis of particles on X-ray Diffraction technique was carried out, Fig.3 shows the XRD pattern, Its visible in the graph that there is a huge humpstretching from 18° to 29°. This broad hump centered at around 24° further confirms that the silica nanoparticles achieved through this technique are completely amorphous[12], and no sign of crystalline phases were observed, which is in line with the previous studies.





Fig. 4 XRD pattern of silica nano particles

3.5 Contact Angle:

Static water contact angle was calculated by capturing a macro photograph of water droplet on the coated surfaces of metal and glass, 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.5, The average contact angle was found to be 154° and 151° for metal and glass substrate respectively, which is considered the contact angle of water on a superhydrophobic surface[13].



Fig. 5Water contact angle (WCA) on (a) metal and (b) glass substrate

IV. **CONCLUSION:**

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based superhydrophobic surfaces were successfully fabricated by using cost efficientsol-gel method, the resulting contact angle of water was also found to be in the range of superhydrophobic $i-e > 150^{\circ}$ on both targeted surfaces i.e. metal and glass, the obtained superhydrophobic surface coatings were well suited at different thermal conditions for antiwetting and anti-corrosive applications, this study furthersuggests that SiO₂can be used as a feasible and very low cost source of amorphous silica nanoparticles because it is easily available in the local market due to its wide applications across field of material sciences

REFERENCES:

- Q. Liu, Y. Wu, and Z. Li, "Facile [1]. preparation of super-hydrophobic fabrics composed of fibres with microporous or microspherical coatings using the static breath figure method," Prog. Org. Coatings, vol. 149, no. September, p. 105938, 2020, doi: 10.1016/j.porgcoat.2020.105938.
- [2]. Τ. Darmanin and F. Guittard, "Superhydrophobic and superoleophobic properties in nature," Mater. Today, vol. 18, 2015. 5. pp. 273-285, doi: no. 10.1016/j.mattod.2015.01.001.
- J. Gao, X. Huang, H. Xue, L. Tang, and R. [3]. K. Y. Li, "Facile preparation of hybrid

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microspheres for super-hydrophobic coating and oil-water separation," Chem. Eng. J., vol. 326, pp. 443–453, 2017, doi: 10.1016/j.cej.2017.05.175.

- [4]. O. H. Trinh, D. B. Nguyen, M. M. Hossain, S. Mok. "Deposition and Υ. of superhydrophobic coatings on glass substrates from hexamethyldisiloxane using a kHz-powered plasma jet," Surf. Coatings Technol., vol. 361, no. January, pp. 377-385. 2019, doi: 10.1016/j.surfcoat.2019.01.068.
- [5]. D. Lin, X. Zeng, H. Li, X. Lai, and T. Wu, "One-pot fabrication of superhydrophobic and flame-retardant coatings on cotton fabrics via sol-gel reaction," J. Colloid Interface Sci., vol. 533, pp. 198–206, 2019, doi: 10.1016/j.jcis.2018.08.060.
- [6]. L. Lv, H. Liu, W. Zhang, J. Chen, and Z. Liu, "Facile UV-curable fabrication of robust, anti-icing superhydrophobic coatings based on polyurethane," Mater. Lett., vol. 258, p. 126653, 2020, doi: 10.1016/j.matlet.2019.126653.
- [7]. Y. Liu et al., "One-step modification of fabrics with bioinspired polydopamine@octadecylamine nanocapsules for robust and healable selfcleaning performance," Small, vol. 11, no. 4, pp. 426–431, 2015, doi: 10.1002/smll.201402383.
- [8]. Z. Huang et al., "One-step preparation of durable super-hydrophobic MSR/SiO 2 coatings by suspension air spraying," Micromachines, vol. 9, no. 12, pp. 1–11, 2018, doi: 10.3390/mi9120677.
- [9]. A. Ábrahám et al., "Durability of microporous hybrid silica coatings: Optical and wetting properties," Thin Solid Films, vol. 699, no. March, p. 137914, 2020, doi: 10.1016/j.tsf.2020.137914.
- [10]. F. Chi, D. Liu, H. Wu, and J. Lei, "Mechanically robust and self-cleaning antireflection coatings from nanoscale binding of hydrophobic silica nanoparticles," Sol. Energy Mater. Sol. Cells, vol. 200, no. April, p. 109939, 2019, doi: 10.1016/j.solmat.2019.109939.
- [11]. S. He, Z. Li, X. Shi, H. Yang, L. Gong, and X. Cheng, "Rapid synthesis of sodium silicate based hydrophobic silica aerogel granules with large surface area," Adv. Powder Technol., vol. 26, no. 2, pp. 537– 541, 2015, doi: 10.1016/j.apt.2015.01.002.

- [12]. B. Mahltig and H. Böttcher, "Modified silica sol coatings for water-repellent textiles," J. Sol-Gel Sci. Technol., vol. 27, no. 1, pp. 43– 52, 2003, doi: 10.1023/A:1022627926243.
- [13]. D. Y. Nadargi, J. L. Gurav, N. El Hawi, A. V. Rao, and M. Koebel, "Synthesis and characterization of transparent hydrophobic silica thin films by single step sol-gel process and dip coating," J. Alloys Compd., vol. 496, no. 1–2, pp. 436–441, 2010, doi: 10.1016/j.jallcom.2010.01.157.

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