

WATER GLASS BASED SUPERHYDROPHOBIC SILICA AEROGEL IN DIFFERENT ENVIRONMENTAL OF PREPARATION

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Abstract. Silica aerogel made from water glass is a subject of interest to researchers because of its low cost and safe work. Three different types of silica aerogels based on water glass were prepared and investigated in this work. The effect of stimulating the amberlite resin with and without hydrochloride acid was studied and compared with that of silica aerogel prepared via TEOS as a precursor. Gel time, contact angle, density, surface area, and transparency were studied. FTIR and XRD are used to look at how the bands connect and how big the granules are. Contact angle is used to figure out how hydrophobic the material is. Besides the structural and morphological properties measured by BET and FE-SEM analysis, results indicate that treatments of the resin with or without HCl affected the aerogel properties, where an increase was observed for both the contact angle and surface area. Gel time was slowed. This result was inversely in the case of not adding HCl, where the utilized HCl as a catalyst reduces the water content in wet-gel. The results showed that silica aerogel based on TEOS gave the closest characteristics to those of water glass, with the difference in transparency and gel time.

Keywords: silicic acid, sodium silicate, aerogel, hydrophobicity, contact angle.

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1. Introduction

Silica aerogels are advanced materials and novel in the field of materials science. They consist of 4% of a weak structure from the matrix of silicon dioxide. The remaining 96% is air. Consequently, they are one of the lightest weight solids ever conceived (Fricke, 1988; Rao *et al.*, 2004). An aerogel is made by the sol called the "sol-gel process". which removes the liquid from a wet gel without significant shrinkage or pore collapse. Commonly, the starting point of the sol formation is the mixing of precursors, e.g., Tetraethylorthosilicate TEOS, Tetramethylorthosilicate TMOS, and sodium silicate (Na_2SiO_3), etc. Traditionally, silica aerogels were usually synthesized by the supercritical drying (SCD) process so as to obtain a porous structure (Sagara *et al.*, 2021). Silica aerogel consists of more than 90% air and less than 10% soled silica in the form of a highly cross-linked network structure. These are unique porous materials (Rao *et al.*, 2001) . Having high transparency in the visible light (Pan *et al.*, 2017; Tabata *et al.*, 2010). Very low thermal conductivity (~ 0.01 w/mk) (Brinker & Scherer, 2013; Pan *et al.*, 2017; Zhang *et al.*, 2021), very low sound velocity (< 100 m/s) (Al-Mothafer *et al.*, 2021; Gurav *et al.*, 2010; Prakash *et al.*, 1995) and surface area as large as 1600 m^2/g (Feng *et al.*, 2018; S. He *et al.*, 2015; Hrubesh, 1998; Pan *et al.*, 2017) and low refractive index (~ 1.05) (Khedkar *et al.*, 2019). It's used as adsorption material (Al-sharuee & Mohammed, 2019; Mazrouei-Sebdani *et al.*, 2019). Insulating material (Al-sharuee, 2019; He & Xie, 2015). The production of this monolith generally starts with

the mixing of precursors like Tetraethylorthosilicate TEOS, Tetramethylorthosilicate TMOS, or sodium silicate dissolved in water mixed with a suitable solvent (usually an alcohol) at ambient or slightly elevated temperatures (Guglielmi *et al.*, 2014).

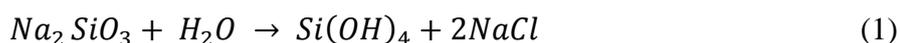
Many researchers have been interested in preparing the aerogel with good specifications and in the easiest ways. Mangesh V. Khedkar and his colleagues show how to make hydrophobic silica aerogels from silicic acid with pH changes. They do this by using a cheap and safe method of drying under ambient pressure. Optical transmittance, thermal stability, hydrophobicity, and high surface area were all shown to be superior at pH 5, according to the researchers (Khedkar *et al.*, 2020). M. Ben Sik Ali and others used ion-exchange resins to remove silica. Some thermodynamic and kinetic features were investigated at various temperatures. The absorbance of yellow or blue-colored silicomolybdic acid was used to determine silica concentrations. Ionic chromatography was used to determine the amounts of anion species. They found Because a strong anion-exchange resin is frequently used in water treatment. (Ali *et al.*, 2004). Steve De Pooter produced aerogels with a thermal conductivity of 22.4 mWm⁻¹K⁻¹ and a standard deviation of 0.3 via an optimized synthesis of ambient pressure dried thermal insulating silica aerogel powder from non-ion exchanged water glass (De Pooter *et al.*, 2018). Yuelei Pan and others prepared the silica aerogel powders by water glass and ambient pressure drying within 4 h. The base concentration proved to have a very important influence on the physical and chemical properties of aerogels. As a result, when the dilution ratio of a water glass as a base catalyst is 3, aerogels with a well-developed mesoporous structure (mean pore size of 20 nm) and superhydrophobicity (contact angle of 160°) can be obtained (Pan *et al.*, 2017). Yajun Huang and others prepared water glass-based aerogel granules under ambient pressure drying. Their characterization results indicated that the resulted silica aerogel has a uniform mesoporous structure and low thermal conductivity (Huang *et al.*, 2019). The superhydrophobic silica aerogel was made by Z. Shao et al. by using sodium silicate, which is less expensive, as the main source of silica and drying it at normal pressure. They got physical properties like less than 10% volume shrinkage, a density of 0.12 g/cm³, a surface area of 684.44 m²/g, a pore volume of 3.55 cm³/g, a contact angle of 146°, and high optical transmission. (Shao *et al.*, 2013). For Mangesh V. Khedkar and colleagues, sodium silicate was made using an ambient pressure drying technique. Sodium silicate sol-gel polymerization was used to positively manufacture hydrophobic silica aerogel via ambient pressure drying. Due to its excellent thermal stability, superhydrophobicity, and huge specific surface area, the resulting silica aerogel has proven its relevance in a variety of applications, including catalysis, coating materials, oil spill cleanup techniques, and insulating materials (Khedkar *et al.*, 2019). Zhuchao Yang and colleagues demonstrated that a hyperplastic and hydrophobic silica aerogel was created using Vinyltriethoxysilane (VTES) and Methyltrimethoxysilane (MTMS), and that it appeared to be a silica-based aerogel with a typical nano-porous structure, a low density, and low thermal conductivity, and a contact angle of 144 degrees (Yang *et al.*, 2019). Another researcher, prepared light hydrophilic silica by the sol-gel neutralization chemical method under ambient temperature and oxalic acid as catalysis. The results showed interconnected macropores with low density in the range. The generated aerogel samples were hydrophilic and floated in the water (Śłosarczyk, 2021). In this work, super-hydrophobic silica aerogel was obtained successfully from water glass at ambient pressure using two methods. Firstly, NH₄OH as a base catalyst for gelation. Another is acid-based catalysis for producing aerogel from sodium silicate.

