# Synthesis and Characterization of Superhydrophobic Alumina-Silane Sol-gel Coating

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*Abstract*— **Superhydrophobic surfaces have attracted significant research interest because of their use for design of water-repellent and self-cleaning coatings. A superhydrophobic surface can be created by modifying a rough surface with a material of low surface energy through a sol-gel coating technique. Adjusting the amount of precursor and the low surface energy material can optimize the hydrophobicity of the coating. In this work, different ratios (4:1, 3:2, 1:1 and 2:3) of silane: alumina coatings were synthesized and applied on glass substrates through sol-gel method. Characterization of the coated samples showed that volume ratios of silane sol and alumina sol in sol-gel coating affected the superhydrophobicity of the coated glass substrate. The solution of alumina sol and silane sol with ratio 4:1 produced superhydrophobic films with contact angle for water larger than 150î and FESEM images showed very intense roughness compared to the other ratios**.

***Index Terms***— **Alumina, Superhydrophobic, Sol-gel coatings Silane**

## I. INTRODUCTION

Surfaces with a water contact angle higher than 150° and low contact angle hysteresis (superhydrophobic or ultra-hydrophobic surfaces) have attracted a lot of interest because of their use for design of water-repellent and self-cleaning coatings. This is particularly interesting for various applications such as the enhancement of surface properties of outdoor windows, glasses, shower boxes, textiles finishes, anti-bio fouling paints for boats, biomedical applications, eyeglasses, self-cleaning windshields for automobiles and stain resistant textiles [1-4]. Superhydrophobic behavior as first observed on the leaves of the lotus plant has been attributed to the superhydrophobicity of the leaves. Water droplets easily roll off the leave surface, collecting and removing contaminants due to the presence of wax crystalloids on top, representing a combination of roughness and low surface free energy [3]. Air is trapped between water droplet and surface, which decreases the contact area between them. Wetting contact angle between the liquid–gas and liquid–solid interfaces is used to characterize the nature of solid–fluid interactions. Some biological surfaces exhibit the amazing property for not being wetted by water [5].

The limited contact area of the solid surface with water causes the inhibition of chemical reactions or bond formation through water on a superhydrophobic surface. Consequently, other phenomena could be reduced on such a surface, for example, snow adherence, corrosion, disease transmission, friction drag, oxidation, and current conduction [6]. Either making a rough surface from a low surface energy material or modifying a rough surface with a material of low surface energy can create a superhydrophobic surface. The former method has the advantage of simplicity at most a one-step process. However, there is a limited choice of material with low surface energy. So, creating a rough structured subtract and then redesign its surface with low surface energy property has attracted more attention due to its freedom in selecting the suitable material [7]. Most methods such as mechanical stretching, laser/ plasma/chemical etching, lithography, layer-by-layer and colloidal assembling, electrical/chemical reaction and deposition, electrospinning and chemical vapor deposition are only suitable for planar surfaces and surfaces of limited size. Moreover, it is difficult to control the formation of the surface structure and sometimes the processing could be complicated and time consuming [8]. Sol-gel processing, on the other hand is simpler and relatively convenient for the synthesis of advanced material and applying them as surface coatings [9,10]. The sol-gel chemistry can provide efficient incorporation of organic components into the inorganic polymeric structures in solution under extraordinarily mild thermal conditions [11]. Sol-gel technology is also more economical and produces strong adhesion of coating to substrate due to chemical bonding [12]. This paper reports the successful application of alumina sol-gel on glass substrate by spin coating. Contact angles were measured and microstructure of coating were analyzed to determine the superhydrophobicity of the coating.

