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Superhydrophobic and High Transmittance Coated Silica Aerogel Prepared in Ambient Pressure as Self-Cleaning Windows

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ABSTRACT: ***Background:*** *Highly porous nanomaterials have been promising materials in recent years. Silica-based materials have attracted the attention of researchers because they are easy to prepare under normal atmospheric pressure. Furthermore, they have broad practical applications that aim to reduce time and cost.* ***Objective:*** *The current work highlights self-cleaning windows for high buildings that are difficult to access. Hydrophobic silica was prepared, and the surfaces of regular glass were coated with gel during the preparation process, taking into justification that the transparency of the glass would not be affected.* ***Methods:*** *The preparation was carried out under normal atmospheric pressure and for a period of only one week, during which samples of hydrophobic silica were prepared with ratios of the starting material tetraethylorthosilicate (TEOS), ethanol, and hydrochloric acid with 0.5 molar concentration, and taking into account the molar ratios in each case for each of TEOS:ethanol:HCl at ratios of 1:5:0.2.* ***Results:*** *The results of the UV test showed that the transparency ratios of samples changed slightly with a variation when the TEOS ratios were changed. The best result was 95% at pH7 with a volume ratio of 1 TEOS. The contact angle was within the superhydrophobic region at 152° and was best at a value of 1TEOS at pH7. This was confirmed by the FTIR test through the weak beams that return to OH, Si-OH, and H-OH groups. Also, the AFM images showed that the roughness ratios are very good, which enhances the role of the prepared models in practical applications. The differential thermal-thermogravimetric TGA-data test also confirmed the stability of the samples at a temperature of 217 °C. This is particularly important, as glass is exposed to varying temperatures throughout the year.* ***Conclusions:*** *It can be confirmed that it is possible to obtain superhydrophobic silica coated on glass slides, which exhibit high transparency and thermal stability. This enables it to withstand external conditions such as high temperatures, rain, and dust. Therefore, it is easy to produce self-cleaning glass covers or windows that are resistant to heat and humidity.*

KEYWORDS: *Silica aerogel; Self-cleaning; Hydrophobicity; FTIR; Transmittance*

INTRODUCTION

Aerogels are sophisticated materials resembling solid smoke, composed of almost 90-95% air. The last comprises a tenuous matrix of silicon dioxide [1], [2]. Aerogel is one of the lightest weight solids ever considered. An aerogel is made by the common way that's sol-gel technique. Where this process, the organic compounds containing silica experience the chemical reaction producing silicon oxide [3], [4]. The sol-gel technique is employed to produce aerogel, which involves the chemical transformation of a liquid solution into a gel phase. Silica is typically derived from precursors like tetramethoxysilane (TMOS) and tetraethoxysilicate (TEOS) [5], [6]. Silica aerogels, which are produced as mesoporous cells, are semi-transparent and heat-insulating materials. It has various distinguishing characteristics, including a huge specific surface area and a high porosity [7]. The experiences of the aerogels depend on influences participating in the preparation procedure; the most significant limits adopted in their preparation are pH factor, R-molar ratio, catalysis, and the precursors [8], [9]. Self-cleaning surfaces for glass have become a central topic in materials research because of their capacity to diminish pollutant deposition, increase transparency, and boost durability [10], [11]. Investigations on self-cleaning surfaces derive from the natural occurrences termed the "Lotus effect" and the "Photocatalytic effect." The lotus effect is defined by extreme hydrophobicity, allowing water droplets to achieve a significant contact angle on the surface and roll off, so removing dust and other impurities. Conversely, the photocatalytic effect promotes the breakdown of contaminants on the surface by the catalytic activity of UV light, resulting in their spontaneous disintegration [12], [13]. The hydrophobicity of materials provides a reliable assessment of surface modifications, contingent upon the chemical composition and surface tension [14]. Many researchers give more interest to producing waterproof silica gel and applying it in many fields, such as Omer Kesmez, Fabrication of hybrid nanocomposite coatings by the sol-gel technique for hydrophobic and self-cleaning characteristics. They obtained very transparent photocatalytic hybrid nanocomposite coatings that display and possess oleophobic characteristics [15]. While Hamdi and others developed extremely clear, hydrophobic, durable, and self-cleaning coatings as a substitute for fluorine-based coatings applied to glass. The organic modification of the gel with trimethylsilyl ($Si–CH_3$) groups results in a homogenous and hydrophobic surface. Appropriate dilution of this gel with ethanol produces a very clear covering while maintaining its hydrophobic characteristics [3]. Whereas Keting Li *et al.* prepared superhydrophobic cotton as self-healing made from silica aerogel for oil-water departure, they exhibited that the treated cotton fabrics exhibited the superhydrophobic property with contact angle 161° [16]. In this work, it prepared the superhydrophobic coated silica film on the glass surface for use as self-cleaning windows with high transparency. The important parameters that control the contact angle and transparency are the pH value and volume ratio of TEOS.