The two were compared with silica aerogel obtained from TEOS as a precursor. The effects of catalysis and the type of precursor on the physical and chemical structure were studied and analyzed.

2. Materials and Methods

Sodium silicate (Na_2SiO_3) (CAS No. 10213-79-3, M.W.212.14 g/mol, purity 99.9%) used as a precursor with Tetraethylorthosilicat Si (OC_2H_5)₄ (TEOS, 98%) was obtained from Sigma-Aldrich (Germany). For surface modification, it used Trimethylchlorosilane TMCS, > 98% (CH_3)₃SiCl (TCI Japan) and n-Hexane (C_6H_{14} > 98 %), provided by Chemo-LAB (Belgium). Ethanol (99%) was provided by Schariau (Spain). HCl M.W. 36.45 g/mol (99.0%) and Ammonium Solution (NH_4OH M.W. 17.03 g/mol) as acid-base catalysis were provided by CDH (India). (Na^+) from sodium silicate solution by Amberlite (IR-120 Na), which is used as an ion exchange resin.

The precursor employed is sodium silicate, since it is both cheap and plentiful in nature. It was diluted in distilled water at a ratio of 1:4 and the pH were 13. The solution passed through an amberlite ionic exchanging resin in order to remove Na^+ which is packed inside a column to replace it in the solution by protons H^+ . Silicic acid was prepared according to the equation 1:



Three different types of samples have been produced (W1, W2, and W3). W1 prepared according to equation 2, HCl with [5M] was added to the cation resin prior to ionic exchange.



Then the sodium silicate is passed through it to get silicic acid with a pH less than 2. After 10 minutes, add drop by drop of 1N NH_4OH until the pH of the produced Si-OH was 8 ((the amount of added NH_4OH was greater than the W1 sample)) for 2 hrs. and soaked in (50% water+50% ethanol) for 24 hours two times. The surface modification made by TMCS and n-Hexane was kept at a ratio of [1:6 M] under 60 °c for 24 hrs. Then add n-Hexane only for 24 hours. Then, wrap the holder in a small perforated plastic lid and leave it to dry at room temperature for 72 hours. put the samples under 120 °c every 10 °c to get the hydrophobic silica aerogel.

W2 was prepared from condensed silica only with the same molar ratio in W1 and without mixing with silicic acid, and make the same procedures of modification. The third sample W3, add 5ml of condensed silica (TEOS: Eth: HCl [0.1M]) with (1:5: 0.2) to the silicic acid, after 10 min add drop by drop of NH_4OH of 1N (pH for solution 8). It converted to gel after 2 hours, aged for 2hr, and soaked in ethanol for 24 hours two times, the surface modification makes by TMCS and n-Hexane were kept at ratio [1:6 M] respectively under 60 °c for 24 hr. Then add n-Hexane only for 24 hr. two times, then around the holder in small perforated plastic lid and remain it to dry in ambient pressure for 72 hr. After removed all the solutions from modified gel, put it in an oven under 120 °c every 10 °c to get hydrophobic silica aerogel.

