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Synthesis of Highly Transparent and Hydrophobic Aerogel Coatings

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Abstract:

The sol-gel processing technique is used for coating on various types of substrates like glass, metals, polymers etc. There is a need to have superhydrophobic aerogel coatings for anticorrosive applications on different metallic surfaces. Furthermore, the hydrophobic and transparent coatings are essential for the protection of statues and sculptures to prevent their deterioration with time since they contain natural minerals such as aluminium and silica that react with moisture present in the atmosphere. Therefore, successful attempts have been made to produce transparent and hydrophobic coatings using sol-gel processing. In the present work, considering the importance of hydrophobic and transparent aerogel coatings on glass windows and wind screens, systematic, step by step experimental investigations have been conducted to coat the silica glass substrate by the sol-gel processing using the simple dip coat method. By this method uniform, highly transparent (> 85% at 700 nm) and hydrophobic coating on glass substrate with the contact angle of water exceeding 94o were obtained.

Introduction:

The exceptional properties of the aerogels in bulk form are very well known. They have very low thermal conductivity (<0.05 W/mK), very high surface area (~1600 m^2/g) and extremely low-density (<20 kg/m^3) [1]. Due to such fascinating properties, it has tremendous scientific and technological applications [2]. However, silica aerogels in the form of thin films or coatings are needed for certain applications. For example, electronic applications such as thermal insulation require very thin aerogel films on a substrate and the window applications need a monolayer of hydrophobic and transparent silica coatings on the glass substrates [3,4].

In this paper, we report a very simple and effective dip coating method of preparing such hydrophobic and transparent silica coatings on the glass substrates using methyltrimethoxysilane (MTMS) as the precursor by single step sol-gel process.

Experimental Procedure and Characterization Methods:

The MTMS precursor was diluted in methanol (MeOH) solvent and the hydrolysis and the condensation reactions were carried out under the basic conditions using ammonium hydroxide (NH_4OH) as the base catalyst. Clean and dry glass substrates were held vertically dipped in the alcosol for various time intervals. Then these substrates were removed from the sol, slowly and kept in an oven (Termaks, Norway) at 200ºC for 2 hours so that the thin film deposited on the glass substrate is totally dried. Initially, the MeOH/MTMS molar ratio, M was varied from

1.76 to 14.08. Later, in order to achieve the best quality transparent and hydrophobic coatings, the base catalyst concentration and the water quantity was systematically varied. Finally, to study the effect of deposition at higher temperature, the silica sol was maintained at different temperatures ranging from 27ºC to 50ºC and the deposition was taken.

The hydrophobicity was characterized in terms of contact angle measurements of a water drop placed on glass substrate. The presence of various organic groups responsible for the hydrophobic nature of the surface was confirmed by the Fourier Transform Infra-red (FTIR) spectroscopic studies. The percentage of transparency of the coated surface was tested in a spectrophotometer.

Results And Discussion:

Effect of MeOH/MTMS (M) molar ratio:

To study the hydrophobic nature of the coatings on the glass substrates, the contact angle (θ) of a water droplet on the substrate surface was measured. The molar ratio of Methanol (MeOH)/ Methyltrimethoxysilane (MTMS) was varied from 1.76 to 14.08. It was found that the contact angle increased from 52º to 73º with the increase in the M value from 1.76 to 7.04. But, with the further increase in M to 14.08, the contact angle decreased to 66º as shown in table 1. Hence, further experiments were carried out by keeping M value fixed at 7.04.

Table 1. Some physical properties of the hydrophobic coatings for various MeOH/MTMS molar ratios.

Molar Ratio MeOH/MTMS	Deposition Time	Gelation Time	Contact Angle	Optical Transparency (%)
1.76	50 min.	1 hr.	62º	98%
3.52	1 hr.15 min.	1 hr.30 min	64º	97%
7.04	2 hrs.	2 hrs.30 min.	73º	90%
14.08	3 hrs.30 min.	4 hrs.	66º	96%

Effect of deposition time:

The glass substrates were kept dipped in the silica sol for various time intervals from 10 minutes to 2 hours from the instant the sol was prepared. The maximum deposition time was decided by taking into account the gelation time. For example, in the case of the sol with the molar ratio, M=1.76, the gelation time was one hour and so the maximum deposition time was 50 minutes, while for M=14.08, the gelation time was ~4hours and the deposition was taken upto 3hours and 30 minutes. From figure 1, it is clear that in all cases, as deposition time increases hydrophobicity increases. This is because, when the silica sol transforms into silica gel, particle formation is completed, resulting in the formation of a thin, uniform, and fragile particle network on the glass substrate. If the glass substrates are taken out much prior to the gelation, non-uniform deposition was observed. This can be attributed to incomplete hydrolysis and condensation reactions. This resulted in lesser hydrophobicity, which is measured in terms of contact angle of a water drop on the surface of the coated substrates.

