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INSTITUTE OF ENGINEERING
PULCHOWK CAMPUS**

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Synthesis of Silica Aerogel with and without Glass Wool

by

Sunny Kumar Sah

A THESIS

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ABSTRACT

Aerogel is one of the most researched and versatile products in the current era. Since its invention, silica is mostly used for making aerogel by using three major processes called supercritical drying, ambient pressure, & freeze-drying process. So, this project illustrated the making of silica aerogel from laboratory reagent-type sodium silicate (commercial grade) available in the Nepali market and glass wool was utilized for reinforcing the structure.

In this project, the work is focused on the simple method called the ambient drying process to synthesize the silica aerogel with and without glass wool. The silica gel samples were prepared from sodium silicate through the sol-gel process with and without glass wool. The gel samples were passed through the solvent exchange process using ethanol and then modified using 4:30:1 & 20:30:10 ratios of TMCS (Trimethyl Monochlorosilane): n-hexane; ethanol which gave two types of samples called Type-1 & Type-2 respectively. The modified samples were then dried at ambient temperature to get the final product. From the characterization, it was revealed that all Type-2 samples and only S1, S2, S3 & A1 of Type-1 showed the presence of Si-C bond to confirm the hydrophobic nature. Further, the contact angle of 146.7° & adsorption of organic oil was higher for the Type-2 sample. But the mechanical test was not feasible for the sample with glass wools due to cracks and improper binding between silica & glass wool. Hence, the sample made from commercially available sodium silicate resembled the properties of the silica aerogel but did not fully fulfil the criteria due to impurities in the source chemical.

Keywords: Ambient Pressure Drying; Silica Aerogel; TMCS; Glass Wool; Organic Oil Adsorption

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ABBREVIATIONS

XRD	: X-ray Diffractometer
TG-DTA	: Thermogravimetric and Differential Thermal Analyzer
FT-IR	: Fourier Transform Infrared Spectrometer
FE-SEM	: Field Emission Scanning Electron Microscope
BET	: Brunauer-Emmett-Teller
UV-Vis	: Ultraviolet-Visible Spectrophotometer
AAS	: Atomic Absorption Spectroscopy
TMCS	: Trimethyl Monochlorosilane
EtOH	: Ethanol
HMDS	: Hexamethyldisilazane
MTES	: Methyltriethoxysilane
APD	: Ambient Pressure Dying
TMOS	: Tetramethylorthosilicate
TEOS	: Tetraethylorthosilicate
NASA	: National Aeronautics and Space Administration

CHAPTER 1: INTRODUCTION

1.1 Background

Aerogel is a kind of foam that can be found in various forms and shapes. Aerogel does not have a specific chemical formula to be considered as specific material or mineral but it comprehends the material to give a specific geometrical structure. The structure of the aerogel is highly porous with a solid foam appearance. Till now the majority of aerogel is made of silica but some are also made of iron oxide, gold, carbon, organic polymers, copper, and semiconductor nanostructure. The aerogel exhibits unique composition with 99.8% of air and very little amount of solid due to which it appears like frozen smoke (Thomas, 2012). The aerogel prepared by NASA (National Aeronautics and Space Administration) is shown below.

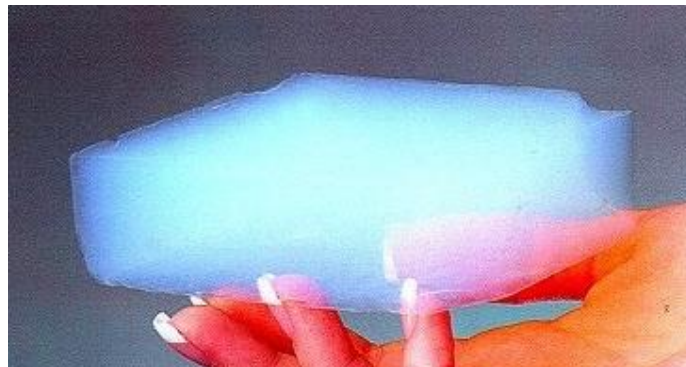


Figure 1.1: Aerogel prepared (Thomas, 2012)

Aerogel is famous for its lightweight due to which it is recorded as the lightest solid in the Guinness Book of the world record (Thomas, 2012). The density of the aerogel varies from 1.1×10^{-3} to 0.50 g/cm^3 and it is typically around $2 \times 10^{-2} \text{ g/cm}^3$. In general, aerogel is 15 times heavier than air but it is also found to be lighter i.e., only three times heavier than air. Later, the metallic micro lattice was recorded as a lighter solid than silica aerogel which was further outcasted by aero graphite. But silica aerogel still possesses the most desirable properties such as the best insulator with low thermal conductivity, high specific surface area, lower dielectric constant, refractive index, and sound speed.

In 1931, Steven S. Kistler prepared the first aerogel to prove that aerogel possessed a network of solids in a continuous manner having the same shape and size as contained by the hydrogel. To prove this hypothesis, it was necessary to remove the liquid from the hydrogel without breaking the solid structure. The failure of the structure occurred due to the shrinkage when the gel was allowed to dry. The theory was given that when a liquid is stored at extreme pressure

higher than the vapour pressure then the temperature is increased and the liquid will convert into a gas without the presence of two phases condition at that moment. The first aerogel was made by condensing the aqueous sodium silicate in an acidic medium. In the first attempt, Kistler was not able to convert the water present in the hydrogel into supercritical fluid because supercritical water was redissolving in the silica instead of leaving it and silica became precipitated when the water was finally removed. Then, he tried again in another way in which he first removed the salt present in the silica gel by washing it with water and then exchanged the water present in the gel with alcohol. By doing this, he was able to convert the alcohol into supercritical fluid which allowed it to escape the gel without shrinkage and the first silica aerogel was made (Kistler, 1931).

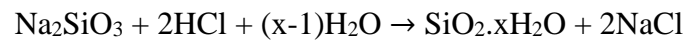
Later, in 1970, Stanislaus Teichner was working on the French government project of rocket fuel and oxygen in porous materials. With his assistance, he adopted the Kistler method for this project but sooner it was realized that Kistler's method was time-consuming and took weeks to prepare one aerogel sample. As they had to make many samples for the research, he came up with the idea of replacing the sodium silicate with tetramethylorthosilicate (TMOS) called alkoxysilane. By using this as a precursor, the two major drawbacks of Kistler's methods were overcome; the step of solvent exchange (water to alcohol) and inorganic salt presence. Based on the TMOS, the pilot plant for making silica aerogel was established in Sjobo, Sweden by the Lund group. After that at Berkley lab, TMOS was replaced by tetraethylorthosilicate (TEOS) in 1983 by Arlon Hunt and Microstructure Material Group because it was toxic to work with and they also introduced the supercritical drying process for replacing liquid from the hydrogel using liquid carbon dioxide. Since then, much research was done to make aerogel by using different sources, including low-cost aerogel, organic polymer aerogel, etc. (Hunt & Ayers, 1983).

1.2 Sol-Gel Method

Most aerogels are made from gels based the sol-gel chemistry. Sol-gel is the method in which nanoparticles of solid are distributed together in the liquid to create a 3D network of continuous type all over the liquid. In this sol-gel condition, the two phases are mixed at the nano-level i.e., solid and liquid but in aerogel, the solid and gas are mixed intimately. Both conditions are termed colloids which is the homogenous solution. In the sols, the liquid is in the continuous phase whereas the solid is in the dispersed phase. Here, the gel is considered as a solid of wet type material which contains an organized solid network of nanostructures elongated in the

liquid volume and in this, the solid network is in the continuous phase & liquid is in the dispersed phase. Similarly, aerogel also comes under the colloid material which has a continuous solid structure with air pockets through the solid. The factor affecting sol-gel chemistry are; pH, temperature, solvent, duration, catalysts, & agitation (Steiner, 2022).

The polymerization process occurs in the sol-gel method where the network of the nanostructure solid is developed in the liquid medium which produces a siloxane bridge ($\equiv\text{Si}-\text{O}-\text{Si}\equiv$) when a silica source like water glass is used. In the initial stage, the linear oligomers are developed which is called sol due to silica particles that are present in colloid liquid as dispersed form. In the next step, all linear oligomer links with each other to form the three-dimensional network in the liquid which is called gel. Such as the chemical reaction during the sol-gel process between water glass and HCl is presented below (Pierre & Rigacci, 2011):



1.3 Solvent Exchange Method

The solvent exchange process is very important in decreasing the shrinkage of the gel structure during the drying process. Here, the water has high surface tension and if the gel is kept for drying then the water molecules pull the silica 3D structure inwards due to higher intermolecular interaction during evaporation. Because of this phenomenon, the gel structure gets collapsed. To prevent this, a solvent like water is replaced from the gel using lower surface tension organic solvents like ethanol, methanol, etc. (Lebedev et al., 2021).

1.4 Drying Processes

There are three popular methods for performing the drying of sol-gel which are explained below:

1.4.1 Freeze Drying

In this process, the solvent present in the pores of the gel is frozen first and then evaporated under the vacuum condition. During this process, the frozen solvent has directly transformed the state from solid to vapour without going through the liquid phase which prevented the collapsing of the structure due to surface tension. The first drying is done at low pressure in a vacuum chamber after freezing the solvent in the sample and this step helps to remove the excess percentage of solvent from the sample. Next, the remaining solvent molecules which are bonded to the sample are removed by applying heat (Hilgedick, 2020).

The final product obtained from freeze-drying is referred to as cryogel (Maleki et al., 2014). When the silica-based aerogel is made then the solvent in the pores can cause a crack in the structure due to crystallization of the solvent. So, to prevent this, the low surface tension solvent is preferred having high pressure for sublimation. Further, liquid nitrogen is adopted for rapid freezing of the solvent.

1.4.2 Supercritical Drying

The drying of the solvent in the pores of the gel is replaced with gas above its supercritical states i.e., above critical pressure and temperature. This process prevents the solvent not to go through the liquid-vapour interface due to which the rise in capillary pressure gradient is eliminated. This process can be carried out in two ways; the first is called the hot process in which organic solvents like ethanol are used having a supercritical temperature of 241°C at 62.7 bar. In the second process, the solvent is replaced by supercritical gas called carbon dioxide at the temperature above the supercritical temperature of the carbon dioxide i.e., 31°C at 73.8 bar and this process is called the cold process. Among the hot and cold processes, the cold process is safe as it used non-flammable carbon dioxide as compared to the hot process.