The completed of modification and nature of bands vibration were investigated by FTIR spectroscopy (Bruker FTIR Spectrometer ALPHA II, USA). The crystallites were confirmed by XRD analysis (GaliPIX^{3D} X-ray Detector | 2D Hybrid Pixel XRD Detector) using Cu K radiation (1.5406). Also, the morphological and the percentage of

materials was confirmed by FESEM and EDS instruments (FESEM EBSD Instrument: ZEISS SIGMA VP. The bulk density of doped Aerogel was calculated by its mass and volume measurements. The volume of Aerogel was estimated using the formula $V = \pi R^2 h$, where R is the radius and h is the height of the Aerogel. If "m" denotes the mass of the Aerogel, its bulk density equals m/V . While the specific surface area and the average pore volume determined by BET analysis BET examination (BELSORP-mini II). The quantity of N₂ gas adsorbed at different partial pressures ($0.01p/p_{01}$). Finally the hydrophobicity property was examined via contact angle measurements, where a water drop (3μ) was placed on the top surface of a sample. From the measurement of the height (h) and width (w) of the drop, the contact angle(θ) was calculated according to the equation 3 (Khedkar *et al.*, 2019).

$$\theta = 2 \tan^{-1} \frac{2h}{w} . \quad (3)$$

3. Results and Discussion

FTIR analysis

The vibration modes as a result of FTIR spectra, and the hydrophobicity property of a silica aerogel sample, which was cleared in FTIR spectra for all samples, were illustrated in Table 1 and Fig. 1 (a, b, c). The adhesion of the Si-CH₃ group to the gel's surface is very clear. A weak peak at 3650, 3746, and 3753 cm⁻¹ in a silica aerogel sample suggests that a fraction of the Si atom refers to the hydroxyl groups on the surface of the silica aerogel. The stretching band of hydrogen in O-H at 3650 cm⁻¹ a linked molecule of water is responsible for the broad absorption peak centered at (H-O-H). The peaks at 1900 and 1300 cm⁻¹ correspond to -OH and Si-OH. indicating that the silica aerogel surface is unchanged (Bhagat *et al.*, 2008; Cheng *et al.*, 2016). In the adjusted range, the peaks at 750, 1200, and 2900 cm⁻¹ correspond to Si-CH₃ bending. These peaks are ascribed to TMCS altering the silica aerogel surface. The hydrophilic groups Si-OH were replaced by hydrophobic groups Si-CH₃ which are collections after surface modification of the gel with Hexane-TMCS. As evidenced by the disappearance of a peak at 1600 cm⁻¹. The uneven bending and stretching vibrations, which correspond to the bonding of Si-O-Si, show a significant absorption peak at 1010 and 1020 cm⁻¹, respectively. In both modified and unmodified aerogel samples, the silica system was established. (Sarawade *et al.*, 2010; Shi *et al.*, 2017). Table 2 shows the percentage of intensity variation depending on Si-O-Si length.

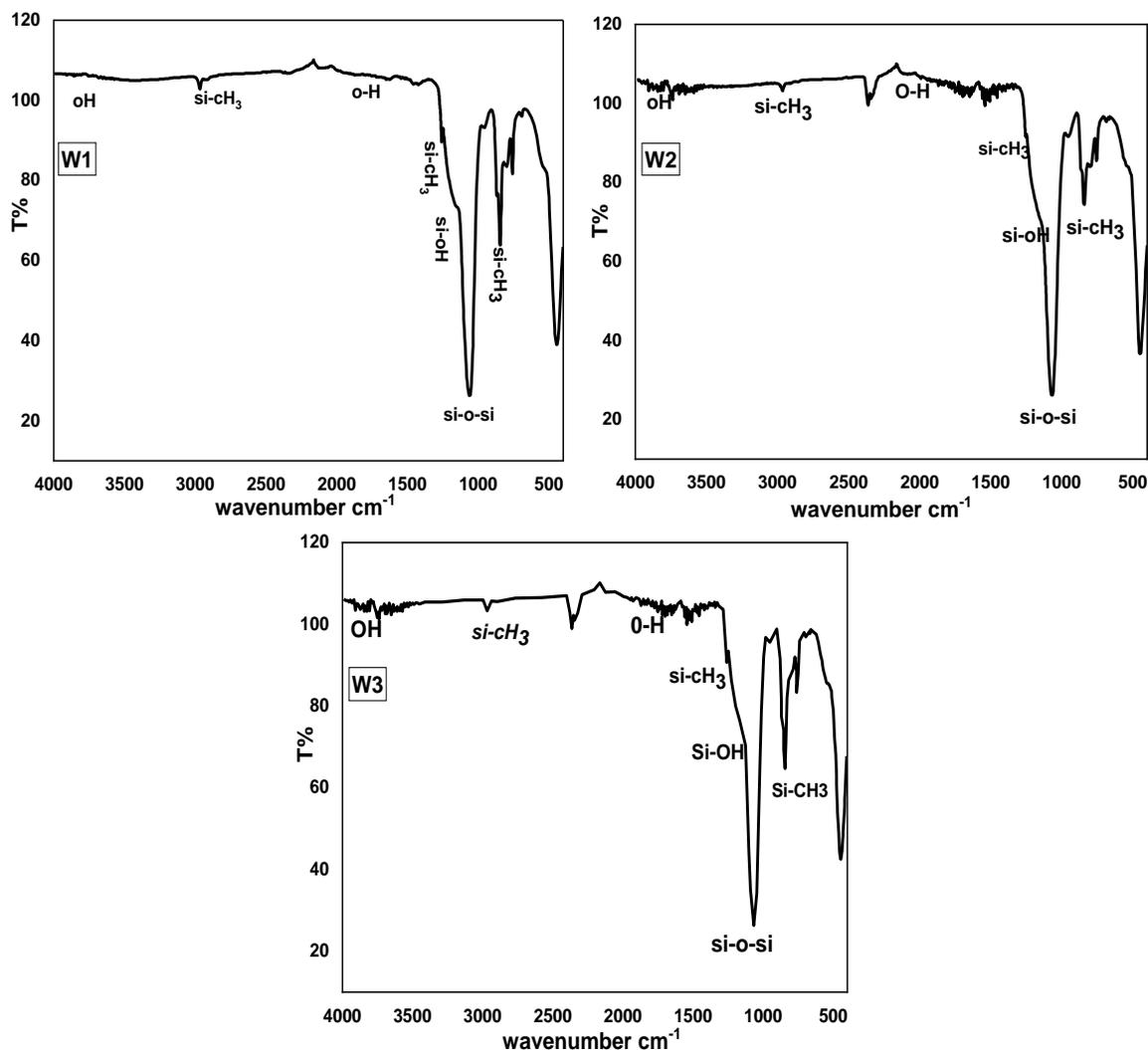
Table 1. The vibration of bands as a result of FTIR examination