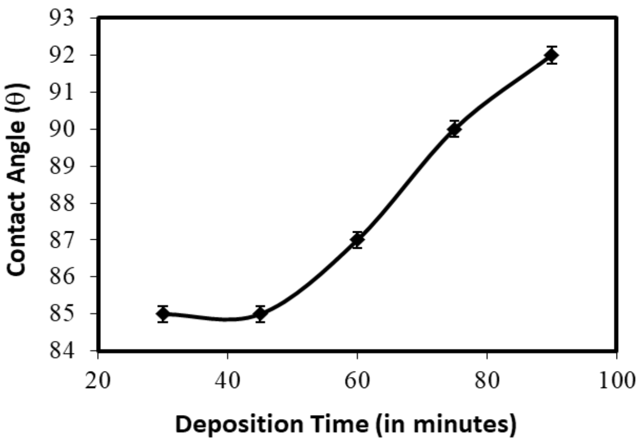

Figure 1: Variation of contact angle with deposition time.

Effect of water quantity:

To investigate the influence of water in the silica sol on hydrophobic aerogel coatings, the molar ratio of basic water to MTMS (H) was systematically varied from 1.98 to 3.96. With the increase in water quantity in the sol, the gelation time decreased from 4 hours to 40 minutes. Accordingly, the deposition time also reduced from 3 hours and 30 minutes to just 30 minutes. Initially, the contact angle increased from 73° to 92° with the increase in water quantity from 1.98 to 3.56. It was observed that the contact angle decreased to 80° when H was increased to 3.96 as shown in figure 2.

Since water directly participates in hydrolysis, a lower water content produces fewer hydrolysable monomers (Si-OH groups) for cross-linking. An optimum water content at H = 3.56 promotes extensive hydrolysis of metal alkoxide groups, forming a highly branched polymeric network. However, at higher H values, the contact angle decreases because of trapped water and increased uncondensed silanol groups [3].

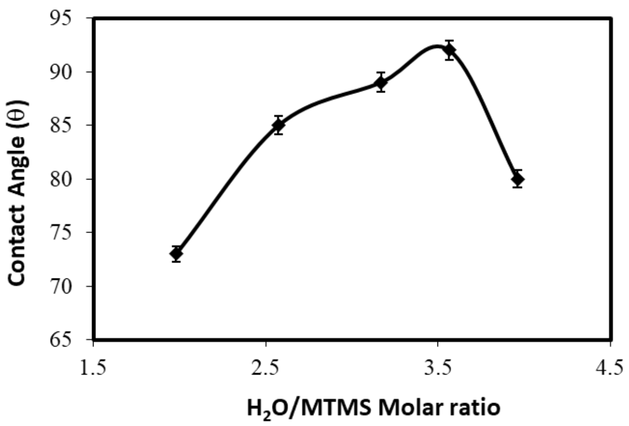

Figure 2: Variation of contact angle with H_2O/MTMS molar ratio at constant MeOH/MTMS molar ratio of 7.04

Effect of base catalyst concentration:

To determine the influence of base catalyst (NH_4OH) concentration (B) the alcosol was prepared by keeping the molar ratio of MTMS: MeOH: H_2O constant at 1:7.04:3.56 and the B value was varied from 6M to 13.36M. The contact angle was observed to increase, reaching a maximum of 94^o for B=8M (as shown in figure 3) and it was less for both B<8M and B>8M. As the B value increases upto 8M, the particle size increases and hence the connectivity between the particles increases which results in a uniform silica network. However, for B values greater than 8M, the rate of condensation becomes very fast that leads to smaller particle size. As a result the connectivity between the particles decreases and the network becomes uneven. Thus, in order to achieve the best results, B value was fixed at 8M.