1.4.3 Ambient Drying Process

This process is an easier method of drying the gel to make aerogel as compared to the previous two methods. The gel is dried at ambient pressure and ambient temperature due to which no high-pressure chamber is required in this process. The possibility of utilizing aerogel for thermal insulation is increased by this process where aerogel prepared from the supercritical drying method is hard to use (Jones & Paik, 2013). Most thermoelectrical devices use aerogel made from ambient pressure drying. This process also facilitates to use of plastic and metal alloy in the aerogel because the drying process is carried in the ambient pressure and temperature which does not affect the metal and plastic but this is impossible in a supercritical process.

1.5 Problem Statement

Oil has become an essential source for running the world as it's used in the vehicle as a fuel to run, lubricate machinery parts, make paints, plastics, fertilizer, and so on. With the increase in population, energy demands are also increasing and around 33% of the energy is still fulfilled by oil in the world (Rapier, 2020). The oil made the possibility in the evaluation of locomotives like vehicles, planes, trains, ships etc. along with other types of machinery but it also causes

the environmental problem like air pollution due to the emission, as well as land & water pollution due to dumping of the used oil. Here, the oil and water do not mix as the oil is non-polar and water is polar in nature and due to this, oil makes a thin layer on top of the water. So, to remove this oil from the water there are many methods that are currently used like mechanical recovery, non-mechanical recovery, and manual recovery. Further, the porous material can be also used to remove the oil from water. There is the advantage of using porous material because it does not mix with water due to hydrophobicity and adsorbs the oil due to oleophobicity. There are three types of porous materials: an inorganic mineral which includes silica aerogel, exfoliated graphite, organoclays, petite, etc.; synthetic organic which includes polyurethane & polypropylene; last is organic porous material under which rice straw, mildew, cotton, etc. (Olalekan et al., 2014).

The work is focused on making the silica aerogel using sodium silicate (commercial grade) under an ambient drying process (APD) for making highly porous aerogel that can adsorb the organic solvents or oil in high amounts to solve water pollution.

1.6 Objectives

The main focus of the research is explained in two sections; main and specific objectives as follows:

1.6.1 Main Objective

The key aim of the work is synthesizing the silica aerogel using sodium silicate as a precursor (i.e., commercially available water glass) under the APD technique.

1.6.2 Specific Objectives

- Preparing silica aerogel with and without glass wool.
- Identifying the presence of the needed group responsible for amorphous property and studying the surface texture.
- Estimating the organic solvent or liquid adsorption rate by the sample and the time required to evaporate it.
- Understanding the electrical property of the sample based on the bandgap energy.
- Ensuring higher resisting strength in glass wool sample as compared to pure aerogel.

1.7 Limitations

- Most of the testing machines are not available in Nepal so Thermogravimetric and Differential Thermal Analyzer (TG-DTA) & Brunauer-Emmett-Teller (BET) are not conducted on the prepared samples.
- The pH measurement of the sol-gel solution just before gelation is found difficult as solidification occurred instantly after adding the acid.

1.8 Report Outline

The report consists of the following chapters:

Chapter 1: Introduction- This section explains the background of aerogel, related terminologies, problem statements, and the objective of the research.

Chapter 2: Literature Review- This section deals with published papers related to the present research.

Chapter 3: Chemicals and Methods- This section explains the selection of the chemicals, apparatus, and testing equipment. Further, it covers the process of making aerogel samples and the procedure for performing the needed tests.

Chapter 4: Result and Discussion- The interpretation of the obtained results of the samples with supporting figures and data are presented in this chapter.

Chapter 5: Conclusion and Recommendation- This section includes the summary of the research as per the results and states the needed recommendations for future work.

CHAPTER 2: LITERATURE REVIEW

The papers were gathered and studied to understand the synthesis of aerogel, reinforcement with different fibres, and use for removing the organic solvents or oils.

2.1 Aerogel Synthesis

Khedkar et al., (2019) synthesized superhydrophobic silica aerogel via an ambient drying process. The sodium silicate was used as a precursor along with hexane, amberlite ion exchange resin, methanol, ammonium hydroxide, and trimethylchlorosilane (TMCS). Here, the TMCS was used to modify the surface of the hydrogel to prevent shrinkage during the ambient drying process which was done in many steps. The authors performed the characterization of obtained aerogel with the help of various methods like X-ray Diffractometer (XRD), Fourier Transform Infrared Spectrometer (FT-IR), TGA-DTA, & Ultraviolet-Visible (UV-Vis) spectrophotometry tests. The thermal stability was noted to be below 478°C and changed to hydrophilic after 478°C due to oxidation of -CH₃. The surface area was noted to be 792.308 m²/g which was very large with average pores size of 5.779 nm obtained from BET. Likewise, the superhydrophobic nature was stated due to the 151° contact angle and the insulating nature was confirmed by the UV-Vis spectrophotometer due to the higher bandgap energy of 4.25eV.

Gurav et al., (2009) examined the physical properties of the aerogel made from sodium silicate by following ambient pressure drying. The chemicals used for making aerogel were sodium silicate, tartaric acid, methanol, and TMCS. The authors illustrated the new method of removing Na⁺ ions from hydrogel by bypassing the water vapour on it which replaced the ion-exchanged method that took several hours to complete. By using the water vapour passing method, it was confirmed by Atomic Absorption Spectroscopy (AAS) that 95% of Na⁺ was removed. In this way, the other physical properties like porosity, contact angle, pore volume, etc. were also examined by changing parameters like gel aging time, tartaric/Na₂SiO₃ molar ratio, & TMCS/Na₂SiO₃ molar ratio. FT-IR explained the presence of more silylation in aerogel when vaporization was done for 0.5h and pointed to the hydrophobic nature due to the presence of Si-CH₃. Further, hydrophobicity was maintained up to 473°C confirmed by TG-DTA.

Lee et al., (2021) prepared the powder of silica aerogel by ambient pressure drying with thermal gelation. The chemical used in this experiment were water glass (sodium silicate), acetic acid, ethanol, n-hexane, & HMDS. In this process, the hydrogel was prepared at 60°C higher than room temperature which came under thermal gelation. Hexamethyldisilazane (HMDS) was

used for surface modification which was done for three times and aerogel powder was obtained by drying a hydrogel sphere at ambient pressure. The pH value played an important role in the aerogel power of highly porous which was varied with the concentration of acetic acid and water glass. The silica acid solution with 4.5 pH showed low thermal conductivity but with higher pore volume and specific surface area examined by BET. The photograph of the water droplet was taken on the powder and measured the contact angle of 147° for 6.6% water glass solution

2.2 Aerogel Reinforcement

Shao et al., (2015) made the composite silica aerogel reinforced with silica fibre. The necessary chemicals used were sodium silicate, TMCS, methyltriethoxysilane (MTES), ethanol, ammonia, n-hexane, and 732 cation exchange resin. The synthesis was based on APD and surface modification was done in two steps; first with MTES and then with TMCS. The silica fibre was added to the aerogel from 3.5 to 17.5 wt% which provided density from 0.108 to 0.146 g/cm³ along with porosity variation from 93.4 to 95.1% and thermal conductivity was found between 20 to 30 mW/(mK). The average modulus was achieved up to 535 kPa for aerogel with 17.5 wt% silica fibre.

Huang et al., (2019) explored the fast way to synthesize the silica aerogel blanket reinforced with glass fibre. The authors utilized sodium silicate as a precursor of silica and the process was altered during sol-gel preparation in which ion exchange & rotary evaporation were carried out followed by the addition of ethanol & base catalyst (ammonium hydroxide). From the analysis, the aerogel with glass fibre showed a decrease in bulk density when ethanol was raised from 38.7 to 51.3% but was later found to be increasing with an increase in ethanol. But the variation of the porosity was opposite to that of bulk density. In conclusion, 45.53% weight was achieved for aerogel reinforced with glass fibre when ethanol was maintained at 51.3%. From the 3-point loading test on the sample, 12 MPa as a flexure strength were noted. The lower thermal conductivity was found to be 0.026 W/mk.

Slosarczyk, (2021) reinforced the silica aerogel with carbon fibre (0% to 15% by vol.) using water glass by APD method. Water glass, carbon fibre (dia 13 μm & length 700 μm), nitric acid, methanol, n-hexane, & TMCS. The unique work was the modification of carbon fibre in nitric acid for increasing the amount of oxygen functional group on the surface and to provide a large number of connection facilities between carbon fibre with silica structure. After characterization, the carbon fibre-reinforced silica aerogel was thermally stable up to 400°C.

From the experiment, it was observed that after increasing the carbon fibre above 10% by volume, the surface modification with n-hexane/TMCS was lowered by 24 hours. The mechanical test suggested that mechanical properties like compressive strength of 0.054 ± 0.012 MPa were better for 10% but electric conductivity was three-time higher for 15% vol.

Salimian & Zadhoush, (2019) inspected the feasibility of using silica aerogel as a filler in polymer nanocomposite. In this, the epoxy resin called Diglycidyl of bisphenol A was used and silica gel was prepared with water glass based on APD. Here, the silica aerogel was mixed by wt% as 1, 2, and 4% with epoxy resin to make epoxy nanocomposite. From the N₂ adsorption isotherm, it was found that the specific area was 810 m²/g, pore volume was 3 cm³/g, and pore size was 10 nm. Moreover, the hydrophobic nature was established by a higher water contact angle of 150°. Likewise, the storage modulus also increased with silica content and aerogel changed from a glassy state to rubber at 130° under DMA. Moreover, the flexural modulus was improved to 40% and strength to 80% for 4% of silica aerogel due to the homogenous distribution of filler in the matrix.

Motahari & Abol, (2015) prepared the silica aerogel to use as the fire shield for the steel frame structure after reinforcing the aerogel with glass fibre composite. In this method, the glass fibre matric was immersed in the aqueous solution of sodium silicate and HCl and followed the APD process. Here, the 8 samples were prepared under two pH and found aging periods. To check the fire resistance, 4mm thick glass fibre silica aerogel was used to wrap the 5mm thick steel plate. From the result, the high resistance was shown by the sample made at 4 pH rather than 8 pH. Further, the sample made at 4 & 8 pH and aged for 12 hours showed higher resistance. And the fire resistance ability of the sample was increased with an increase in porosity. In conclusion, the silica aerogel with glass fibre composite showed greater fibre resistance when made at 4 pH.