## II. MATERIALS AND METHOD

The coating was prepared using alumina sol and water repellent agent, silane sol. Isopropyl alcohol (*i*-PrOH), aluminium sec-butoxide (ASB) and ethylacetoacetate (EAcAc) were used as starting materials for the preparation of alumina sol. *i*-PrOH was added to ASB as a drying control chemical additive (DCCA), mixed and stirred at room temperature for 1 hour. EAcAc was then added to the mixture and stirred for 3 hours. EAcAc acts as a chelating agent and can prolong the lifetime of the sol. Finally, water diluted with *i*-PrOH was added to the mixture. Silane sol was prepared by mixing and stirring heptadecafluorodecyltrimethoxysilane (FAS) with *i*-PrOH before adding diluted *i*-PrOH. Five (5) samples of coating solutions with varying volume ratios of

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alumina sol and silane sol were prepared as listed in Table 1. The coating solution was spin coated on a glass substrate at a rotational speed of 2,000 rpm for a holding time of 30 s. The coating was then allowed to dry for 24 h at ambient temperature. Dried coating was then subjected to treatment at 60°C in hot water for 20 min for the precipitation of nano-structured film. After heat treatment, it was oven dried at 100°C to allow densification of coating for another 24 h. A contact angle meter was used to measure the angle between the tangent line of a water droplet and the substrate surface. The microstructure of the coated substrate was analyzed under a Zeiss Supra 40VP field emission scanning electron microscope (FESEM).

**Table 1** Volume Ratio of Silane Sol and Alumina Sol

| Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 |
|---|---|---|---|---|
| 4:1 | 3:2 | 1:1 | 2:3 | 1:4 |

## III. RESULTS AND DISCUSSION

Measurements of contact angles of water droplets were taken for both uncoated and coated substrates as shown in Figure 1. The contact angle of water on uncoated glass substrate shown in Figure 1(a) was 58.21°. For coated substrates low surface energy is introduced by silane sol, while roughness is introduced by alumina sol. The contact angles for different volume ratios of silane sol and alumina sol (4:1, 3:2, 1:1 and 2:3) were 115.588°, 134.607°, 148.62° and 136.607°, respectively (Figures 1(b) – (d)). These measured values were lower than 150°. When the volume ratio of silane sol and alumina sol was 1:4, the contact angle was 152.315°, which was higher than 150°. The contact angle values were plotted against the volume ratio of alumina:silane sol as shown in Figure 2. Decreasing amount of silane sol gives higher contact angle. However, for the sample with 2:3 ratio the value is slightly lower than the sample with 1:4 ratio but still higher than the coated substrate with 1:1 ratio. This could be due to the unevenness of coating on the glass substrate.

Figure 3 indicates the FESEM images of the coated substrate morphology with different volume ratios of the silane sol and alumina sol. The FESEM images show the formation of flower-like microstructures formed on the surface of the film that was dried at room temperature and then immersed in hot water treatment at 60 °C for 20 minutes. Surface with 4:1 ratio silane : alumina coating (Figure 3 (a)), ratio 4:1 shows low roughness compared to the coating with ratio 1:4 (Figure 3(b) that shows very intense and fine roughness.

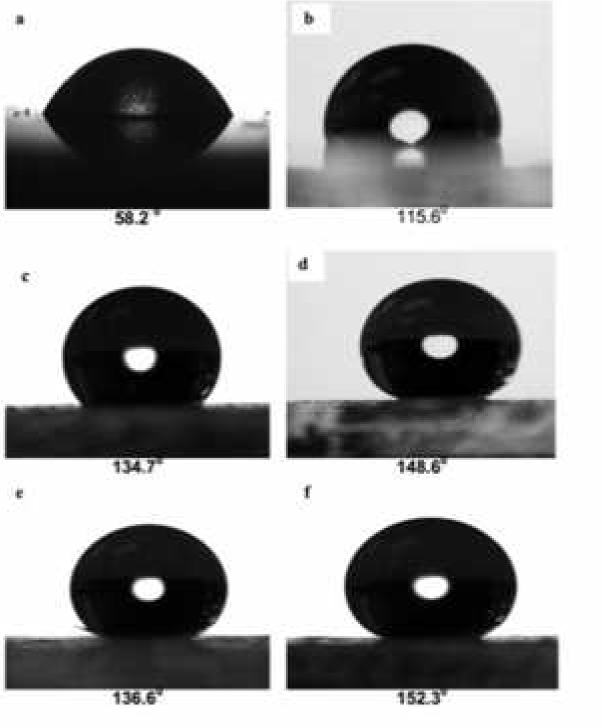


**Fig. 1.** Contact Angle Meter images of water droplets on uncoated glass (a) and coated glass substrates with volume ratios of silane sol and alumina sol (b) 4:1, (c) 3:2, (d) 1:1, (e) 2:3 and (f) 1:4

