The Impact of Silanization Techniques on the Wettability of Silica Surfaces: Insights from TGA, Isotherm, and Contact Angle Studies

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Introduction

Purpose:

- Examine the impact of silanization on the wettability of porous silica surfaces
- Study how the wettability affects water adsorption on porous beads

Background:

- Silanization

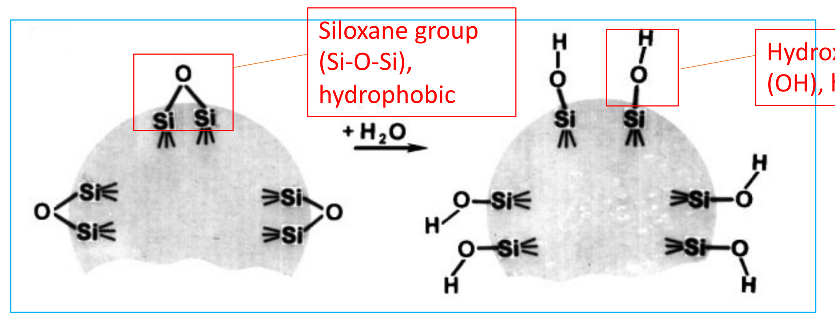


Figure 1: Rehydroxylation process for silica to form hydroxyl (OH) groups on its surface [1]

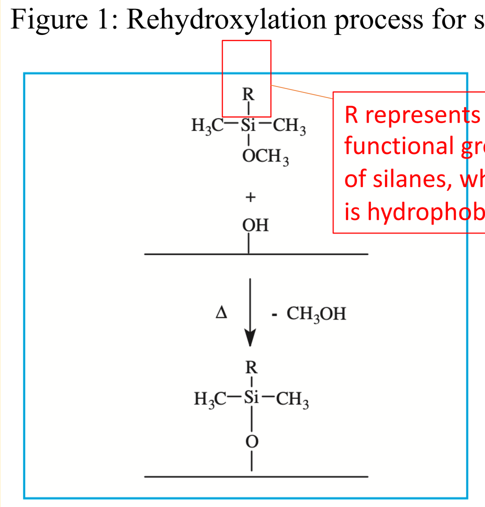


Figure 2: Anhydrous deposition of silanes. The silane molecules covalently bind to the hydroxyl groups [2].

- Contact Angle and Wettability

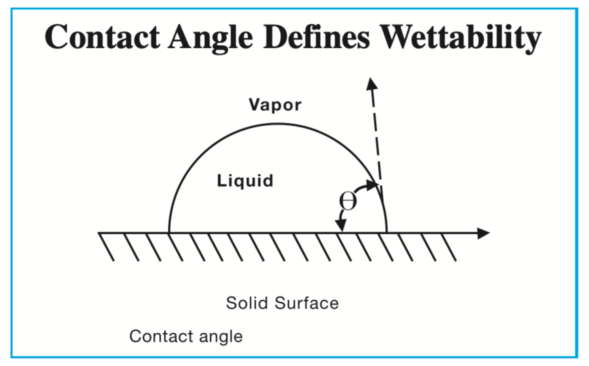


Figure 3: A surface is said to be wetted if a liquid spreads over the surface evenly without the formation of droplets, which can be quantified by contact angle Θ in degrees [2].

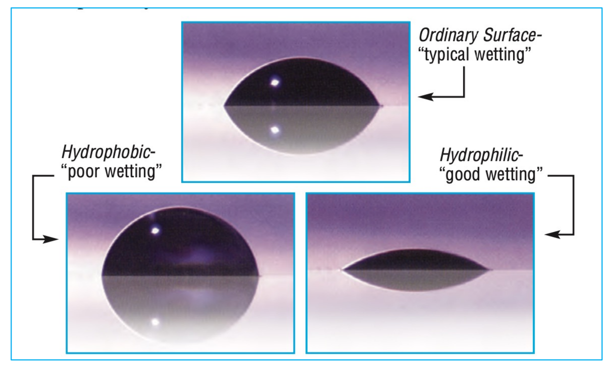


Figure 4: The pictures show a quantitative method for defining the relative degree of interaction of a liquid with a solid surface. Specifically, if the contact angle of water is less than 30°, the surface is designated hydrophilic ("good wetting"); surfaces with contact angles greater than 90° are designated as hydrophobic ("poor wetting") [2].