MATERIALS AND METHODS

Materials

Tetraethylorthosilicate (TEOS, 99% purity, Sigma–Aldrich, USA), Ethanol (99% purity, Spain), Hydrochloric acid (HCl, 98% purity, India) 0.1 M molar concentration, and Ammonium hydroxide solution with 0.5 M molar concentration (NH_4OH, 25–28%, India). For modification, it needs Trimethylchlorosilane (TMCS, 99% purity, TCI, Japan) and n-Hexane (98% purity, India), also, to dilute both aluminum and hydrochloric acid, distilled water was used.

Method of Preparation and Characterizations

silica aerogel prepared via 2-steps acid–base catalysis, the molar Ratio of the precursors (TEOS:ETHO:HCL) was(1:5:0.23) in the magnetic stirrer for 15 minutes, and then NH_4OH (0.5) was added drop by drop until the required acidic function is obtained, three values of pH have been selected, namely (5,7,9) to investigate which of the models can yield the highest transparency and the highest contact angle value that suits the current work objective.The volum ratio of TEOS was changed from (1,2,3) ml, the sum of all samples, was 9. for every pH value. After very good cleaning of the glass slides (1×5 cm), they were coated with condensed silica by covering one face and then converted to gel (the gel time depended on pH). the alcogel was soaked for 24 hr. with pure ethanol and then washed with ethanol three times every 1hr, the residue materials were removed by washing with pure n-hexane two times every 30 min; to obtain the modified gel, it prepared the modified solution (n-hexane + TMCS) with a ratio of 6:1 volume. The gel is to be put in the oven starting at 60 °C, steeping at 20 °C for 20 min, until 120 °C, and then let to cool to room temperature. Finally, it was washed with pure Hexane 3 times, and then the modified gel was left to dry at Room temperature. The absorption spectrum was examined using a UV spectrophotometer to analyze the transmittance properties which calculated by relates to absorbance (A) through the logarithmic formula as illustrated by (1) [17]:

$$A = -\log(T) \quad \text{OR} \quad A = -\log(I/I_0), \tag{1}$$

where incident light (I_0): The intensity of light that hits the sample or substance, transmitted light (I): The intensity of light that successfully passed through the sample, percent transmittance (%T): The transmittance value multiplied by 100, making it easier to interpret as a percentage. To confirm the molecular bands and verify the modification process, the FTIR spectrum was used in this step. The surface and pore characterization were examined via nitrogen adsorption-desorption analysis, and the topography of the surface was studied by atomic force microscopy. The modified surface and the degree of hydrophobicity are confirmed by contact angle measurement. The TGA curves were acquired using the DTG-60H apparatus concurrently with the DTA-TG Shimadzu and evaluated with thermogravimetric software, focusing on mass loss as a function of temperature.

RESULTS AND DISCUSSION

UV Spectra Analysis

The transmission (T%) of the samples was calculated using the absorption spectrum depended on (1), as illustrated in Figures 1, 2 and 3.