2.3 Oil or Organic Solvent Removal

Wang et al., (2012) utilized silica aerogel for the removal of the oil from the oil-water emulsion and pure liquid. The experiment was conducted on crude, motor, & vegetable cooking oil to examine the sorption kinetics as well as the equilibrium. Here, it was found that oil from oil-water emulsion was 5 to 10 times slower as compared to the adsorption of organic solvent from an aqueous solution. When the surfactant proportion was increased to make the emulsion steady, the capacity of adsorption by the aerogel was noted to be declined. Further, the

adsorption rate and capacity of the aerogel were found to be low for the jet fuel because of the greater stability of the emulsion of wastewater oil.

Manzocco et al., (2021) explained the use of aerogel in the food industry. It could be used as an ingredient having low calories because it contained most of the air in it and also be helpful to regulate the release of nutrients & control feeding. Further, the aerogel might be useful in the delivery system for loading the compound during the preparation of the aerogel. This could be done before the drying process which was called wet impregnation and if the loading was done after making aerogel, then the process came under post-drying impregnation. The aerogel showed a high porosity property based on which it could be used to replace the fat. Because of this, the oleogel could be prepared to act as a fat substitute to replace the fatty acid from the food to make it healthier. Likewise, aerogel could be used in food packaging due to its lightweight, proper thermal insulation, & mechanical strength with reinforcement.

Hrubesh et al., (2001) carried out the removal of the solvent from the water by using an aerogel. The solvents like chlorobenzene, trichloroethylene, ethyl alcohol and toluene were used for the experiment. The adsorption of the aerogel was compared with standard granulated activated carbon. From the analysis, it was noted that the adsorption capacity of the aerogel was 30 times higher than granulated activated carbon for soluble solvents and 130 times for insoluble solvents.

Perdigoto et al., (2012) tested the removal of the toxic compounds by using silica xerogel & aerogel from the aqueous liquid. Using the Langmuir model, the highest adsorption of the benzene was noted to be 192.31 mg/g of aerogel but a further higher capacity was recorded for the toluene. The aerogel & xerogel was able to remove the toluene and benzene in greater amount, Further, the ozone oxidation of the aerogel was done which showed the regenerated property however the hydrophobicity was lost.

Yi et al., (2019) prepared the silica aerogel i.e., SiO₂ (AG) under the APD method which had a maximum thermal stability temperature of up to 500. The aerogel had microporous along with high surface area and pore size so the adsorption test was performed on methylene blue, phenol, and nitrobenzene. The adsorption was conducted with hydrophobic and hydrophilic SiO₂ and found that hydrophobic was effective to remove slightly soluble organic solvent whereas hydrophilic was better in removing the highly soluble organic solvent. Further, both types of SiO₂(AG) removed more amount of the MB than phenol.

2.4 Research Gap

Most of the researchers have used the precursor sodium silicate (Na_2SiO_3) having a weight ratio of $\text{Na}_2\text{O} : \text{SiO}_2$ as 1:3.3. Other researchers such as Gurav et al., (2009) & Lee et al., (2021) have also used sodium silicate in which SiO_2 content is 3 times higher than Na_2O by weight to synthesize silica aerogel. Now this reveals that the precursor needs to have more silica (SiO_2) to make efficient aerogel. So, the focus of this research is to utilize the silica source available in the Nepali market. As per the market survey, the available silica source to make the aerogel in Nepal is commercial water glass (commercial grade) which contains the solution of sodium silicate manufactured by NIKE Fine Chemical, India. Here, the plan is to utilize the source and add the commercial glass wool during preparation for making the final aerogel sample with a stable structure.

CHAPTER 3: CHEMICALS AND METHODS

3.1 Chemicals

A. Sodium Silicate

The sodium silicate was purchased from the local market which was in the form of a viscous liquid often called water glass in the market. The manufacturer of this chemical was NIKE Fine Chemical, India. The specifications on the packet were not provided as it is used in the form of laboratory reagent (commercial) and claimed as extra pure.

B. Hydrochloric Acid (HCl)

The 35-37% assay (acidimetric) HCl was adopted having a molarity of 12 concentration.

C. Ethanol (EtOH)

The analytical reagent ethanol was selected having assay (by vol.) 99.9% made by Changsh Hongsheng Fine Chemical Co.Ltd.

D. n-Hexane

The selected n-hexane chemical was the product of LOBA Chemie Pvt. Ltd. and had 86.18 molecular weight with 99% assay by CG.

E. TMCS

The trimethyl monochlorosilane was imported from Hebei Guanlang Biotechnology Co. Ltd. which was a colourless liquid with a purity of 99.53%.

F. Benzene

The benzene was used as an organic solvent for the adsorption test which was 99% minimum assay and 78.11 molecular weight.

G. Edible Oil

The Sunflower refined oil was adopted also for the adsorption test.

H. Glass Wool

The available glass wool in the local market was used for this project to the reinforcement of the indented silica aerogel.

3.2 Instruments & Apparatus

The list of instruments and apparatus are present for making the samples as well as for testing as follows:

A. Magnetic Stirrer

The aqueous solution was mixed by solving the water glass in the distilled water with the help of a magnetic stirrer.

B. HotBox Oven

The hotbox oven was used for drying the sample to remove the moisture and other chemicals. Further, the oven was also used to maintain the required temperature of the sample at a certain stage.

C. Digital Weighing Machine

The weight of chemicals and samples was measured using a digital weighing machine having a maximum weight capacity of 210g

D. UV-Vis Spectrophotometer

The UV-Vis spectrophotometer model UV2600i was selected which was manufactured by Shimadzu to measure the transmittance of the sample under the visible light spectrum.

E. FTIR Spectrophotometer

FTIR spectrophotometer model IRPrestige-21 was used to measure the transmittance under the infrared spectrum of the sample. This instrument was available at Kathmandu University and manufactured by Shimadzu.

F. Contact Angle Tester

The contact angle was measured by using the instrument available in the Physics department of the KU.

G. XRD

The XRD analysis was planned to evaluate whether the sample is crystalline or amorphous in nature.

3.3 Method

The samples are made in two categories based on modification solution ratio i.e., 4:30:1 (let's say Type-1 sample) and 20:30:10 by volume of TMCS: n-Hexane: Ethanol (let's say Type-2 sample). And the sample with glass wool was made only for Type-2.

3.3.1 Diluting HCl

The molarity concentration of the 37% assay was calculated and found to be 12M. After that, the 1 M HCl solution was made by diluting conc. 8.33 mL with distilled water to make a final volume of 250 and calculation details are presented in APPENDIX.

3.3.2 Aqueous Solution

The first step was to make an aqueous solution of the sodium silicate or water glass with distilled water in 1:1 (w/v). So, 10 g of viscous water glass was measured in the beaker using the weighing machine. Then, the 10 mL of distilled water was measured in a measuring cylinder. The 10 mL distilled water was poured into the beaker containing sodium silicate and then, a magnetic stirrer was placed into the beaker. After that, the beaker was placed on the magnetic stirrer machine and let the solution to mix until the sodium silicate was dissolved. The mixing was done at an ambient temperature of around 5 minutes to obtain the final sodium aqueous solution of the sodium silicate.

3.3.3 Making Sol-gel

3.3.3.1 Without Glass Wool

The 26 mL of the 1 M HCl was mixed with an aqueous solution of sodium silicate in a beaker. After that, the solution was swirled with a hand and quickly poured into a plastic mould for gelation. The solution was left for a certain time in the mould and gelation was completed around 2 minutes. After 2 minutes, the gel of silica was obtained and extra unreacted HCl was drained from the mould.

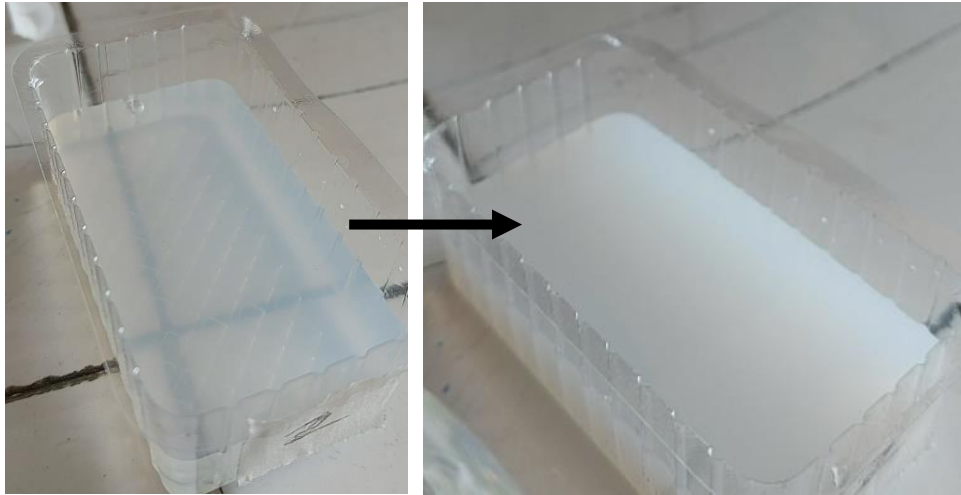


Figure 3.1: Gelation without glass wool

3.3.3.2 With Glass Wool

At this first, the 1 g & 1.5 g of glass wool was measured and then spread in the mould properly. Next, the 26 mL of 1 M HCl was mixed with an aqueous solution of sodium silicate in a beaker. After swirling, the solution was poured into the mould having glass wool. Next, the solution was let for gelation and the unreacted solution was drained from the mould.