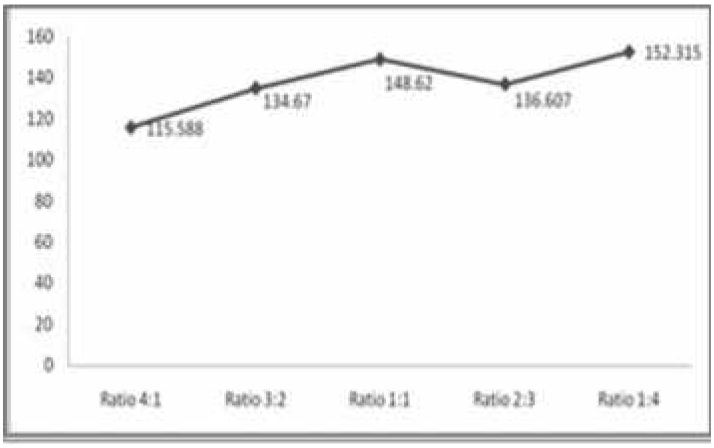
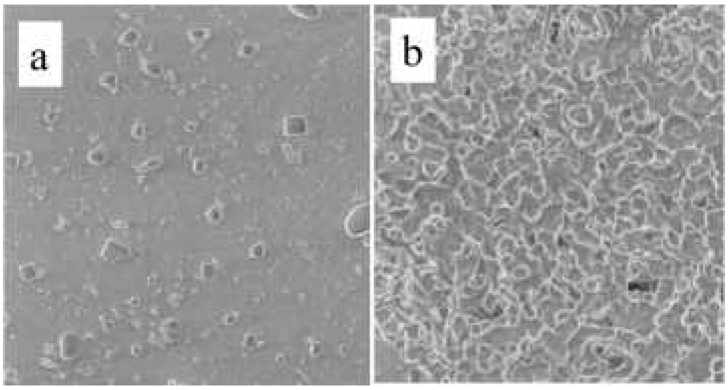


**Fig. 2.** Contact angle measurements versus ratios of silane sol and alumina sol



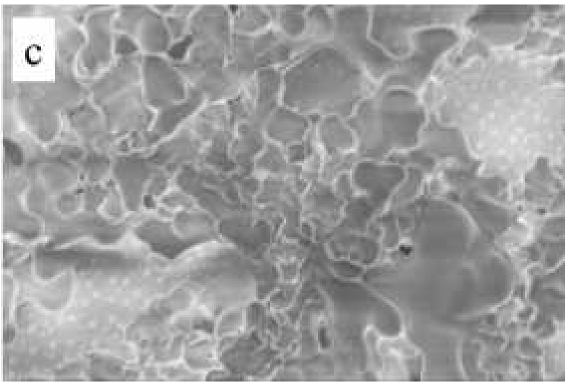


**Fig. 3.** FESEM images of the coated substrate morphology with different volume ratios of the silane sol and alumina sol (a) 4:1, (b) 1:4, and (c) 1:1

In Figure 3(c), where the ratio is 1:1, large roughness is observed compared to Figure 3(b). This implies that the rougher the surface, the higher is the degree of contact angle. This is due to the large amount of entrapped air on the surface of the substrate. Air trapped between the water and the substrate's surface decreases the contact area. Contact angle above 150° indicates surface topography to be in an optimized range to trap sufficient air, which subsequently reduces the contact area between the surface and the water droplets. However when the contact angle is less than 150°, the surface roughness is not in the optimum range for superhydrophobicity.

## IV. CONCLUSIONS

From the results, it can be concluded that superhydrophobic coating films have been successfully synthesized and applied on glass substrates through sol-gel method. Characterization of the coated samples show that volume ratios of silane sol and alumina sol in sol-gel coating affected the superhydrophobicity of the coated glass substrate. Roughness was found on the surface of the films after undergoing the hot water treatment. The solution of silane sol and alumina sol with ratio 1:4 produced superhydrophobic films with contact angle for water larger than 150° and showed very intense roughness compared to the other ratios.

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