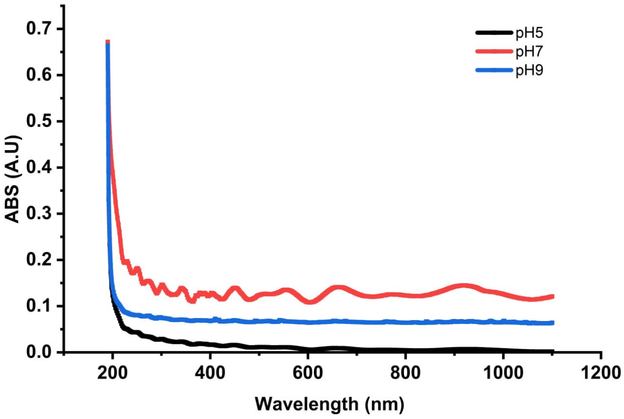

Figure 1. UV spectra for coated silica aerogel (1TEOS, 1TMCS) at different pH

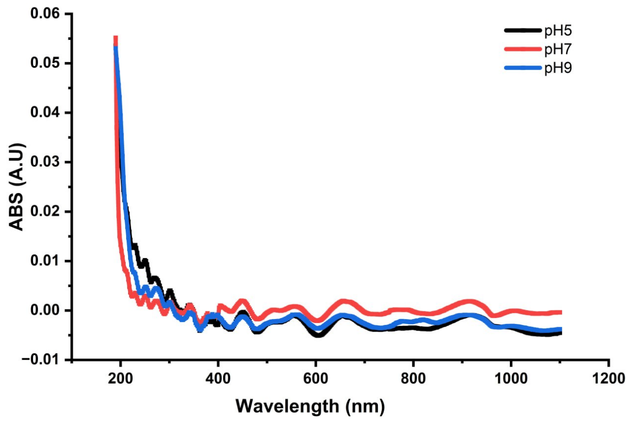

Figure 2. UV spectra for coated silica aerogel (2TEOS, 1TMCS) at different pH

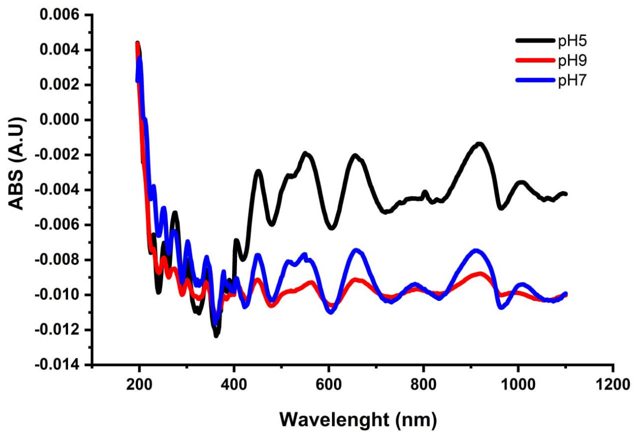

Figure 3. UV spectra for coated silica aerogel (3TEOS, 1TMCS) at different pH

Silica gel does not have a specific absorption spectrum. Where as silica gel is characterized by its composition being mostly air surrounded by a network of silicon dioxide. This explains the product's high transparency. However, preparation conditions can alter this transparency to some extent, reducing it from 90% to 70%. The most influential factors are pH, precursor concentration, the addition of the modification solution, and the preparation steps (one-step or two-step acid-base catalysis) [8]. Its optical properties are transparent (or translucent), and it does not absorb visible light in a way that would define its spectrum. This explains the question: why doesn't it exhibit a characteristic absorption spectrum in the visible light range? In other words, it allows visible light to pass through it rather than absorbing it [18]. This is clearly shown in the absorption spectrum illustrated in the figures. However, this does not negate the primary objective of examining the spectra of the samples. When varying the proportions of the silica precursor (TEOS), a change in the spectrum was observed,

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allowing us to calculate the transmittance at different TEOS ratios and pH values, which have the greatest effect. Table 1 shows the magnitude of the change in transmittance for the samples with varying precursor ratios and pH. At lower TEOS ratios (1 mL), the samples exhibited the highest transmittance compared to those in Figures 1 and 2. Increasing the precursor ratios can lead to uneven polymerization and silicon cluster formation due to the proximity of the Si-O-Si molecules, which appear as irregularities in the absorption spectrum.

Table 1. Percentage transmittance of silica-coated glass samples at different pH

	~T% (pH5)	~T% (pH7)	~T% (pH9)
T1M1	93	95	90
T2M1	90	92	89
T3M1	87	89	81

FTIR Spectrum Analysis

Figures 4, 5 and 6 show the FTIR spectra for silica Aerogel slides with different pH and different volume ratio of TEOS with (1, 2, 3) ml and constant volume ratio of TMCS.