Figure 3.2: Glass wool used for reinforcement

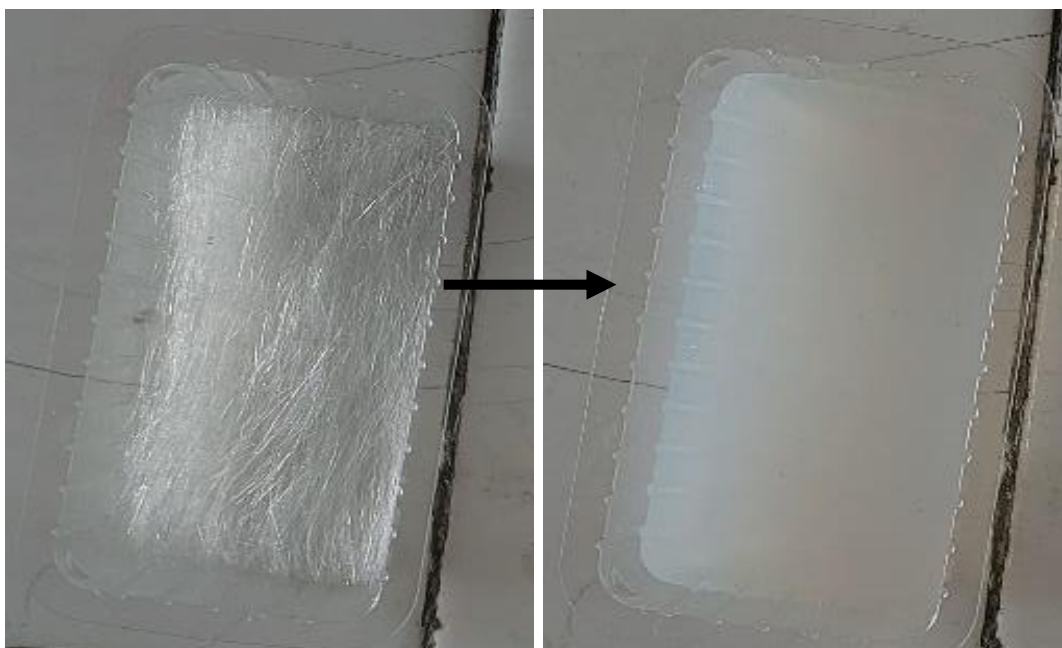


Figure 3.3: Gelation with glass wool

3.3.4 Strengthening & Sodium Salt Removal

The gelation process gave a 3D silica network silica gel of dimension $7.3\text{cm} \times 3.7\text{cm} \times 1.2\text{cm}$ which was then emerged into the distilled water for strengthening the silica network for 24 hours. During gelation, HCl reacted with sodium silicate because of that NaCl and 3D silica networks were formed. It was necessary to remove the salt from the silica gel. For that, the distilled water was changed two times a day for three days to remove salt after completing the strengthening of the silica gel.

3.3.5 Solvent Exchange

In this step, the major task was to replace the -OH of water with the -OH group of the alcohol. To do so, ethanol was used for replacing the -OH group of the water. After removing the sodium salt, the silica gel was submerged in the ethanol.

3.3.5.1 For Type-1 Sample

The replacement of the ethanol was done in 4 cycles within 24 hrs during the solvent exchange.

- 1st Cycle = The sample was submerged in ethanol for 2 hours at room temperature.
- 2nd Cycle = After 2 hours, the ethanol was changed and again, left for 2 hours.
- 3rd Cycle = Then, the ethanol was again changed and left for 18 hours overnight.
- 4th Cycle = Next day, the ethanol was replaced and left for 2 hours.

3.3.5.2 For Type-2 Sample

The replacement of ethanol was for two days in 4 cycles for solvent exchange.

- 1st Cycle = The sample was submerged in ethanol and left for 24 hours.
- 2nd Cycle = Next day, the ethanol was replaced and the same sample with ethanol was heated for 1 hour at 50°C.
- 3rd Cycle = The previous batch of ethanol was again changed and left for 24 hours at ambient temperature.
- 4th Cycle = At last, the silica gel was washed with ethanol to complete this exchange process.

3.3.6 Modification

The next step was to replace the ethanol -OH group which -CH₃ and this was accomplished by using TMCS. The modification was done with TMCS, n-hexane, & ethanol in the ratios 4:30:1 & 20:30:10 (by volume mL).

3.3.6.1 Modification of Type-1 Sample

The modification of the sample was done only one time with TMCS, n-hexane, & ethanol in the ratio of 4:30:1 by volume. Here, the solution was prepared by mixing the TMCS, n-hexane, & ethanol as per mentioned ratio. Then, the sample from the solvent exchange method was submerged in the solution for different periods in air tight container as mentioned in the table.

Table 3.1: Modification time for Type-1 samples

Group	S Group				A Group		
Sample	S1	S2	S3	S4	A1	A2	A3
Time	48 hrs	36 hrs	24 hrs	12 hrs	24 hrs	12 hrs	6 hrs

3.3.6.2 Modification of Type-2 Sample

The process was carried out two times by submerging the Type-2 sample in a 20:30:10 ratio and heated at 50°C for 24 hours.

- 1st cycle = The three chemicals; TMCS, n-hexane, & ethanol were mixed in the beaker and silica gel obtained from the solved exchange method was submerged into the solution. The whole sample was placed in an airtight container and kept in an oven for 24 hours at 50°C.
- 2nd cycle = The previous solution was replaced with a new fresh mix of three chemicals and silica gel was again submerged in it at the same temperature for the same period.

3.3.7 Washing with n-Hexane

After completion of the modification, the separation of the ethanol in the solution was seen which was then drained out. To remove untreated TMCS and other chemicals, the silica gel was washed with n-Hexane three times. This was also done by submerging the treated sample in n-hexane for 24 hours.

3.3.7.1 Washing Type-1 Sample

The modified sample was washed by submerging it into n-hexane for 1 hour and this process was repeated 4 times.

3.3.7.2 Washing Type-2 Sample

- 1st cycle = First treated sample was completely immersed in the n-hexane for 24 hours at ambient temperature and kept in an airtight container.
- 2nd cycle = The n-hexane was replaced and kept in the same condition for the same period of time.
- 3rd cycle = The same steps was repeated as mentioned for the 2nd cycle.

3.3.8 Drying

The final stage was the drying process for converting the hydrogel into the dry sample. The drying process was done in several steps by increasing the temperature from low to high temperature. The drying process was critical to prevent collapsing of the structure due to the shrinkage of the solid sample. Here, the drying was done at the ambient pressure i.e. APD.

3.3.8.1 Drying Type-1 Sample

The drying process was done in 4 steps as mentioned below:

- 1st Step = The washed sample was kept at ambient temperature for 24 hrs for drying.
- 2nd Step = Next day, the sample was dried in the oven at 50°C for 24 hours.
- 3rd Step = Finally, the sample was further dried at 150°C for 2 hours.

3.3.8.2 Drying Type-1 Sample

- 1st Step = The sample was kept for drying for 24 hrs at the ambient temperature under normal pressure.
- 2nd Step = Next, the sample was placed in the oven and kept for 24 hours at 50°C.
- 3rd Step = Again, the sample was heated for 2 hours at 100°C.
- 4th Step = Finally, the sample was heated for 1 hour at 150°C.

Table 3.2: Sample detail

S.No.	Sample	Glass Wool	Modification ratio
1	S1, S2, S3, & S4	-	Type-1 Sample (Modified in TMCS: Hexane: EtOH = 4:30:1)
2	A1, A2, & A3	-	
3	N3, N4	-	Type-2 Sample (Modified in TMCS: Hexane: EtOH = 20:30:10)
4	W3 & W4 (With glass wool)	1g	
5	WW1 & WW2 (With glass wool)	1.5g	

