



# Superhydrophobic Surface from Sol Gel Synthesis of Thin Sol Gel Fluorinated Silica Coating

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**Abstract:** A thin fluorinated silica coating was synthesized via a sol-gel route using silica nanoparticles as surface roughness agents functionalized with long-chain fluoroalkylsilane. The coatings were characterized for surface wettability, optical transmittance, infrared spectra, and morphology. All samples exhibited excellent optical transparency as verified by UV-visible spectroscopy. The fluorinated silica coating demonstrated markedly enhanced water repellency compared to TEOS and TEOS-FAS systems, confirming the critical role of fluorinated silica in improving hydrophobicity. These findings indicate its strong potential for application as a transparent hydrophobic coating in optoelectronic devices, lenses, and window materials.

**Keywords:** Silica; Sol gel; Superhydrophobic

## Introduction

The wettability of a surface is one of important and attractive characteristics to many functional materials in the field of materials technology and development. These interesting characteristics mainly rely on both of their chemical composition as well as geometrical structure (Cassie & Baxter, 1994; Wenzel, 1936). A surface with water contact angle (CA) greater than 150° is commonly defined as a superhydrophobic surface (Nosonovsky & Bhushan, 2008). Superhydrophobicity was first observed on lotus leaves where a high water contact angle and low hysteresis cause water fell on the leaves to bead and roll off from the surface, thereby leading to water repellency and self-cleaning characteristics (Neinhuis, 1997). As of May 2023, the number of articles on superhydrophobic surfaces published >27,000 documents including papers, patents, and books in the Web of Science demonstrating a high level of interest in this area for all manner of uses, from

self-cleaning, anti-fog, or anticorrosion surfaces to basic research (Li et al., 2023).

Generally, in order to create a biomimetic superhydrophobic surface, two factors should be considered. First, the surface should have low surface energy. Second, the surface should be roughened. It is thought that surface roughness has a complicated effect on wetting (Ji et al., 2013). It increases the surface area so that the same solid-liquid contact area can be achieved for a liquid drop sitting on a rough hydrophobic surface with a higher CA than on a smooth hydrophobic surface. More importantly, roughness combined with hydrophobicity often results in air pockets being trapped between the solid and liquid, thus, leading to a significant decrease in the solid-liquid adhesion and an increase of the CA. In addition, roughness can pin the solid-liquid-air line at the edge of an asperity and thus increases the liquid adhesion to solid. Therefore, artificial superhydrophobic surfaces can be achieved by two different strategies, either by

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creating a rough topography on a hydrophobic surface or by modifying a rough surface with materials of a low surface free energy (Feng et al., 2002).

In this project, fluoroalkylsilane (FAS) was used as a co-precursor to induce hydrophobicity of the coating surface. Liu et al. (2015) mentioned that FAS are well-known as low surface energy materials. This is due to its long chain of fluorine in its chemical structure. The tail-end group of the FAS molecule is the hydrophobic fluoroalkyl group and the monolayer of FAS self-assemble on the solid surface, with the molecular axis perpendicular to the surface. Generally, FAS layers are applied to lower the surface-energy of the high surface-energy rough solid surfaces in order to provide hydrophobicity to the coating surface. Silica is used as roughness agent, to increase the hydrophobicity of the surface coating. Commonly, carbon-based material such as graphene is used due to their high surface area and non-polar carbon structure. However, graphene is quite expensive which is not preferable in industry (Nishina, 2024).

Although graphene oxide can be synthesized to replace graphene, the dark colour of graphene oxide can affect the see-through coating (Jiříčková et al., 2022). This is because transparent hydrophobic coating promises wide range of application especially in industry compared to translucent or opaque hydrophobic coating. In this case, the silica was used as roughness agent to increase the hydrophobicity of the coating. Despite the fact that silica is not as effective as graphene oxide, it still can result in transparent hydrophobic coating (Yilbas et al., 2018). The process involved is sol-gel polymerization process of tetraethylorthosilicate (TEOS) under hydrolytic condition by using acid. According to Guglielmi (2020), sol gel method enables the synthesis of amorphous or crystalline oxide and hybrid organic-inorganic materials starting from solutions of suitable precursor, generally alkoxides, through their reaction with water. The two principal chemical reactions involved are the hydrolysis of the precursor with the formation of partially hydrolysed products and their condensation with formation of an oxide network.