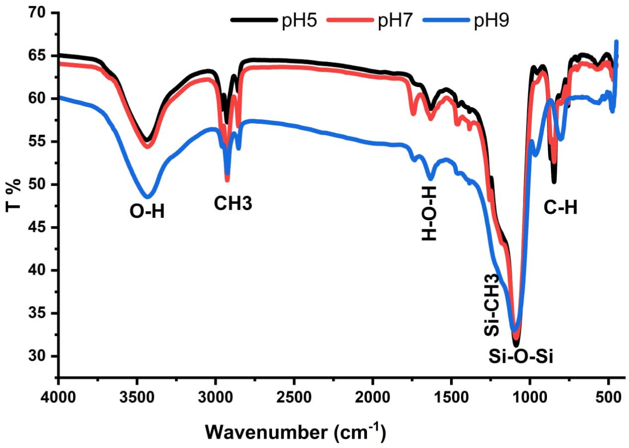


Figure 4. FTIR spectra for coated silica aerogel (1TEOS, 1TMCS) at different pH

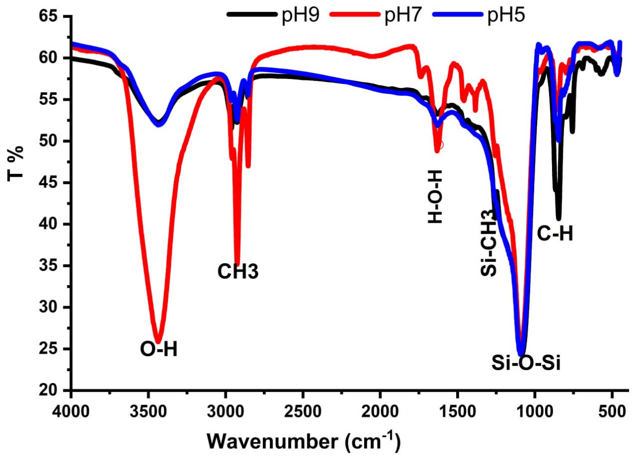


Figure 5. FTIR spectra for coated silica aerogel (2TEOS, 1TMCS) at different pH

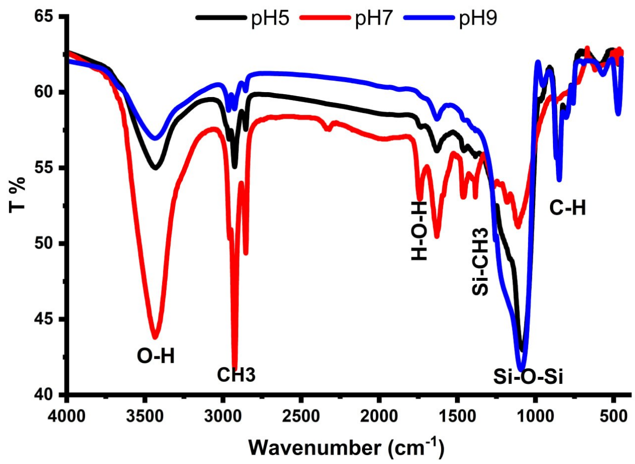
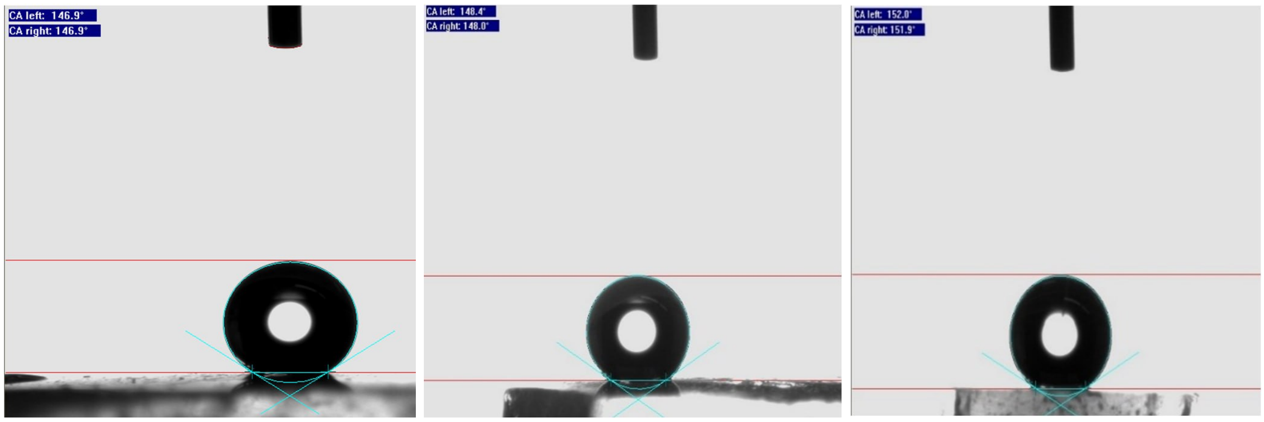


Figure 6. FTIR spectra for coated silica aerogel (3TEOS, 1TMCS) at different pH

The weak band at 3436.61 cm^{-1} for (1TEOS, 1TMCS) and at 3437.39 cm^{-1} for (2TEOS, 1TMCS) and at 3435.97 cm^{-1} for (3TEOS, 1TMCS) at attributed to OH-hydroxyl groups bands [19]. The water absorption at 1631.37 cm^{-1} for (1TEOS, 1TMCS) and at 1632.10 cm^{-1} for (2TEOS, 1TMCS) and (3TEOS, 1TMCS) at 1630.56 cm^{-1} these weak Band Because of the modification proses with TMCS which make the Replacing H-OH Bands with H-CH_3 at region 846.01 cm^{-1} [20], [21]. In the ratio of (1TEOS, 1TMCS) and at 866.98 cm^{-1} (2TEOS, 1TMCS) and at 846.12 cm^{-1} for (3TEOS, 1TMCS) and HCl at 1631.49 cm^{-1} for (1TEOS, 1TMCS) and at 1632.37 cm^{-1} for (2TEOS, 1TMCS) and at 1630.56 cm^{-1} for (3TEOS, 1TMCS) the strong band at 1087.77 cm^{-1} for (1TEOS, 1TMCS) and at 1088.93 cm^{-1} for (2TEOS, 1TMCS) and at 1082.52 cm^{-1} (3TEOS, 1TMCS) [22], [23]. due to stretching vibration of Si–O–Si. The band at 3000 represented to the stretching vibration is supported by tow peaks around 3000 and 820 cm^{-1} the Intensity of band for FTIR spectra are near to each other until when pH changed only in case of transmittance it noticed many changing in the slides as minted in UV–absorption figures.

Contact Angle Measurement

The degree of hydrophobicity and the success of modification are confirmed by the contact angle degree, which is represented in Figure 7, showing the contact angle for 1TEOS and 1TMCS with different pH. Generally, all samples have a high level of hydrophobicity, since the lowest contact angle is 146°, while the highest is 152°. This means that the ability of water to be thrown away in all samples is very good, leading to ease of use in the self-cleaning windows.