Type of vibration Sample	Si-OH H-OH	ν_s C-H	ν_{as} C-H	δ H-O-H	δ_s Si-CH	ν_{as} Si-O-Si	ν_p Si-OH	ν Si-CH	ν_s Si-O-Si
W1	3650	2971	2177	1706	1262	1065	1138	842	488
W2	3746	2968	2364	1774	1266	1067	1126	844	486
W3	3753	2963	2362	2089	1255	1072	1137	841	482

" ν stretching vibration; ν_s symmetric stretching vibration; ν_{as} antisymmetric stretching vibration; δ deformation vibration; δ_s symmetric deformation vibration (bending); ν_p in-plane stretching vibration" (Chandrasekhar *et al.*, 2003).

Table 2. The percentage of intensity variation depended on Si-o-Si length

sample	length	Si-OH	Si-CH	CH	OH
W1	36.55	17.58	4.48	1.317	81.70
W2	35.78	15.49	3.14	2.76	80.43
W3	37.15	14.55	2.65	1.93	79.82

**Fig. 1.** FTIR spectrum for silica aerogel prepared in different procedures

Contact angle measurements

The hydrophobicity qualities can be determined by measuring the contact angle between a water drop supported on the external surface and the surface under consideration. The influence of environmental preparation on contact angle, and the variation in contact angle of the Aerogel's samples for W1, W2, and W3 are represented in Fig. 2 (a, b, and c) respectively. From the figures, with comparing between the W1 and W4 samples, it can be said that the hydrophobicity property is affected by the environmental conditions of preparation. The addition of HCl to resin lowers the contact angle and the transparency property, while the gel time takes three times longer; this lateness may help to complete the modification, leading to the contact angle rising to 150.89° and the surface area to $961.1 \text{ (m}^2\text{/g)}$ as shown in Table 3. In the case of the

W1 sample, more transparency, high surface area, and lowest gel time, the sample seemed to be a light weight whereas the density was 0.096 gm/cm^3 . This explains the high transparency of the sample as shown in Fig.2.

Table 3. Surface area, average pore diameter and volume for silica aerogels prepared in different procedures

Sample	V_m (cm^3/g)	Surface area (m^2/g)	Total pore volume (cm^3/g)	Mean pore diameter (nm)	Average Particle size	Density (g/cm^3)	Gel time (min)	Contact angle
W1	158.78	961.1	2.23	12.941	38,618	0.156	120	150.89
W2	181.62	790.49	2.84	14.42	43,23	0.096	15	138.70
W3	159.1	692.47	1.67	9.6634	39,49	0.135	30	141.68

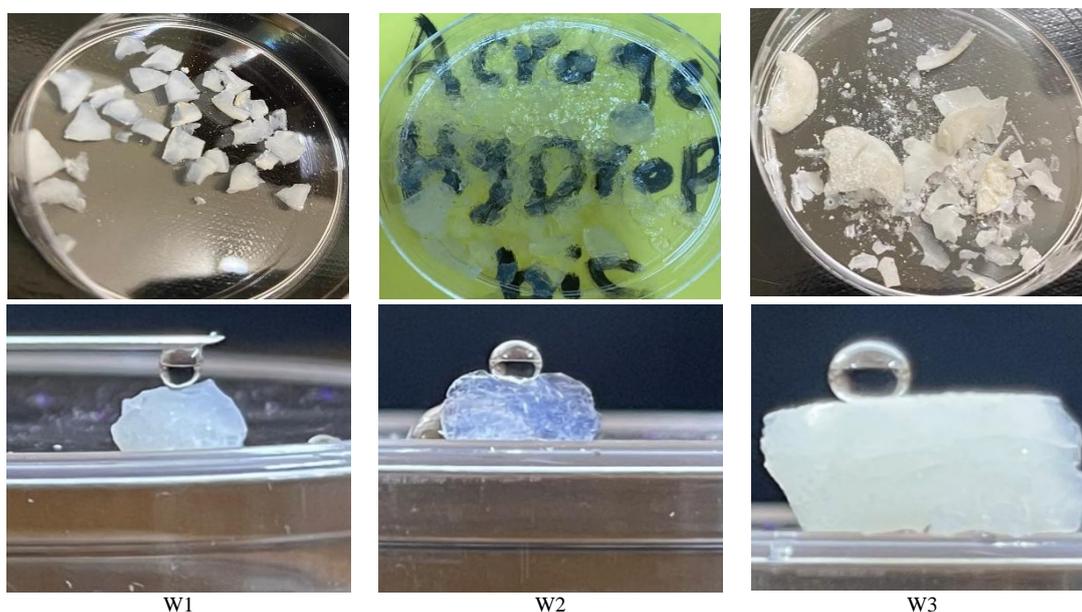


Figure 2. The photographs of superhydrophobic aerogels with high contact angles of 150.89 degrees XRD analysis