## Method

### Materials

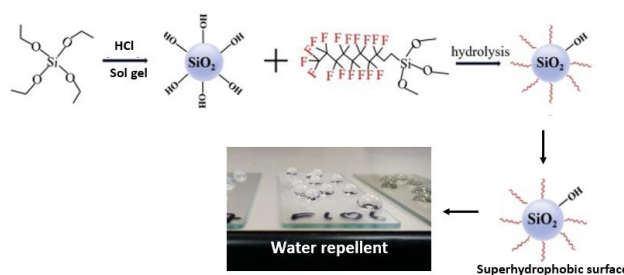
The chemicals used in this study included tetraethoxysilane (TEOS, Merck), fluoroalkylsilane (FAS), specifically heptadeca fluoro decyl trimethoxysilane (Fluorochem), silica (Sigma), ethanol (J. Kollins), hydrochloric acid (Merck), and distilled water. All chemicals were used as received without further purification.

### Pretreatment of glass microslides

Pre-treatment of the glass substrate was an essential initial step to ensure the removal of dirt and to obtain a clean glass surface (Theodorakopoulos et al., 2025). Commercially available glass microscope slides (Sail) were immersed in 0.5 M HCl for 10 minutes, thoroughly washed with distilled water, and finally rinsed with ethanol. The slides were then dried in an oven at 60 °C for several hours prior to use. This precautionary procedure helped to achieve a homogeneous and uniform coating layer on the glass surface.

### Sol gel synthesis of superhydrophobic fluorinated silica

A silane sol containing fluorinated functional groups was synthesized by mixing tetraethoxysilane (TEOS) as the primary precursor with heptadecafluorodecyltrimethoxysilane (FAS) serving as the co-precursor and surface modifier. Distilled water and ethanol were employed as solvents, while hydrochloric acid (HCl) acted as the catalyst. The mixture was continuously stirred to facilitate the formation of a stable sol-gel network. To improve the surface roughness of the resulting coating, silica nanoparticles (Sigma) was incorporated into the sol at a concentration of 0.03 wt% relative to the total mass of the coating master batch (Figure 1). The hydrophobic surface was fabricated via spray deposition using a spray coater (JSpray 125-A) equipped with a 0.3 mm fluid nozzle. Prior to deposition, the glass substrates were preheated to 60 °C. During the coating process, the distance between the spray nozzle and the substrate was maintained at 25 cm, and the spray pressure was kept constant at the default setting of 0.1 MPa. The deposited hydrophobic films were subsequently cured in a vacuum oven at 100 °C for 1.5 hours to ensure complete drying and to eliminate any residual solvents or unreacted materials



**Figure 1.** Schematic formulation of fluorinated silica thin sol gel synthesis

### Characterization

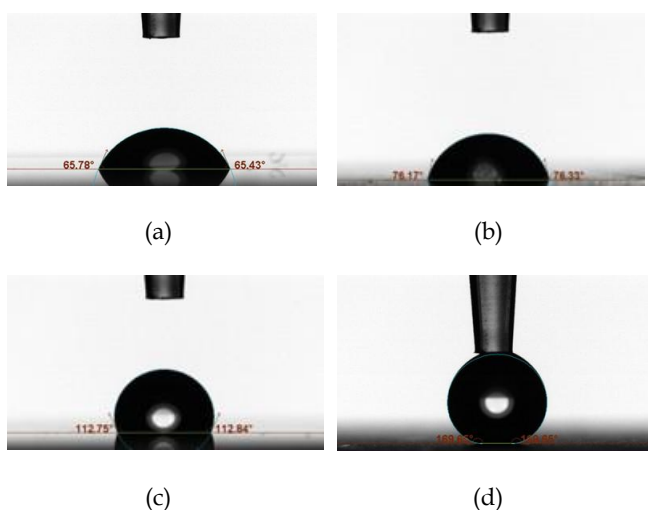
The water contact angle (WCA) was measured by using Attention Theta Lite Optical Tensiometer (Biolin Scientific, TL 100) by sessile drop method at room temperature. Wetting liquid used for contact angle