(a) (b) (c)

Figure 7. Contact angle measurement of silica-coated glass (1TEOS, 1TMCS) with different pH: (a) pH9, (b) pH5, (c) pH7

Nitrogen Adsorption-Desorption Examination

The ratios for surface area, pore volume, and rate of change in pore dimensions are shown in Table 2, in addition to the hysteresis rings of the samples, as illustrated in Figures 8 and 9.

Table 2. The structural properties of silica-coated glass samples at different pH

	Surface aera (m^2 g^{-1})	**Total pore volume (cm^3 g^{-1})**	**Mean pore diameter (nm)**	**rp, peak (Area) (nm)**	**Vm(cm^3 g^{-1})**
T2P5M1	876.7	2.2391	10.216	1.22	201.42
T2P7M1	1282.3	1.3054	4.0721	1.22	141.8

The structural properties, such as pore volume, average pore radius, and specific surface area of the synthesized silica aerogel, were determined. At a pH of 7, with the same volume ratio of TEOS and TMCS, the surface area is 1282.3 m^2 g^{-1}, the volume of pores is 1.3054 cm^3 g^{-1}, and the mean pore diameter is 4.0721 nm. In the case of pH5, the surface area was found to be 876.7 m^2 g^{-1}, the volume of pores was 2.23 cm^3 g^{-1}, and the mean pore diameter was 10.216 nm. It was observed that the surface area is very high at pH7. On the other hand, the pH parameter can be influenced by the

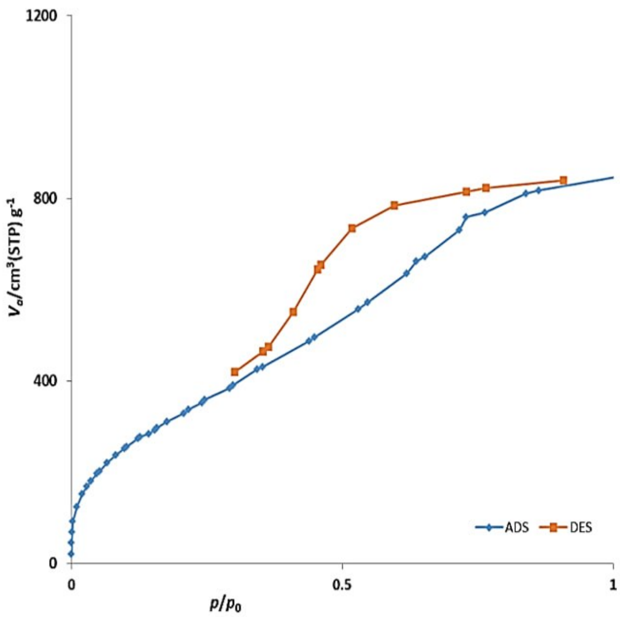


Figure 8. Adsorption desorption isotherm of silica-coated glass with 2TEOS, 1TMCS and pH7

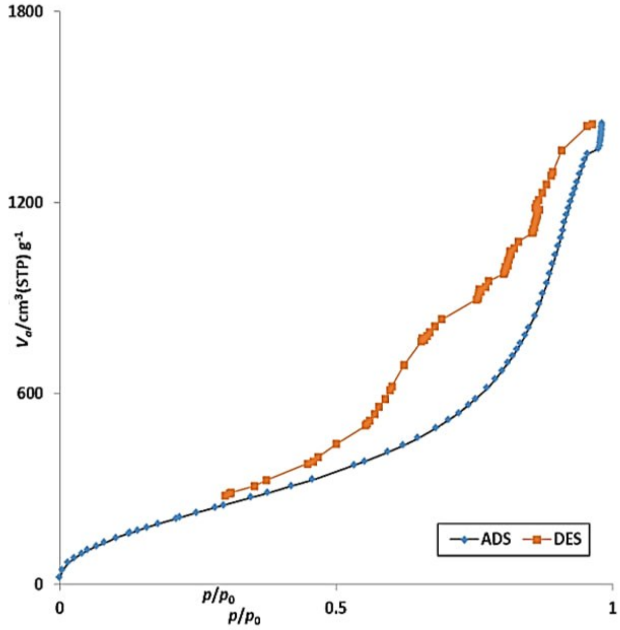


Figure 9. Adsorption desorption isotherm of silica-coated glass with 2TEOS, 1TMCS and pH5

properties of the samples, which can increase the surface area when the samples are converted from an acidic to a neutral state. This property enables the glass to function as self-cleaning windows for an extended period. The result indicates that it could be utilized in many applications, such as self-cleaning windows. However, the increase in surface area leads to higher porosity; furthermore, surface modification plays an important role in improving the characterization properties.

The hysteresis loop of physisorption The isotherms for silica aerogel samples exhibit indications of hysteresis (Type IV and III) for pH7 and pH5, respectively [24]. These findings align with the standard defining properties of mesoporous materials [25], [26]. A large hysteresis loop indicates that the silica structure has more holes. The hysteresis loop resulting from capillary condensation in mesoporous materials inside the adsorbent occurs at elevated relative pressures, illustrating the adsorption-desorption behavior of N on the adsorbent. The pore size distributions of surface-modified silica aerogel were derived from desorption isotherms using the Barrett-Joyner-Halenda (BJH) technique. As illustrated in Figures 10 and 11, they include addressing the difference between the pore radius relationship and the change in volume, with the conclusion that the relationship is inverse to the acid function pH5 and pH7.