Fig. 3 shows the X-Ray Diffraction of samples (W1, W2 and W3) and the calculation of green size for each sample shown in Table 4. The difference between W1 and W3 is the addition of HCl to sodium silicate before it makes the ionic exchange. This means that the stimulation of cation resin plays an important role in making an amorphous sample similar to the silica aerogel from TEOS parcourse, as shown in sample W3, which indicates an amorphous structure with a broad band at 2θ between 22-34 degrees. From the XRD pattern, it can calculate the granule size depending on the Scherrer equation (Monshi *et al.*, 2012), for W1 and W2 which supported in value with FESEM particle calculation.

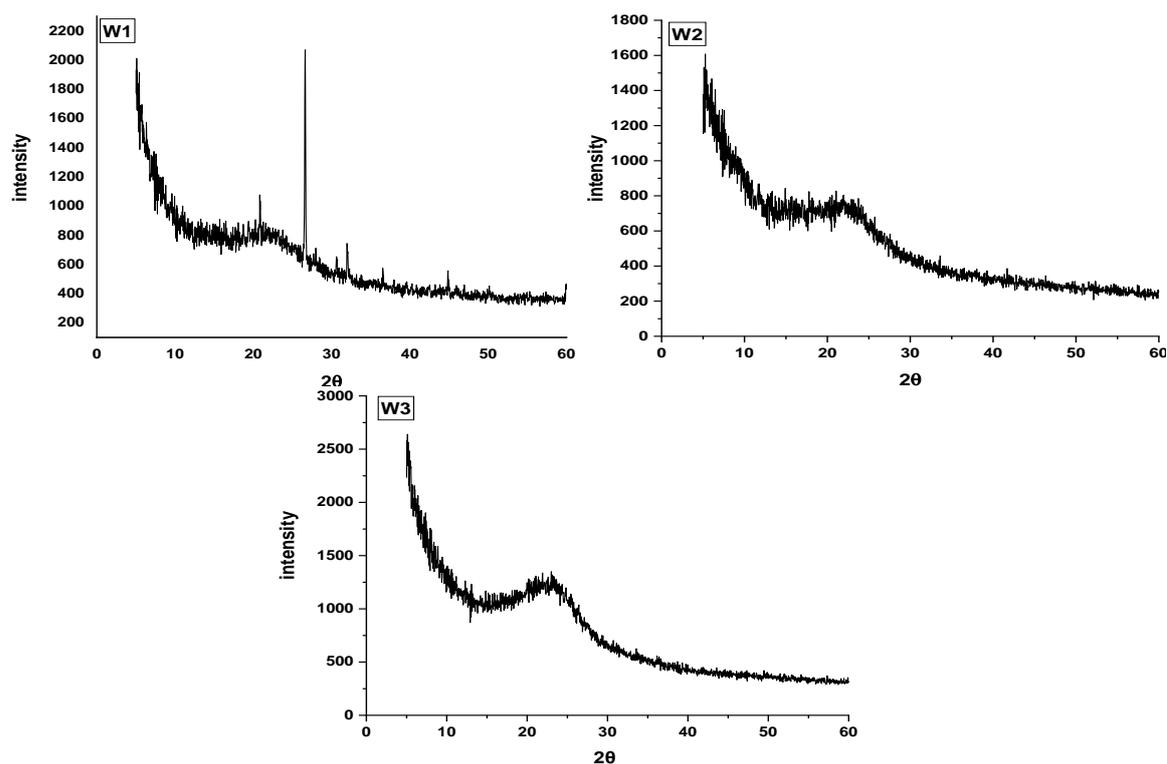


Figure 3. XRD pattern for silica aerogel (a) W1, (b) W2, and (c) W3

Table 4. Analysis of XRD from 2θ and the granule size for aerogel W1 and W3 samples

	2θ	Height	FWHML 2θ	Granule size
W1	20.82	196	0.06	23.49
	26.599	1195	0.11	12.95
	30.59	100	0.08	17.97
	31.97	221	0.08	18.08
	36.54	106	0.06	24.35
	39.50	40	0.1	14.72
	40.2	29	0.1	14.87
	42.5	29	0.2	7.435
	44.86	166	0.08	18.75
	45.95	46	0.1	15.06
	50.1	31	0.3	5.104
	59.96	99	0.11	14.55
W2	0.1767	8	111	22.9
	0.1769	8	56	23.0

BET analysis

Fig. 4 shows the linear isotherm plots for aerogel samples that were prepared under different methods. Clearly, meso- to macro-porous materials have a characteristic hysteresis loop that is associated with capillary condensation and evaporation in the meso-pores. From the figures, it can be noted that W1 sample is classified as an H1 type with well-defined cylindrical pore channels. It appears to have a very large value of surface area of $961.1 \text{ m}^2/\text{g}$. This result may be attributable to the low value of pore volume of $2.359 \text{ cm}^3/\text{g}$. It was also discovered that the surface area of the W2 sample

was $790.49\text{m}^2/\text{g}$ and the pore volume was $2.2404\text{ cm}^3/\text{g}$. in the case of the W2 sample, surface area is up to $790\text{ m}^2/\text{g}$, with the smallest pore volume. As well, it was observed that samples W2 & W3 can be classified as H2 type disordered pores (pore blocking) percolation phenomena, which are related to BET analysis of sample W2's high surface area (AL-Sharuee, 2021).