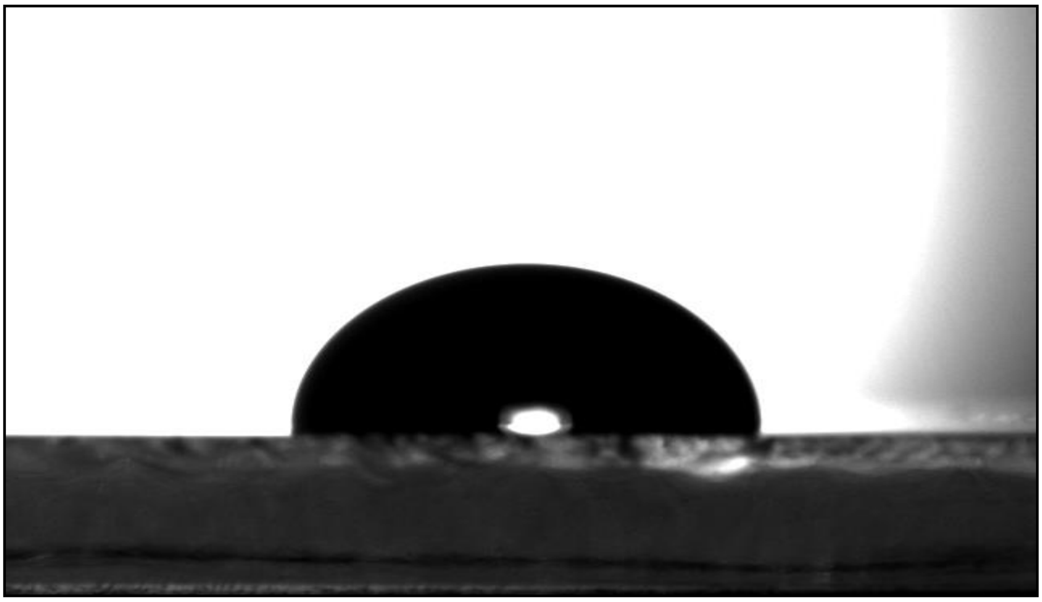

Figure 3: A water droplet on the the surface of silica aerogel coating for M= 8

Effect of deposition temperature:

In order to study the effect of deposition temperature, the silica sol was maintained at various temperatures from 25ºC to 50ºC in an oven (Termaks, Norway). It was found that at higher temperatures, the coating was more uniform, transparent and very much adherent. As the deposition temperature increased from 25ºC to 45ºC, the contact angle increased from 92^o to 96^o. Due to the faster gelation at higher temperatures, the deposition time reduced. At temperatures >45ºC, the deposition could not be taken due to a very short gelation time (<10 minutes).

3.5 FTIR studies

The presence of various organic compounds, which made the surface hydrophobic, was confirmed by the FTIR studies, which is shown in figure 4. The figure shows intense peaks at 1270 and 840 cm^{-1} corresponding to Si-C bonds [5,6]. The peaks around 2900 and 1400 cm^{-1} are due to the C-H bond [7]. Very small peaks corresponding to O-H bonding were observed at around 3200 and 1650 cm^{-1} which confirms the hydrophobic nature of the coatings.

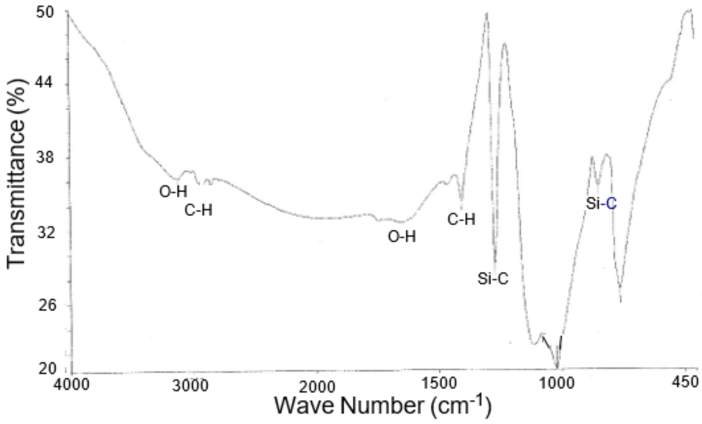

Figure 4: FTIR spectra of silica aerogel coatings with for M = 8

Conclusion:

Transparent and hydrophobic coating of silica aerogels on glass substrates was produced by a single-step sol-gel process that used the dip coat method and MTMS as a primary precursor.

Highly transparent, well adherent and uniform silica aerogel coating was obtained at a deposition temperature of 45°C for the molar ratio of MTMS: MeOH: H_2O at 1: 7.04: 3.56 with NH_4OH (8M) as the base catalyst. The contact angle between water droplet and aerogel coated surface was observed to be 94°.

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