Methods

Main materials applied:

- Methacryloxypropyltrimethoxysilane (MPTMS, $C_{10}H_{20}O_5Si$)
- Pure silica (SiO_2, porous beads and slide)
- Heptane (C_7H_{16})

Method of Silanization:

- Step 1: Cleaning Silica
  - to remove oil-like substance
- Step 2: Heat Treatment
  - to remove water and add hydroxyl groups to the surface of silica
- Step 3: Silanization
  - silane molecules covalently bind to the hydroxyl groups. The surface is then modified.

Key Factors of Silanization: anhydrous environment, silane concentration, choice of silane and solvent, temperature in heat treatment

Analytical Method:

1. Contact Angle Measurement

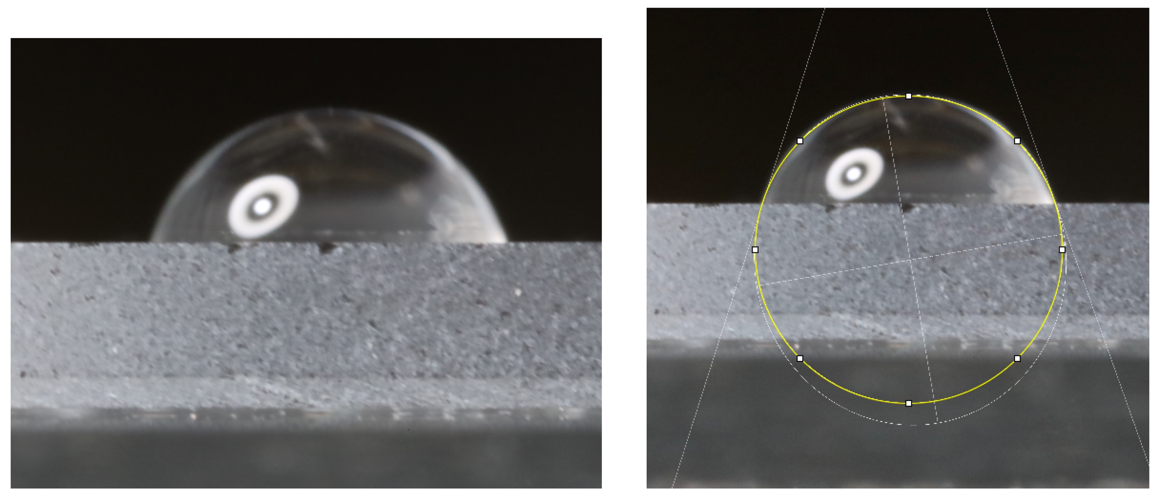


Figure 5,6: An example image of how the water interacts with the surface of silanized silica slide (left image). The contact angle is then measured in Image J (right image).

2. Thermogravimetric Analysis (TGA): Thermogravimetric Analysis (TGA) is a key analytical technique used in our research to evaluate composition changes of silanized silica surfaces. By measuring weight loss as a function of temperature, TGA helps determine the extent of silane bonding to the silica substrate and identifies any volatile components. This method provides essential data on the effectiveness of silanization.

3. Adsorption Isotherm Analysis: Isotherm analysis, specifically adsorption-desorption isotherms, plays a crucial role in this research by relating wettability to water adsorption on silanized silica surfaces. This technique measures the amount of water vapor adsorbed by the substrate at different pressures, providing insights into the surface texture and structural changes resulting from the silanization process. Understanding these properties is vital for tailoring silane coatings to enhance wettability and functionality in various industrial applications.

Results and Discussion

1. Contact Angle Measurement

The measurement shows a value of 66.42 degrees, as an average of contact angle over 50 droplets. Therefore, the surface of the silanized silica is hydrophobic, or poor wetting, which is consistent with hydrophobicity of functional groups.