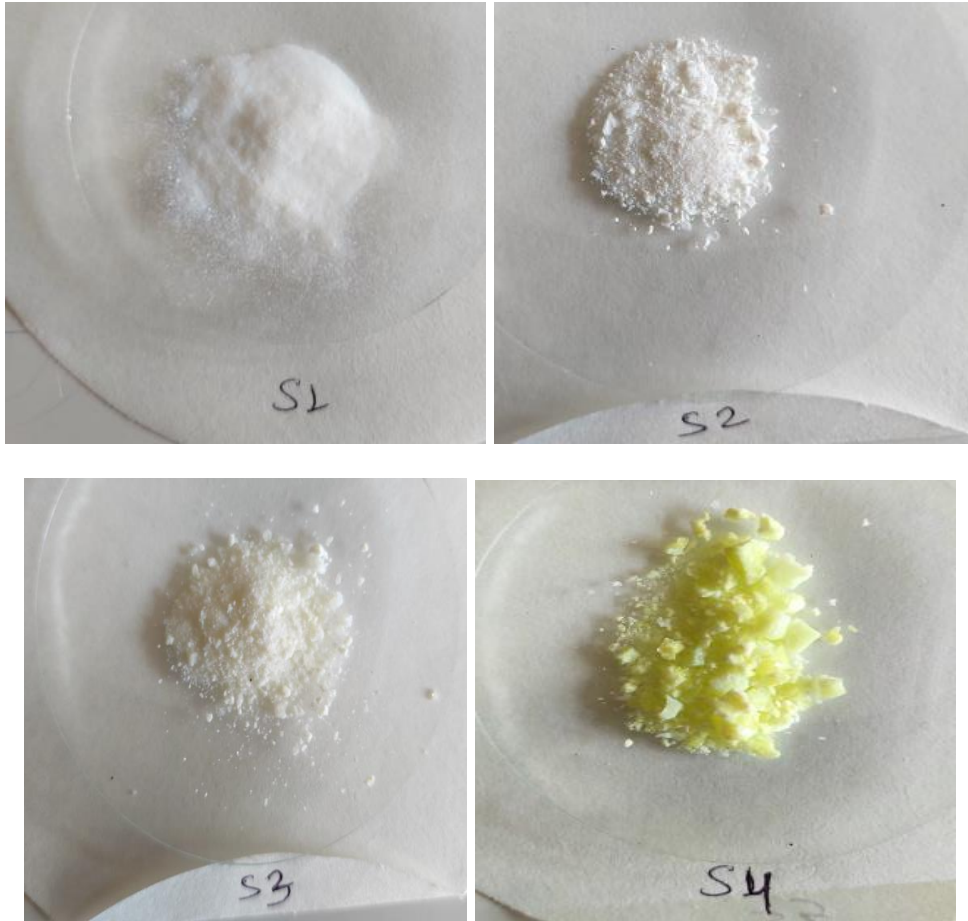


Figure 3.4: S-group samples of Type-1



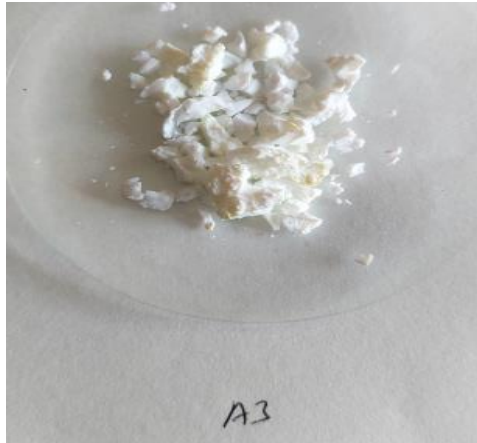


Figure 3.5: A-group samples of Type-1



Figure 3.6: N3 & N4 samples of Type-2



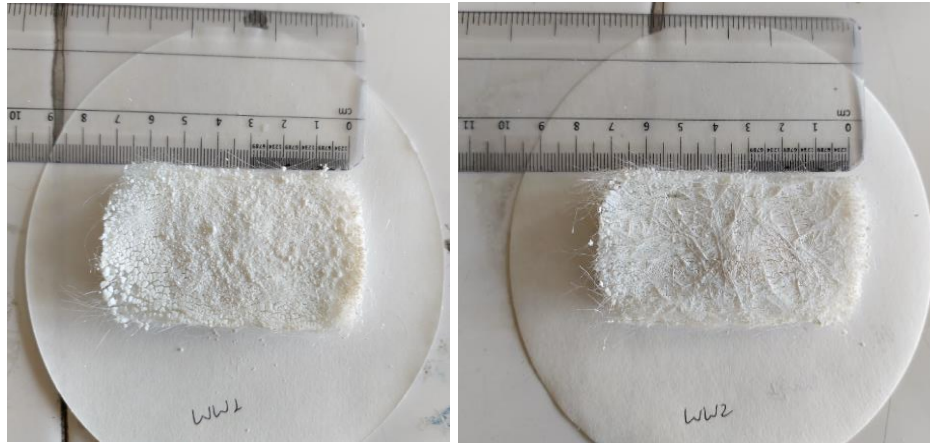


Figure 3.7: Glass wool-reinforced Type-2 samples

3.4 Analysis

3.4.1 X-Ray Diffraction

An X-Ray Diffractometer is an instrument for investigating the material's structure based on the scattered pattern generated by radiation or particles beam such as X-rays interacting with the material. This works on the X-ray diffraction principle and helps to determine whether the material is crystalline or amorphous (Dutrow & Clark, 2022). So, the sample was examined for identifying whether it was crystalline or amorphous.

3.4.2 FTIR

It is the analysis technique to identify the presence of the polymeric, organic or inorganic group. It uses infrared light to scan the samples and analyze the chemical properties. During analysis, it transfers the infrared light having a wavenumber from 10000 to 100 cm^{-1} and absorption by molecules then generates a unique spectral having a wavenumber from 4000 to 400 cm^{-1} . In this way, the presence of the molecule group is detected (FTIR Analysis, 2022). So, this analysis was carried out on the sample to identify the replacement of -OH by the -CH₃ group by studying the transmission peaks over the wavenumber.

3.4.3 Contact Angle Test

The contact angle test is the examination of the surface hydrophobicity of the sample. The contact angle made by liquid-like water on the surface of the material is measured to evaluate the interaction of the surface with the liquid. Due to the surface tension of the liquid, it is a force to form a dome shape. The material is said to be hydrophilic when the contact angle is less than equal to 90° and the material becomes hydrophobic when the contact angle is above 90°. Under hydrophobic, the material is classified into two groups; hydrophobic for a contact

angle between 90° & 150° and super hydrophobic for a contact angle equal to or above 150°. So, the prepared sample was examined for the contact angle to know whether it was hydrophilic or hydrophobic.

3.4.4 Adsorption Test

Silica aerogel is hydrophobic as well as lipophilic in nature and has the ability to adsorb organic solvents (Shi et al., 2014). The organic solvent is adsorbed by the aerogel due to capillary action and filled nanopores present in it. So, the Type-1 (S1) and Type-2 (N3 & WW2) samples were tested for adsorption of organic solvents like ethanol, n-hexane, & benzene. Further, the N3, WW1, & WW2 samples of the Type-2 were used to examine the adsorption of edible oil like sunflower refined oil. The testing trial is explained below.

- i. For S1 & N3 = The respective dry weight of the sample was measured around 0.1 gm (M_d) and then, ethanol was added dropwise until it became saturated. After that, the excess ethanol was drained out. Next, the weight of the wet weight sample was taken (M_w). Using the formula given mentioned by Çok et al., (2021), the adsorption capacity was calculated. The same processes were repeated for the n-hexane and benzene and the experiment was completed in two trials for each organic compound.

$$\text{Adsorption capacity} = (M_w - M_d) / M_d \quad \text{Eq(1)}$$

- ii. For WW2 = The dry weight of the sample was measured first and then ethanol was poured onto the sample until it became saturated and could not adsorb more. After draining the excess ethanol, the wet weight of the sample was measured. The adsorption capacity of WW2 was estimated using eq(1).
- iii. For N3, WW1, & WW2 = The five mixtures of distilled water and 2 mL edible oil were prepared. Then, the dry weight of the N3 was measured and put in each mixture. The mixture was slightly swirled for 10 minutes and the whole mixture was filtered out with filter paper. The sample on the filter contained oil as well which was drained very carefully. Then, the weight of the wet sample was measured using eq(1), and the adsorption capacity was estimated. The adsorption test of the WW1 sample was performed by soaking the sample with oil upto saturation point and then the difference between dry weight & wet was taken. By using eq(1), the oil adsorbed in grams per 1 g of sample was calculated. Next, the mixture of the oil & water was made in a 16:100 ratio by volume and dry weight WW2 was placed in the mixture. After 1 minute, the wet weight was measured & amount of oil removed from the water was calculated.

3.4.5 Desorption Test

The desorption analysis helps to know how long it will take to evaporate adsorbed organic chemicals at a specified temperature under ambient pressure in the sample. For this analysis, the first wet weight of the sample was noted after soaking with respective organic chemicals and kept in the oven at 30°C. Next, the weight of the sample was taken at specified intervals of time until the initial dry weight of the sample was not achieved. The desorption test was carried out for S1, N3, & WW2 samples. The S1 and N3 were in powder form and the dry weight was taken less so the drying weight was measured at 5 minutes intervals. For the sample WW2, the drying weight was measured at 10 minutes intervals.

3.4.6 UV-Visual Spectral Study

The UV-Visual spectral analysis helps to know the presence of silica in the sample as it has low intrinsic absorbance in the visible region between 380 to 700 nm whereas transmittance is high. So, the Type-1 and Type-2 samples were passed through this analysis. Further, the band gap was also estimated by determining the reflectance against wavelength. Here, the Kubelka & Munk theory is adopted to determine the Kubelka- Munk function, $F(R_\infty)$ based on diffusing reflectance (Abdullahi et al., 2016).

$$F(R_\infty) = \frac{(1 - R_\infty)^2}{2R_\infty} = \frac{K}{S} \quad \text{Eq (2)}$$

Where,

R_∞ = Diffuse reflectance

K = Adsorption coefficient

S = Scattering coefficient

Next, according to the Tauc relation, the direct band gap material relation can be expressed as

$$A(h\nu - E_g)^n = \alpha h\nu \quad \text{Eq (3)}$$

Where,

E_g = Band gap energy

α = Linear adsorption coefficient

ν = Frequency of light

A = Proportionality constant

$n = 1/2$ (for direct band gap materials)

By considering the perfect scattering of incident radiation in a diffusion manner, the value of K is 2α and then assuming, S as a constant with respect to wavelength, the Kubelka-Munk function becomes proportional to the α & final relation is

$$A(h\nu - E_g)^n = [F(R_\infty) \times h\nu]^2 \quad \text{Eq (4)}$$

Using eq(4), the energy band gap of the sample was estimated by plotting the graph between $(\alpha h\nu)^2$ against $h\nu$.

3.4.7 SEM Characterization

This type of characterization helps to know the surface morphology, composition of the chemical, material orientation, & crystalline structure of the prepared sample at the atom level. In this, the high beam of energy beam of electrons is used to take the image of the sample so it is called a scanning electron microscope. So, the Type-1 samples S1 and S4 were characterized through this SEM under 2000, 5000, and 10000 zooms.

3.4.8 Thermal Stability

The TG-DTA is used to determine the loss of the mass or decomposition of the sample over the range of the temperature as well as estimate the phase transition of the sample. The thermal stability of the silica aerogel is mainly done using TG-DTA in which the sample's critical temperature is noted when the sample lost its hydrophobic property. But due to the unavailability of the instrument, the thermal stability was checked in the tube furnace upto 300°C only in the presence of the nitrogen gas. Further, the sample is also tested at 200°C by heating in the oven for 1 hr. After heating, the hydrophobic property was checked visually by placing the water droplet on the sample.

3.4.9 Mechanical Testing

The visual interpretation was done to identify whether the flexural & tensile testing was feasible to be conducted on the prepared samples. The sample with and without glass was examined and based on the physical condition, further step for testing was determined.