measurements was distilled water. Fourier transform infrared (FTIR) spectra of the samples were measured by the ATR technique using an FTIR spectrophotometer (Bruker Tension II). The surface roughness was investigated by using Atomic Force Microscope (JPK Nanowizard 2) using contact mode method. The morphology of the samples was measured by using Field Emission Scanning Electron Microscope FESEM (Carl Zeiss, Gemini SEM 500). The optical transparency was determined by using UV-Visible Spectroscopy (Shimadzu, UV-1800) in the visible light region.

## Result and Discussion

### Water Contact Angle

The water contact angles (WCA) of the pristine glass microslide and those coated with various hydrophobic formulations as shown in Figure 2. The uncoated glass exhibited the lowest contact angle of  $65.4^\circ$ , reflecting its intrinsically hydrophilic nature. In contrast, the TEOS-coated surface displayed a slightly higher contact angle of  $76.2^\circ$ , which can be attributed to the absence of a fluorinated or hydrophobic agent, consistent with previous findings (Parvate et al., 2020).



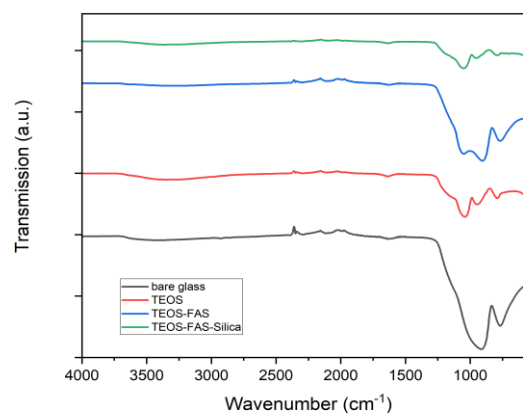
**Figure 2.** Water contact angle images of a bare glass and different thin sol gel coating surface: (a) Bare glass; (b) TEOS; (c) TEOS-FAS; and (d) TEOS-FAS-SiO<sub>2</sub>

In contrast, the TEOS-FAS coating exhibited a markedly higher water contact angle of  $112.8^\circ$ , indicating the transition into the hydrophobic regime where water repellency begins to manifest on the glass surface. This enhancement can be attributed to the incorporation of long-chain fluoroalkylsilane molecules, which reduce surface energy and promote water repellency (Keskin et al., 2025). As for TEOS-FAS-SiO<sub>2</sub> where silica nanoparticles were functionalized with fluoroalkylsilane were introduced into the formulation, the surface achieved superhydrophobicity, with a

contact angle reaching  $169.7^\circ$ . Such behavior closely resembles natural examples of extreme water repellency, as observed on taro leaves and dragonfly wings, where water droplets bead and roll off effortlessly (Bhushan et al., 2009).

### FTIR analysis

The FTIR spectra that reveal distinct absorption bands corresponding to the characteristic chemical bonds within the coating systems. The bare glass displays prominent Si-O-Si stretching and bending vibrations near  $1100\text{ cm}^{-1}$  and  $800\text{ cm}^{-1}$ , respectively as shown in Figure 3. The TEOS thin sol gel film exhibits intensified Si-O-Si bands, confirming successful sol-gel condensation and network formation. With the incorporation of FAS, additional peaks emerge in the range of  $1200\text{--}1140\text{ cm}^{-1}$ , which are attributed to the C-F stretching vibrations of the fluoroalkyl functional groups, verifying the chemical attachment of FAS onto the silica network. The identified bonds from IR measurements can be found in the Table 1. The TEOS-FAS-Silica coating shows further peak broadening within this region, suggesting enhanced interfacial interactions between the silica nanoparticles and the fluoroalkylsilane matrix (Gao & He, 2013). This synergistic effect contributes to the development of a hierarchical micro-nano surface structure that underpins the coating's superhydrophobic behavior (Hao et al., 2021).