2. TGA

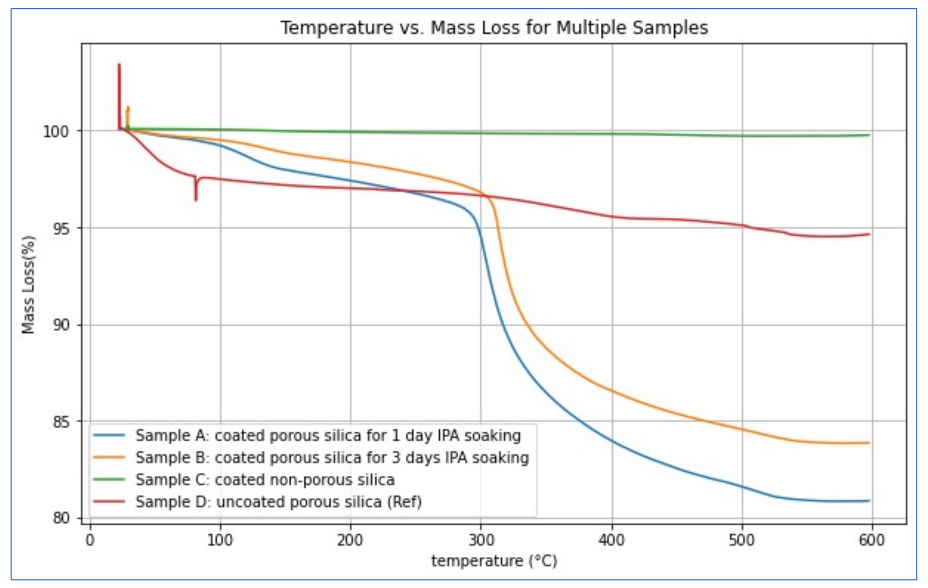
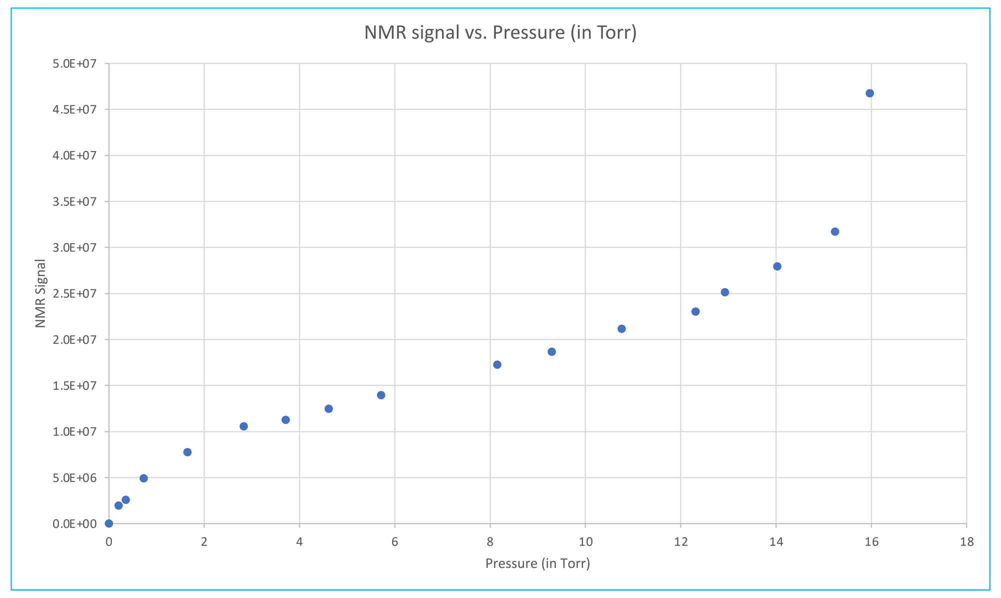


Figure 7: A drop of weight loss percentage for porous silica sample indicates the silanes are successfully coated on the surface of silica

3. Isotherm



What does the graph of isotherm show?

1. Initial Flat Trend: At lower pressures, there is a relatively flat trend where the NMR signal does not significantly increase with pressure. This could suggest that initially, water vapor is not being adsorbed in large quantities, possibly due to low wettability.

2. Subsequent Increase: As the pressure continues to increase, the NMR signal shows a noticeable increase. This implies that beyond a certain pressure threshold, there is a significant amount of water vapor adsorption, which could indicate that either the wettability has improved or more adsorption sites have become available, possibly due to the opening of more pores or changes in the surface chemistry.

3. Steep Increase at Higher Pressures: The steep part of the curve at the highest pressures suggests that water vapor adsorption is becoming more pronounced, which might indicate that capillary condensation is occurring within the pores since the silica surface is porous.

References

1. Zhuravlev, L.T. "The surface chemistry of amorphous silica. zhuravlev model." *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, vol. 173, no. 1–3, Nov. 2000, pp. 1–38, https://doi.org/10.1016/s0927-7757(00)00556-2.

2. Gelest. *Hydrophobicity, Hydrophilicity and Silane Surface Modification.*