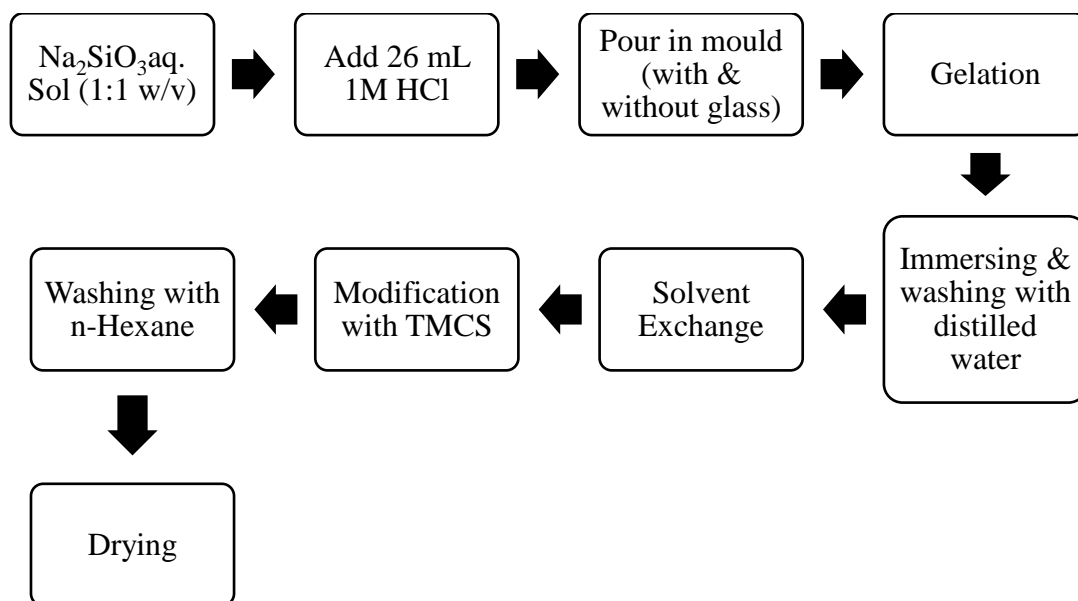


Figure 3.8: Synthesis steps

CHAPTER 4: RESULT AND DISCUSSION

4.1 FTIR Analysis

4.1.1 Water Glass

The study of functional groups presents in the precursor water glass (Na_2SiO_3) used for making an aerogel sample was done. The FTIR graph plotted for transmission over wavenumber or frequency between 4000 to 400 cm^{-1} is presented below. In the plot, the peak is noted at 3244 cm^{-1} which indicates the presence of the -OH group due to the stretching vibration and this functional group between 3600 to 2700 cm^{-1} (Bobrowski et al., 2012) mainly represents the water molecule. The molecular vibration at 2324 cm^{-1} indicates the vibration of the hydrogen bonds between the silicon lattice and $\text{Si}(\text{OH})_4$. The molecule of the alkenes containing the double bonds between carbon atoms usually vibrate between frequency 1600-1680 (Fox, 2013) and this compound in the source is confirmed by molecular stretching at 1647 cm^{-1} . The bands between 1200 to 1000 cm^{-1} (Zimnoch, 2015) show the asymmetric stretching vibration of Si-O-H and it is ensured by the peak at 983 cm^{-1} . Further, the peaks from 800 to 400 cm^{-1} show the bending vibration of the bands [O-Si-O], lattice vibration, and pseudo-crystal.

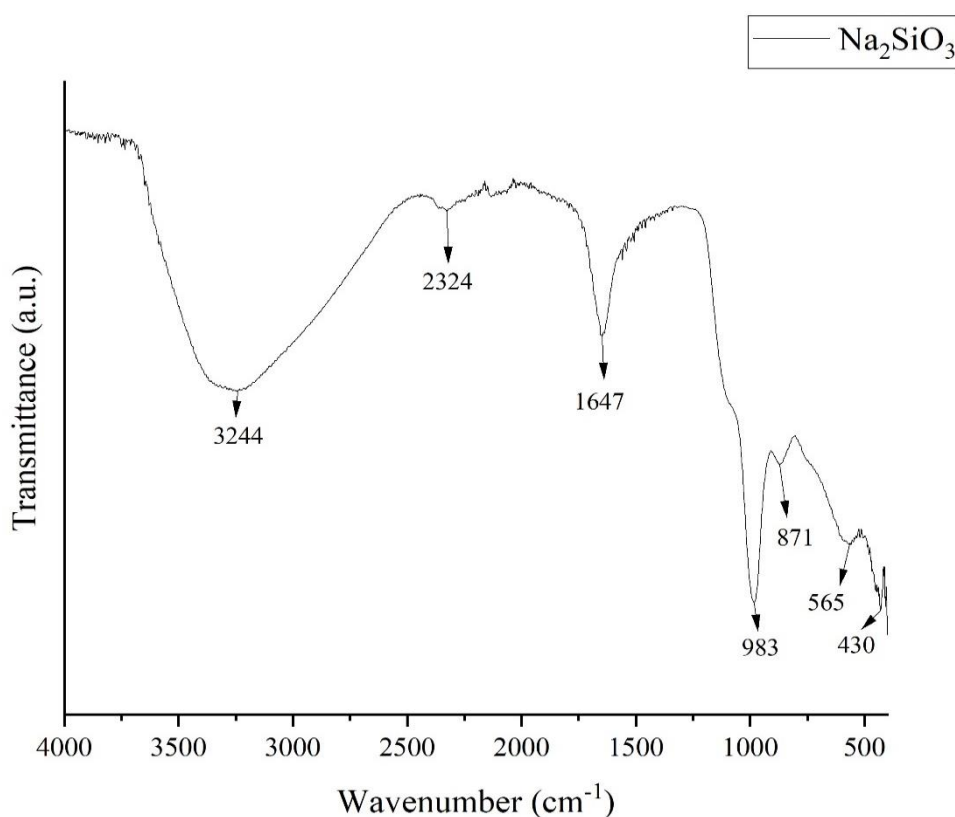


Figure 4.1: FTIR plot of water glass

4.1.2 Type-1 Sample

The properties of the prepared aerogel sample from sodium silicate are studied by evaluating the functional groups using an FTIR plot. The Type-1 sample category contains 7 samples which were prepared by following the sol-gel process, solvent exchange, surface modification in a 4:30:1 ratio of TMCS: n-hexane: EtOH by volume, and drying at ambient pressure.

4.1.2.1 S-Group

For sample S1, the stretching vibration of the -OH group of the water is seen at 3367 cm^{-1} frequency. The presence of the Si-C bond is observed at 2964 cm^{-1} which lies between the frequency range of $2970\text{-}2010\text{ cm}^{-1}$ (Karamikamkar et al., 2019). The peak band at 1631 cm^{-1} also indicates the presence of the -OH group of the water (Maleki et al., 2015). The three dimensions of the network of the Si-O-Si are established by asymmetric vibration at 1056 cm^{-1} which is in between the frequency range $1250\text{-}1000\text{ cm}^{-1}$. The Si-OH bond is found in the sample due to the presence of a peak at 944 cm^{-1} which is between $950\text{ \& }810$ (Launer & Arkles, 2013). Again, the S1 sample shows a peak at 846 cm^{-1} for the Si-C bond between $865\text{ to }750\text{ cm}^{-1}$ (Salimian & Zadhoush, 2019). From this analysis, it is confirmed that the presence of the Si-C is due to the replacement of the -OH group by the -CH₃ group provided by the TMCS during modification. Although, it is also observed that -OH is not completely removed as indicated by peaks 3376 cm^{-1} and 1631 cm^{-1} . The presence of the Si-C bond (i.e., -CH₃) made the S1 sample hydrophobic in nature.

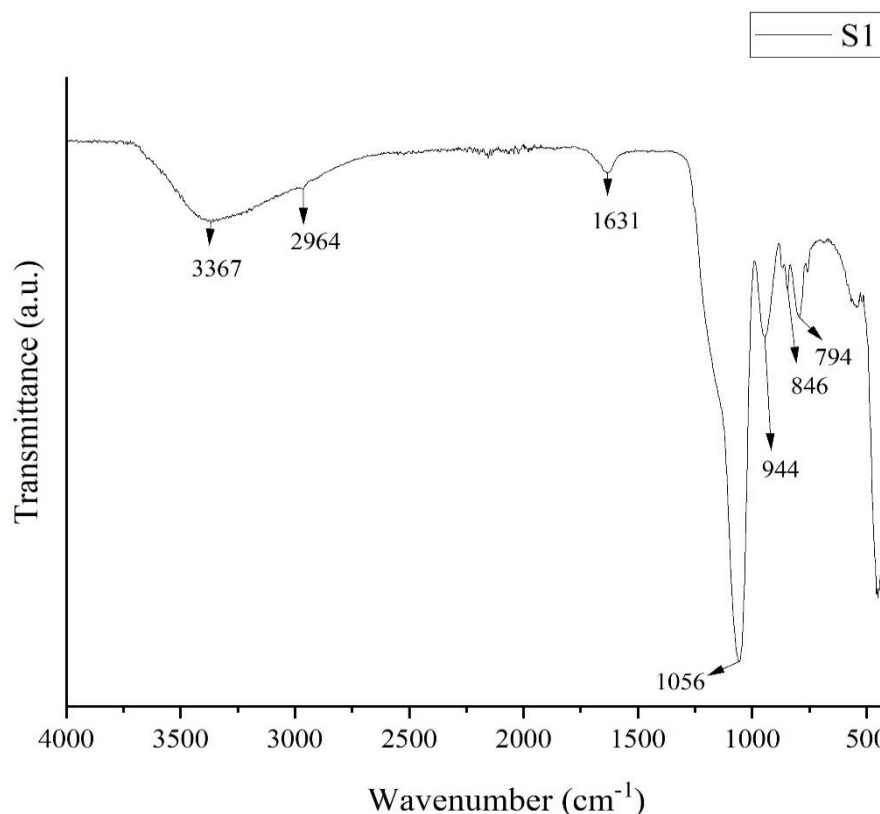


Figure 4.2: FTIR plot of Type-1 sample S1

Next, the comparison of other samples S2, S3, & S4 was done based on S1. Here, the peak at 3367, 1631, 1056, 944, & 794 cm^{-1} frequencies are seen the same as found for the S1 sample. But the only difference is the presence of the peak for the Si-C bond. In sample S2, the peak intensity at 2964 & 846 cm^{-1} is less than found in the S1 sample. Similarly, the intensity of the Si-C bonds at those peaks kept on decreasing for S3 and disappeared for the S4 sample. From Si-C bond intensity variation, it is confirmed that the longer the sample was submerged in the modification solution caused the better replacement of -OH with -CH₃ as S1 was submerged for 48 hrs and S4 was submerged for only 12 hrs. Further, it is established that S1 shows good properties of hydrophobicity due to the Si-C bond even in the presence of -OH molecules. The hydrophobicity is decreased for S2 followed by S3 based on the presence of the Si-C bond and the S4 sample does not show a hydrophobic nature. So, the minimum time for modification is defined to be 24 hours at ambient temperature in the air-tight container because S1, S2 to S3 were able to have -CH₃ as they were submerged for 24 hrs or more in modification solution ratio of 4:30:1. In the figure, the solid circle shows the absence and the dashed circle shows the presence of a peak at 2964 and 846 cm^{-1} .