Differential Thermal-Thermogravimetric (TGA) Analysis

The TG-DTA analysis of certain of silica-coated glass shows a quick spike in the rate of weight loss for hydrophilic silica aerogel between 60–110 °C, this phenomenon is attributable to the release of bound alcohols and water vapor. Subsequent from the condensation reactions of Si-OH and Si (OC_2H_5) groups. Conversely, the amount of weight loss (drying) percentage remains insignificant for hydrophobic aerogels up to their thermal stability temperature, as demonstrated in the samples prepared in the current work. The thermal stability of the material (1TEOS, 1TMCS) at pH7 was examined using TGA, revealing that the material exhibited significant stability at elevated temperatures, with a mere 3.493% weight loss, as illustrated in Figure 11. This weight loss is attributed to the calcination of the coating, rendering the material hydrophobic post-calcination. Experiments were performed using (GO-SA refers to Graphene Oxide-Silica Aerogel hybrid nanofluid used in technical research) after its heating in a quiet furnace at 217 °C for 1 hour in the presence of air to simulate the calcination seen in TGA.

While for (2TEOS, 2TMCS) for pH5 The results indicated that the material exhibited exceptional stability at elevated temperatures, with a mere 8.46% weight reduction, as seen in Figure 12. A lower change in weight indicates that the material is less influenced by heat. This signifies that pH7 is superior than pH5. The findings of these investigations demonstrate that the hydrophobicity of silica aerogel for (1TEOS, 1TMCS) at pH7 and (2TEOS, 2TMCS) at pH5 decreased with an increase in heating temperature to 180 °C, as seen in Figures 12 and 13. Silica becomes completely hydrophilic when the heating temperature is raised to 240 °C [27], [28].

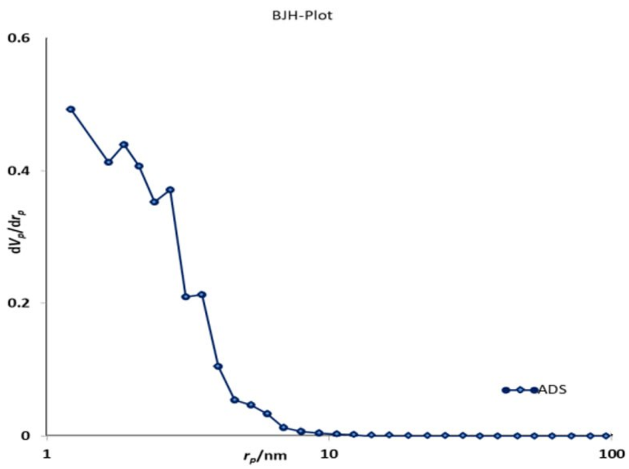


Figure 10. The relationship between the pore radius and the amount of change in volume at pH7

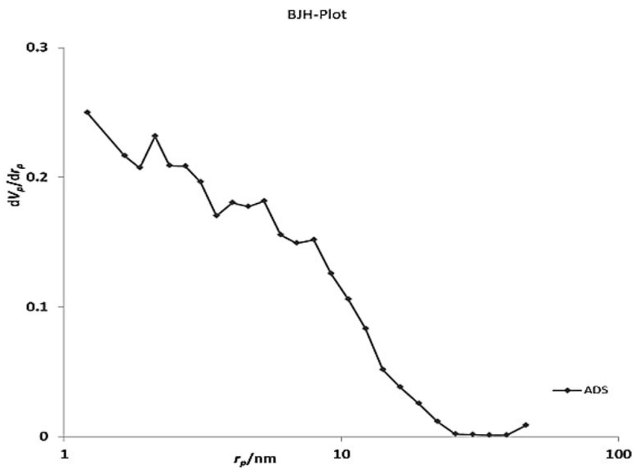


Figure 11. The relationship between the pore radius and amount of change in volume at pH5