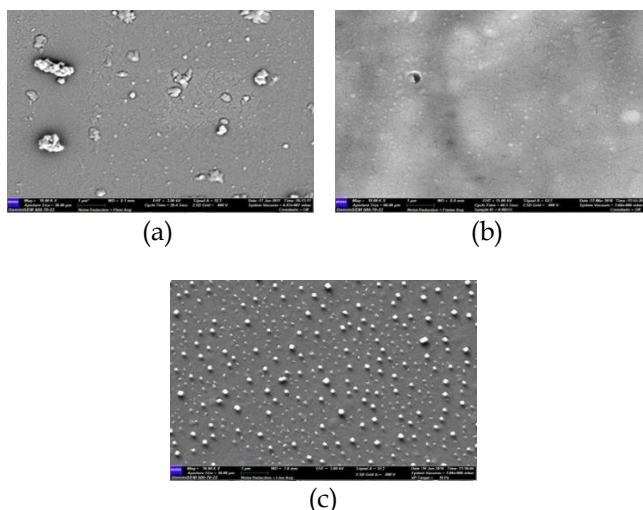


**Figure 3.** FTIR spectra of a bare glass and different thin sol gel films

**Table 1.** Infrared bands identified in thin sol gel films from FTIR measurements

Chemical bond	Wavenumber (cm <sup>-1</sup> )
Si-O-Si	1211, 1068 and 805
Si-OH	974
Si-O-C	1136 and 771
C=O	1722
C-F	1200-1140
-OH	3389 and 1669

## FESEM



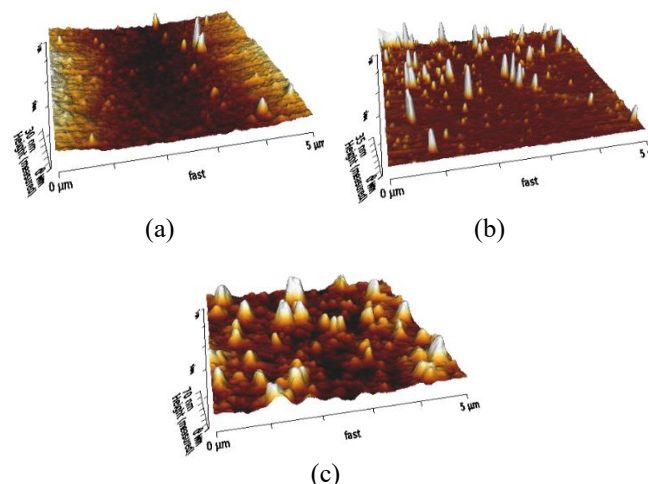
**Figure 4.** FESEM images for different sample of thin sol gel films (10 kx magnification): (a) TEOS; (b) TEOS-FAS; and (c) TEOS-FAS-SiO<sub>2</sub>

Figure 4 shows the surface morphology of the thin sol gel films. They exhibit distinct microstructural features governed by their respective compositions. The TEOS coating (Figure a) presents an uneven and partially aggregated surface, typical of silica networks formed through the hydrolysis and condensation of TEOS (Hagemans et al., 2018). With the incorporation of FAS (Figure b), the surface becomes noticeably smoother and more uniform, indicating that the fluoroalkylsilane molecules enhance film homogeneity by lowering surface energy and improving particle dispersion (Kassaun & Fatehi, 2025). In contrast, the TEOS-FAS-Silica coating (Figure 4c) displays a densely packed distribution of nanoscale particles, forming a hierarchical micro-nano structure that significantly increases surface roughness (Wang et al., 2017). This morphology correlates well with the observed superhydrophobic behavior, where the combination of enhanced surface roughness and the presence of low-surface-energy fluorinated groups facilitates the entrapment of air pockets, thereby minimizing liquid-solid interfacial contact (Kandeloo et al., 2025).