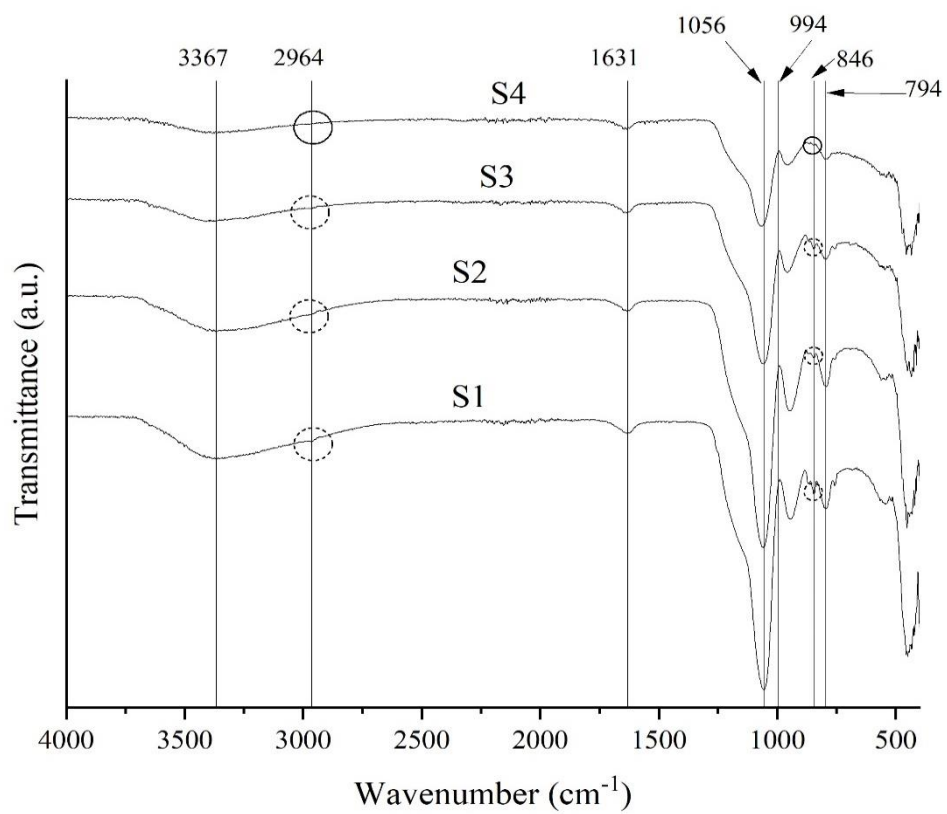


Figure 4.3: Comparison of S-group samples

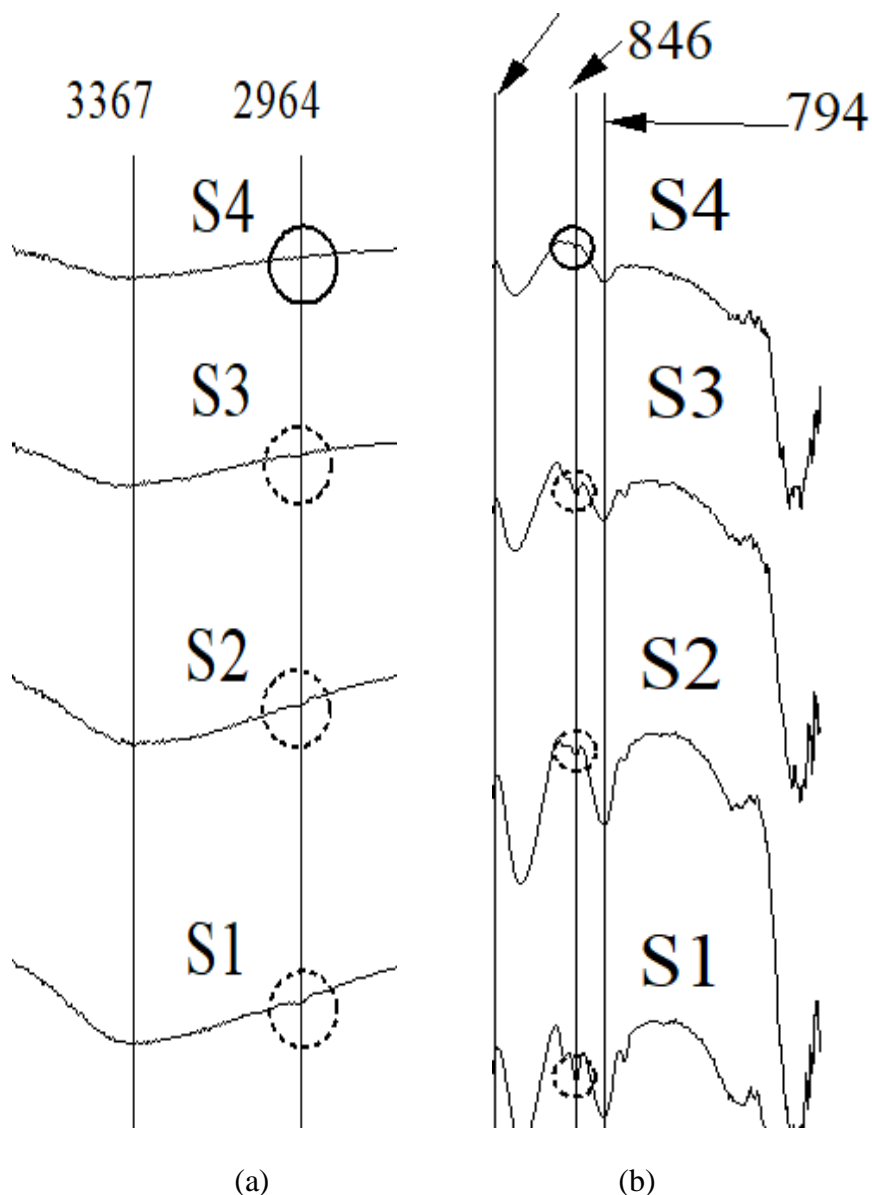


Figure 4.4: FTIR zoomed section at 2964 cm⁻¹ (a) and 846 cm⁻¹ (b) peaks

4.1.2.2 A-Group

The surface modification of the A1, A2, & A3 (A-Group) samples were also done in the same ratio as done for S-Group samples. For A1, the peaks for -OH and Si-O-Si are similar to the S1 sample and major peaks at 2962 cm⁻¹ and 844 cm⁻¹ are observed in the graph which shows that A1 is also hydrophobic. Thus, the A1 sample also contains the Si-C bond which is formed after replacing the -OH group with -CH₃ during modification using TMCS for 24 hours. But the Si-C bond is not found in A2 and A3 samples as their modification time was less than 24 hrs due to which they are still hydrophilic.

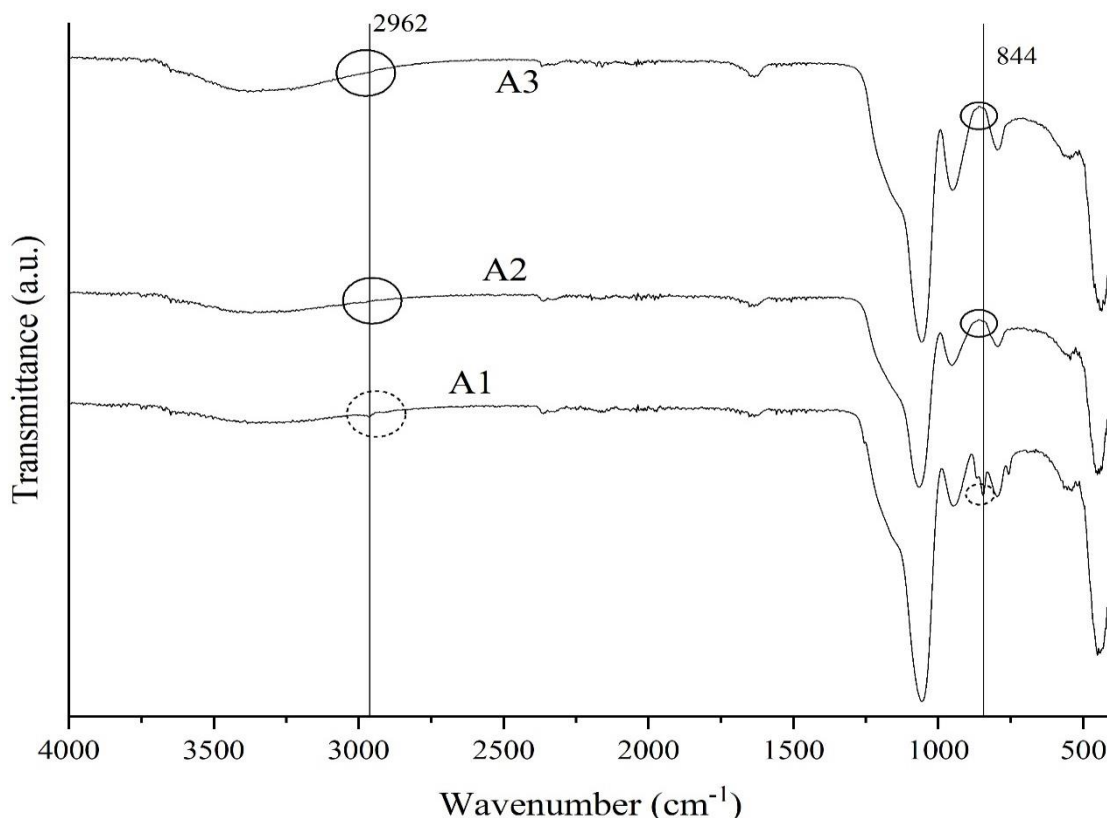


Figure 4.5: FTIR plot of A-group samples

4.1.3 Type-2 Sample

Under the Type-2 sample, 6 samples were made and modified twice by TMCS: n-Hexane: EtOH in a 20:30:10 ratio by volume for 24 hours at 50°C in air-tight containers and the name of the samples are N3, N4, W3, W4, WW1, & WW2. From the FTIR of the N3 sample, the peak of the -OH group between the 3600 to 2700 cm^{-1} frequency range is missing and small stretching is seen at 1635 cm^{-1} which indicated that the -OH group is removed in more quantity as compared to the Type-1 sample. The presence of the Si-C bond is confirmed due to the peak at 2964 cm^{-1} and 844 cm^{-1} . Due to the removal of the -OH and the presence of Si-C, the N3 sample also confirm to be hydrophobicity. After that, the other samples (W3, W4, WW1, & WW2) also the show same peaks as shown by the N3 samples. So, all samples under Type-2 are hydrophobic in nature and contain less -OH group than Type-1 samples because the modification was twice at 50°C which shows better replacement of the -OH group with $-\text{CH}_3$.