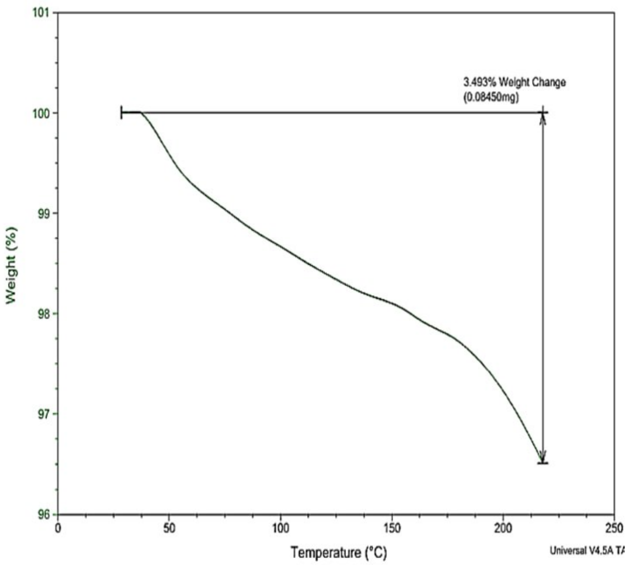


Figure 12. TGA for silica Aerogel outed for (1TEOS, 1TMCS) for pH7

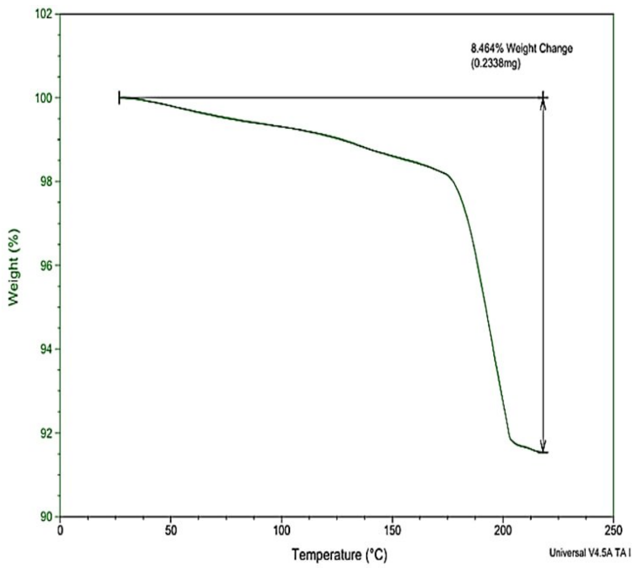


Figure 13. TGA for silica Aerogel outed for (2TEOS, 2TMCS) for pH5

Atomic Force Microscopy

The Atomic Force Microscope (AFM) is a technique employed to investigate the morphological properties of material surfaces at high magnification, providing critical information about surface roughness statistical values. Figure 14 shows the 3-D AFM images of the silica aerogel surface. The sample exhibited a surface characterized by hills and valleys with different characteristics of surface morphology and roughness. The surface roughness is quantified by the quantity of protrusions. An increased number of protrusions correlates with heightened surface roughness, resulting in enhanced material characteristics, an extensive surface area, and the presence of nanoparticle-sized bumps [29].

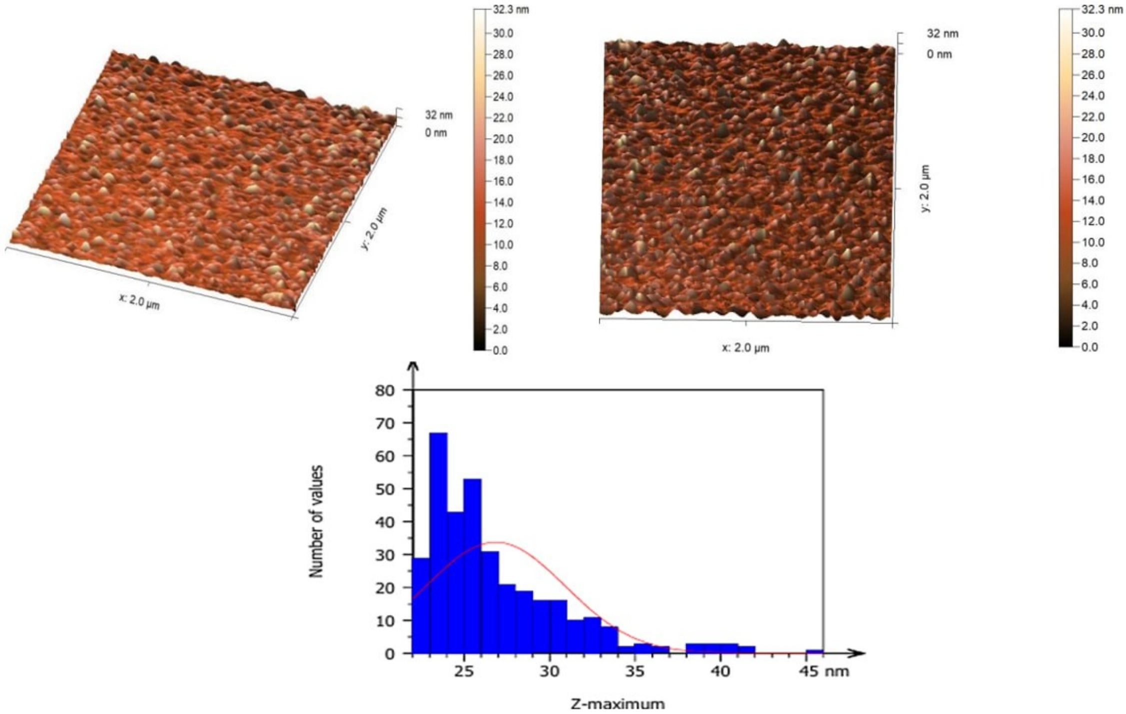


Figure 14. AFM for silica Aerogel outed for(1TEOS, 1TMCS) for pH7

The average value of the coating surface is 11.2948 nm, and RMS roughness (Sq) is 4.04857 nm. For (1TEOS, 1TMCS) at pH 7, the AFM analysis demonstrates that the glass that was treated by silica aerogel with a thin coating had high roughness, ensuring the super hydrophobic surface was constructed. The RMS roughness is 2.94687 nm. For (2TEOS, 2TMCS) as shown in Figure 15 for pH5, the AFM analysis demonstrates the glass that was treated by silica aerogel with a thin coating. It has the RMS roughness of (1TEOS, 1TMCS) for pH7.