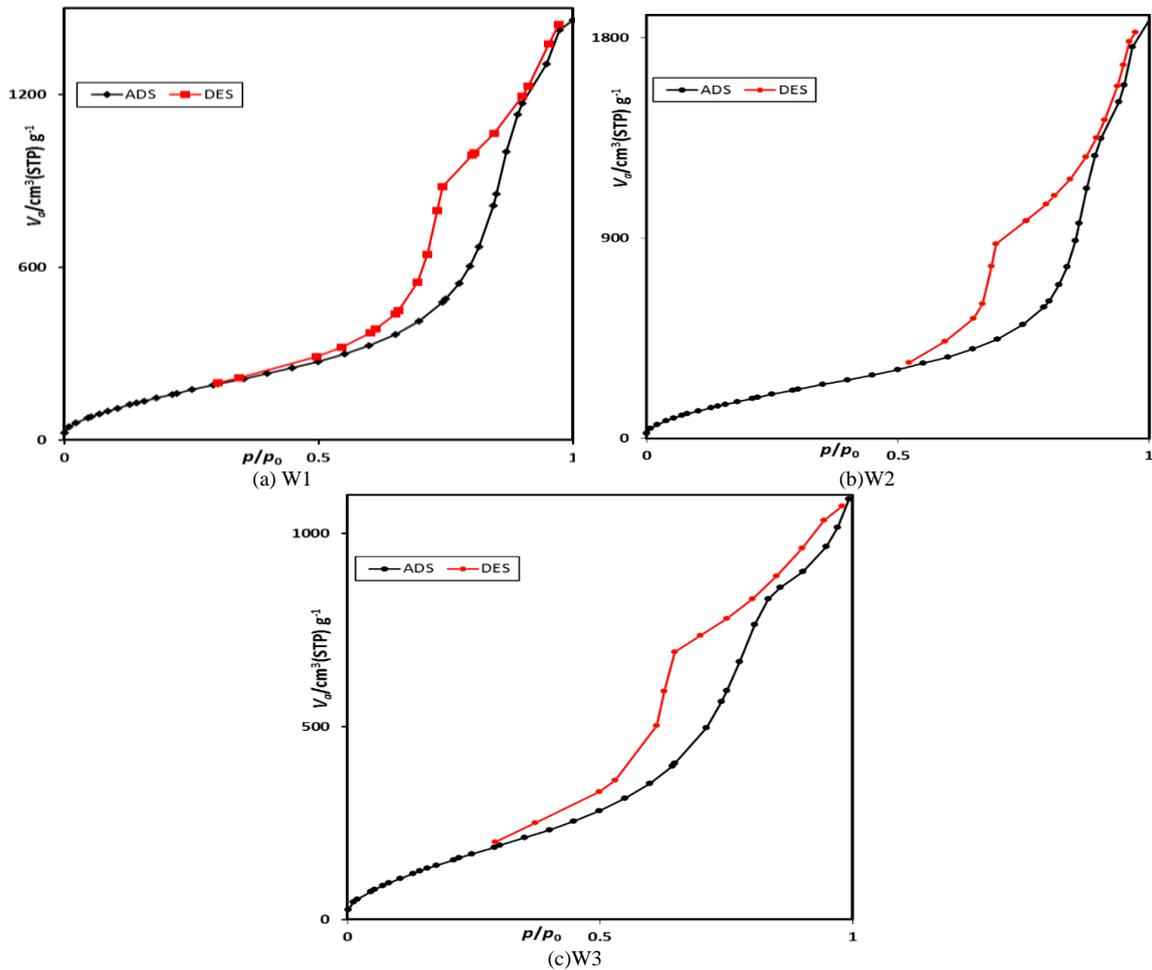


Figure 4. N₂ adsorption–desorption isotherms for the silica aerogel (a) W1, (b)W2 and (c)W3

FE-Scanning Electron Microscopy and EDS image

The surface structure of produced samples specified by FE-SEM images. And the description of materials analysis for silica aerogel for three different types of them shown in EDS images illustrated in Figs. 5,6 and 7. Clearly, the pore characteristics and structure morphology of TMCS modified silica aerogels visualized using FE-SEM micrographs, at two different magnifications. The sample modified using TMCS as sialylation agent had mesopore. The image of sample W1 showed the microstructure by field emission scanning electron microscope (FESEM). The silica aerogel appears a continuous and highly cross-linked network, and this, coupled with its smaller pores size, leads to a very open structure, where individual particles cannot be distinguished and the average particle of the sample $38,618\text{nm}$.