## AFM

The surface topography of the coatings prepared from TEOS sol, TEOS-FAS, TEOS-FAS-Silica, was characterized using Atomic Force Microscopy (AFM), as shown in Figure 5. The images were acquired in contact mode over a scan area of  $5 \times 5 \mu\text{m}^2$ . Distinct surface morphologies were observed for the different formulations. The relatively low roughness and hydrophobicity observed for the TEOS sol coating (Figure 4a) can be attributed to the presence of isolated silica islands sparsely distributed on the substrate

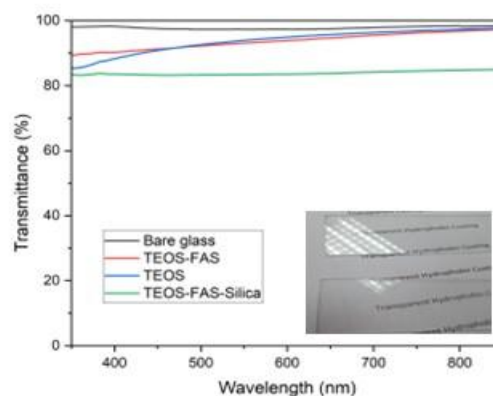
surface (Vidal et al., 2019). With the introduction of FAS (Figure 4b), the surface becomes noticeably rougher and more compact indicating enhanced film uniformity and partial surface modification by fluoroalkylsilane groups. The TEOS-FAS-Silica coating (Figure 4c) exhibits sharp, densely packed, and homogeneous silica features due to the addition of silica nanoparticles that contribute to increased surface roughness and hydrophobicity (Aspinall & Khutoryanskiy, 2023).



**Figure 5.** AFM images of different sample of thin sol gel films: (a) TEOS; (b) TEOS-FAS; (c) TEOS-FAS-Silica

## UV Visible

The UV-Vis transmittance spectra of bare glass and thin sol gel films. Bare glass exhibits the highest transmittance (>98%) across the visible range as shown in Figure 6. The TEOS and TEOS-FAS coatings maintain high optical transparency, with transmittance values increasing from approximately 90% at 400 nm to >95% near 850 nm. In contrast, the TEOS-FAS-Silica coating shows a moderate reduction in transparency, remaining between 83-87% throughout the spectrum, which is attributed to additional light scattering from the incorporated silica nanoparticles (Bagwe et al., 2006).



**Figure 6.** Transmittance degree at visible region of different samples of thin sol gel film

Overall, all coatings preserve good transparency, with TEOS and TEOS-FAS demonstrating optical performance closest to that of bare glass confirming that the sol-gel-derived films remained optically uniform and transparent (Chen et al., 2025). The excellent optical clarity of these thin sol gel coating films makes them promising candidates for applications in transparent protective layers, optical components, and self-cleaning glass surfaces.

## Conclusion

The synthesis and characterization of transparent hydrophobic thin sol gel fluorinated silica coating were successfully prepared. The water contact angle measurements indicate that the coating containing TEOS-FAS-Silica exhibits significantly higher hydrophobicity compared to that with TEOS and TEOS-FAS samples. FTIR analysis verifies the successful grafting of silica and onto the FAS-TEOS network, as evidenced by the emergence of Si-O-C stretching peaks resulting from the reaction between Si-OCH<sub>3</sub> and surface -OH groups. FESEM observations corroborate the presence and deposition of silica microparticles on the substrate surface. UV-Vis spectra confirm that all samples maintain high optical transparency, whereas AFM analysis reveals a progressive increase in surface roughness with the incorporation of silica as a roughness agent that was employed in the study.

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## Author Contributions

All authors jointly contributed to the writing of this article.

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## Conflicts of Interest

The authors declare no conflict of interest.

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