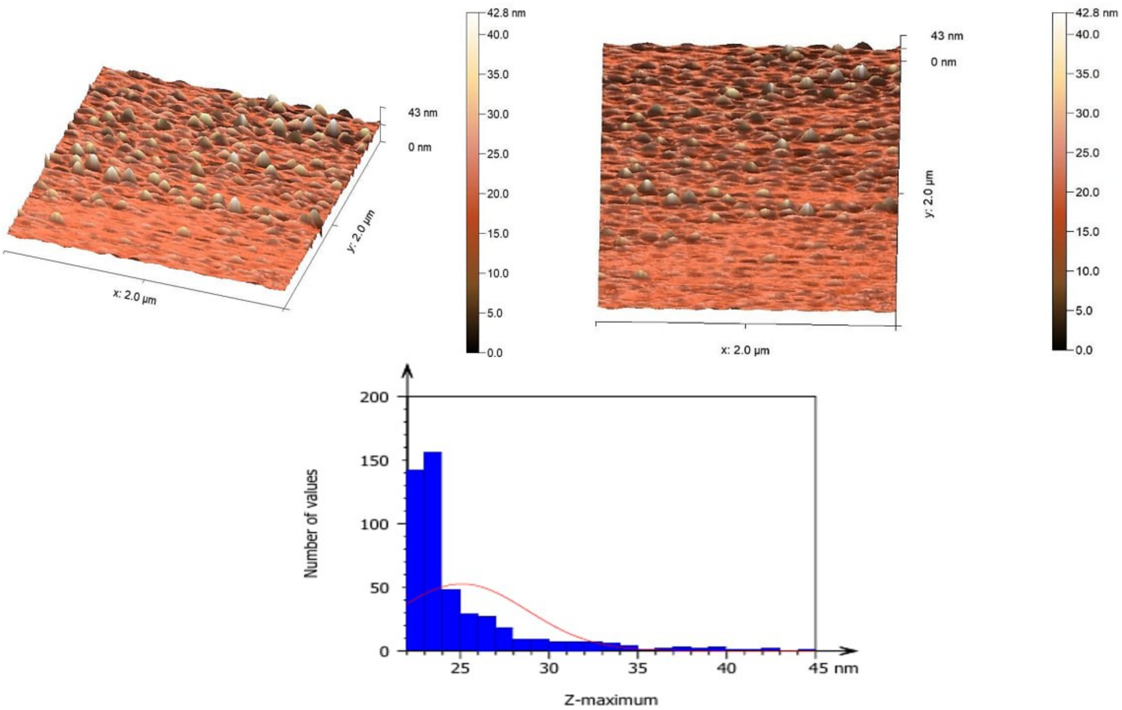


Figure 15. AFM for silica Aerogel outed for(2TEOS, 2TMCS) for pH5

CONCLUSION

The making of superhydrophobic materials with high transparency is the main goal of many researchers. In the present work, the ability to obtain superhydrophobic silica coated on the glass slides 152°, which has high transparency with 95% and thermal stability, can be confirmed. Therefore, it is easy to produce camera lenses, or even car windows and self-cleaning glass covers or windows that are resistant to heat and humidity. This enables it to withstand external conditions such as high temperatures, rain, and dust. The result was confirmed by the FTIR test through the weak beams that return to hat return to OH, Si-OH and H-OH groups, which confirms the success of the improvement achieved on the surface. Through the results, it was shown that the roughness ratios are very satisfactory, which enhances the role of the prepared models in practical applications. The TGA data test also verified the samples' stability at a temperature of 217 °C this is useful very much so, as the glass is exposed to varying temperatures throughout the year, Therefore, it is easy to produce camera lenses, or even car windows and self-cleaning glass covers or windows that are resistant to heat and humidity by preparing membranes made of silica under normal pressure.

SUPPLEMENTARY MATERIAL

None.

AUTHOR CONTRIBUTIONS

Huda Salh Mahdi: Conceptualization, Methodology, Investigation, and Writing - original draft. Israa F. Al-sharuee: Formal analysis, Visualization, and Writing - review & editing, Investigation, and Supervision.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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CONFLICTS OF INTEREST

The author declares no conflicts of interest.

DECLARATION OF GENERATIVE AI USE

The authors declare that no generative AI or AI-assisted technologies were used in the preparation of this manuscript.

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