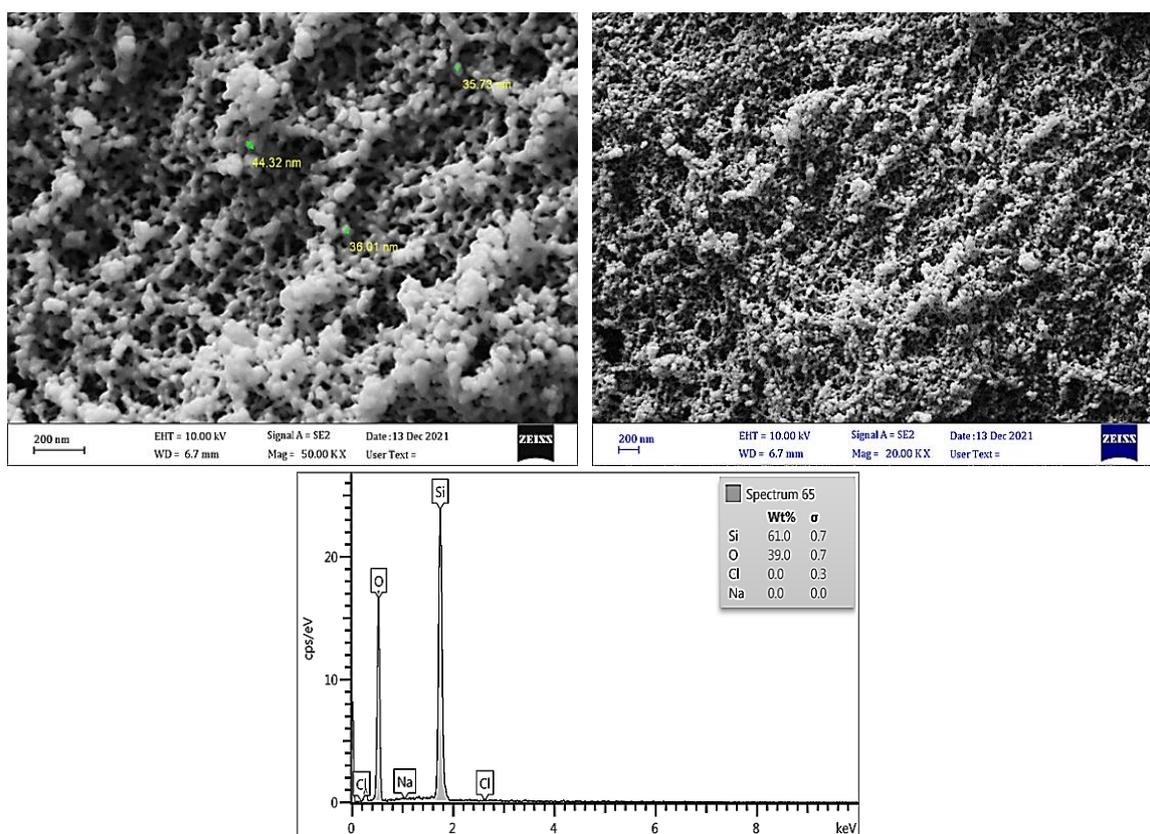


Figure 5. FESEM and EDS images for the W1 sample of silica aerogel

The image of sample W2 showed the microstructure by field emission scanning electron microscope (FESEM). The silica aerogel appears in clusters and when clustered together, the particles are aggregations too, and the surface shape is smooth. Moreover, there was microstructural inhomogeneity, and the average particle size of the sample was 43.23nm.

The image of W3 showed the microstructure by field emission scanning electron microscope (FESEM). The silica aerogel appears to have a fracture nature, which is composed of highly branched clusters of smaller particles. The image shows relatively large pores that rise out of the fracture surface. It can be found that the particles are free of aggregations too, and their surface shape is rough. Moreover, there was no microstructural inhomogeneity, and the average particle size of the sample was 39.49 nm.

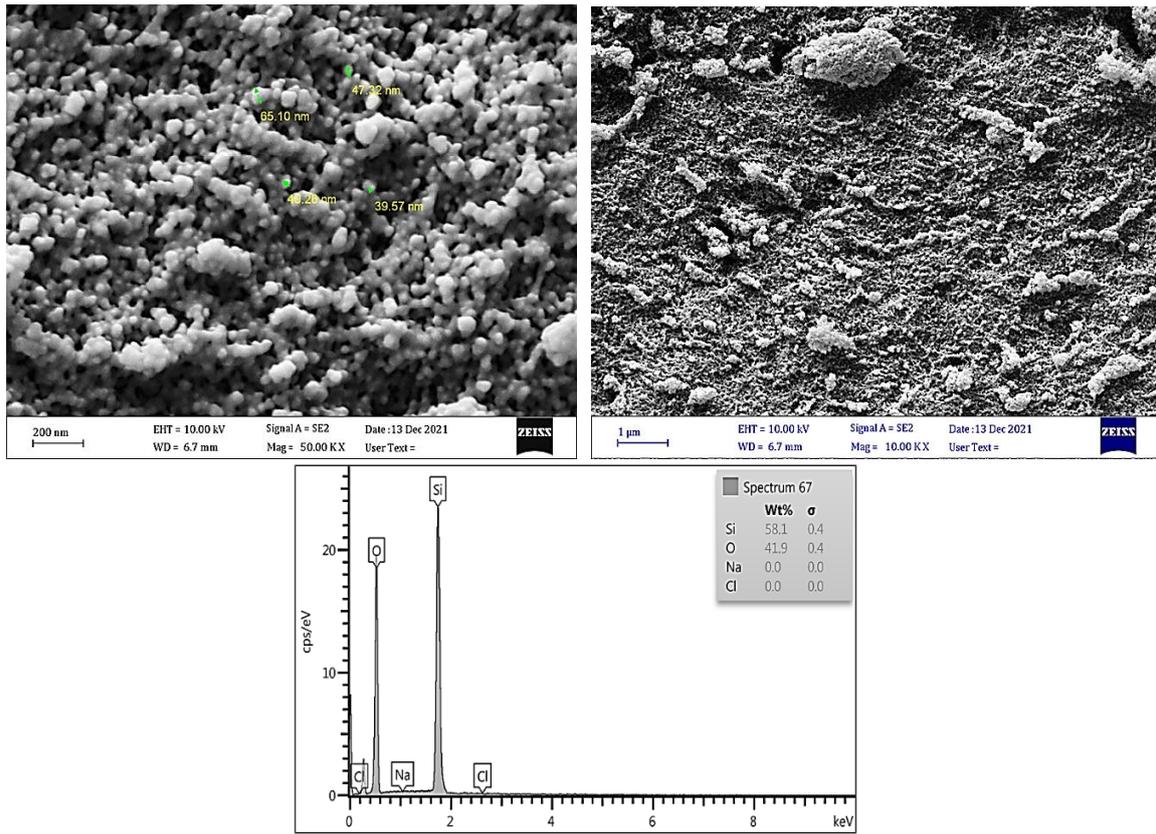


Figure 6. FESEM and EDS images for the W2 sample of silica aerogel

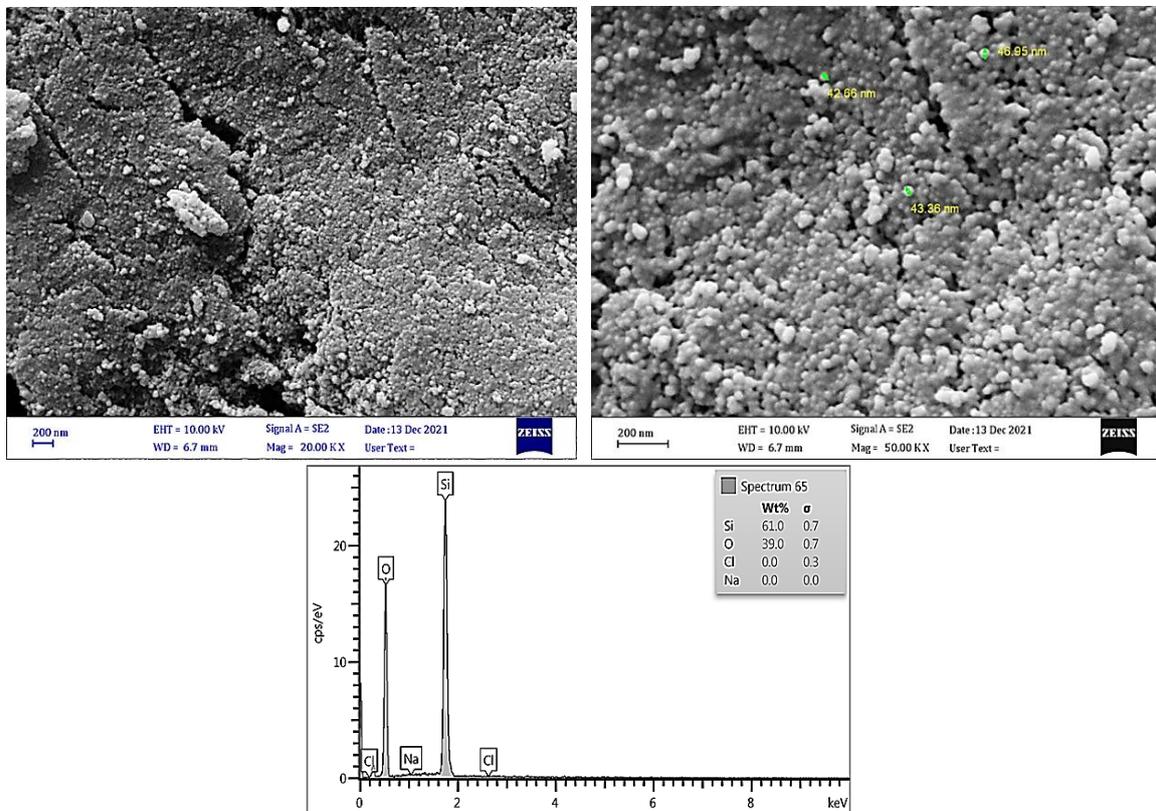


Figure 7. FESEM and EDS images for the W3 sample of silica aerogel

4. Conclusion

A superhydrophobic aerogel based on water glass is produced successfully with or without the activated acidic environment. The comparison with aerogel was made by using TEOS as a precursor, except for the gel time. The work gives a good specification of low density, high surface area, small particle size, and amorphous surface regardless of gel time. Generally, the activity resin in HCl awards the best structural and morphological properties if it doesn't activate the resin in it. The addition of HCl in the solution is critical for improving the pore structure of silica aerogel in this study, where the addition of it in resin leads to reducing the water content in the wet gel and giving the produced aerogel the largest particle size, which is smoother and more uniform.

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