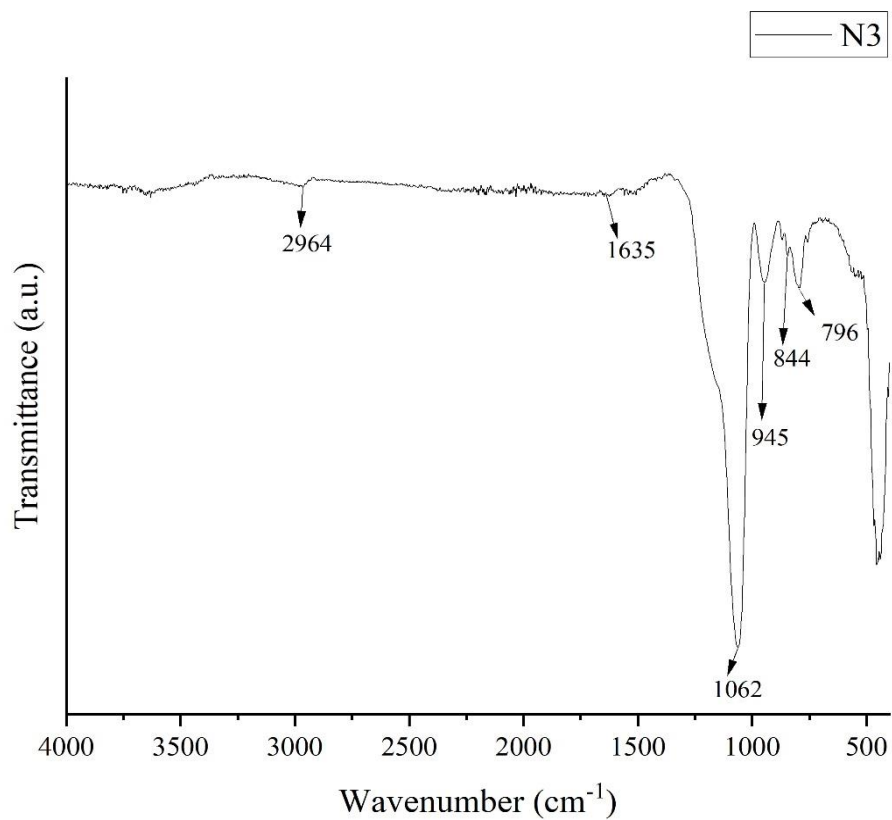


Figure 4.6: FTIR plot of Type-2 sample N3

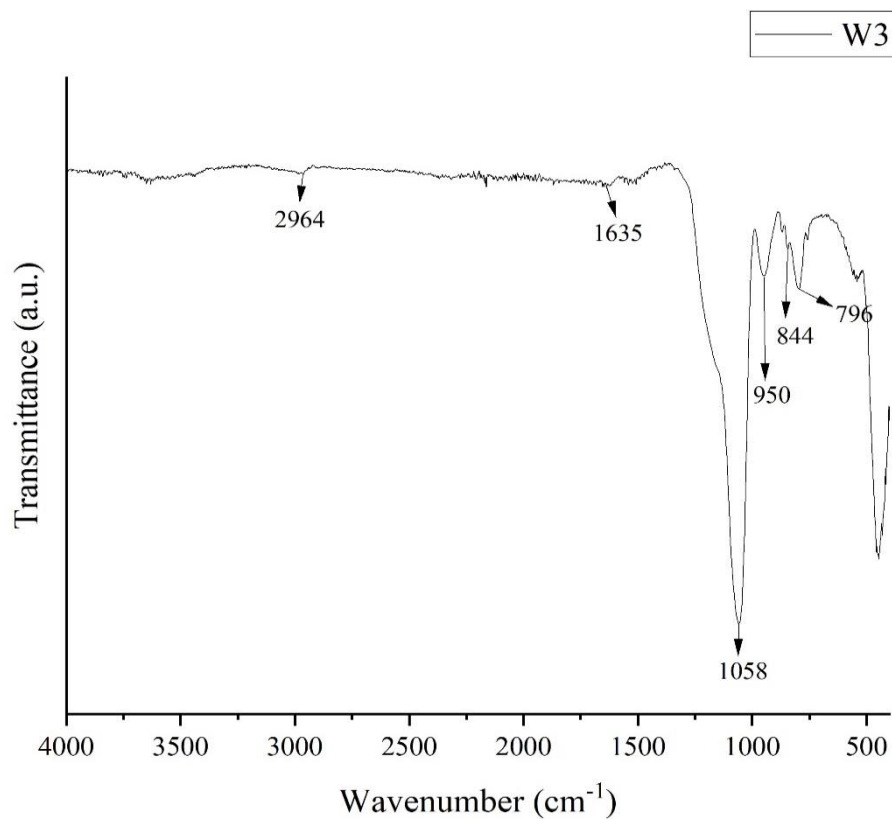


Figure 4.7: FTIR plot of Type-2 sample W3

4.2 Contact Angle

The contact angle between the surface of the sample and the water was measured to confirm the hydrophobicity. As found from the FTIR result, the Type-1 and Type-2 show the presence of the Si-C bond which made the sample hydrophobic. So, the contact angle of water on the surface of samples S1 and N3 was measured. The contact angle for the S1 sample is found to be 124° which was greater than 90° and indicated that the sample is hydrophobic. Next, the contact angle for the N3 is noted to be 146.7° which is close to the 150° achieved by Salimian & Zadhoush, (2019). So, both samples exhibit hydrophobicity based on these results. Here, S1 shows less contact angle than N3 because even after modification, the -OH group is still present in the S1 as -CH₃ was not able to replace the -OH completely in comparison to the N3 sample. The N3 was modified twice with more quantity of the TMCS as compared to the S1 sample which helped to reduce the presence of the -OH group.

Table 4.1: Contact angle result for S1 & N3 samples

Sample Type	Sample Name	Contact Angle	Comment
Type-1	S1	124°	Hydrophobic
Type-2	N3	146.7°	Hydrophobic

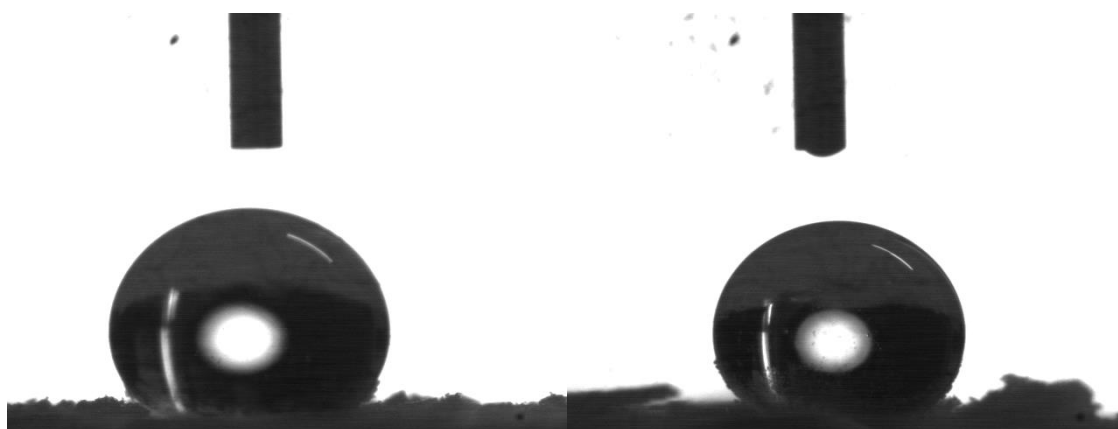


Figure 4.8: Contact angle test of S1 & N3



Figure 4.9: Contact angle testing

The contact angle using oil or organic solvent was difficult to measure as the sample quickly adsorbed the small droplet. As seen in the photo, the oil droplet is almost flat as compared to the water droplet which indicates the prepared sample is oleophilic.

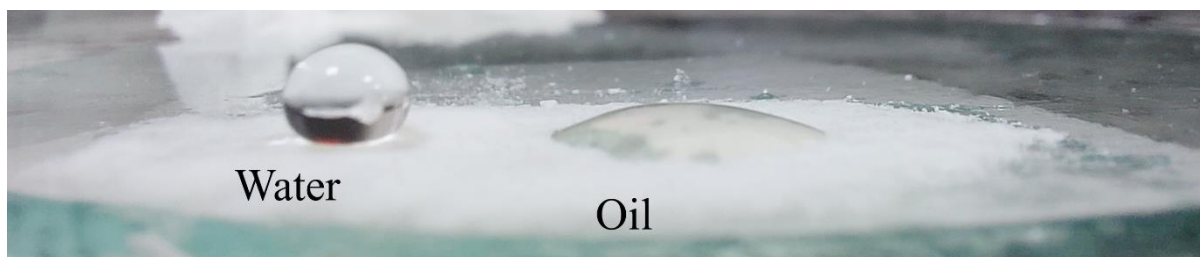


Figure 4.10: Droplet of water & edible oil on the sample

4.3 Adsorption Test

4.3.1 Using EtOH, Benzene, & n-Hexane

The adsorption test was performed by using organic liquids; ethanol, benzene, & n-hexane using S1 and N3 samples. The below table shows the adsorption of each organic liquid per 1g of each sample due to capillary action. Based on the calculation, 1 g of S1 and N3 adsorbed 4.1555 g and 4.9421 g of benzene respectively which is a greater amount than ethanol and n-hexane. After comparing, the adsorption amount by N3 is higher as compared to the S1. Based on this, it is confirmed that the pore size of the N3 is larger than S1. The adsorption capacity

of the silica aerogel stated by Rao et al., (2007) was found to be 19.92 g benzene, 14.54 g ethanol, & 10.95 g hexane by 1 g of sample. By comparison, the adsorption quantity shown by Type-1 (S1) and Type-2 (N3) is less due to less porosity. As it is mentioned that the adsorption capacity of activated carbon is less than 1 g/g for organic solvents (Shi et al., 2014) but silica aerogel has more than 7 g/g adsorption capacity. Here, the adsorption capacity of the S1 and N3 is 3 to 4 times higher than activated carbon.

Table 4.2: Adsorption test result for S1 and N3

Trial	Sample	Organic Liquid	Dry Weight (g)	Wet Weight (g)	Adsorbed weight (g)	Liquid adsorbed (g) per 1g sample	
							Average
1	S1	Ethanol	0.1007	0.4958	0.3951	3.9235	3.9487
2			0.1030	0.5123	0.4093	3.9738	
1		Benzene	0.1011	0.5112	0.4101	4.0564	4.1555
2			0.1013	0.5323	0.4310	4.2547	
1		n-Hexane	0.1009	0.389	0.2881	2.8553	3.0573
2			0.1014	0.4319	0.3305	3.2594	
1	N3	Ethanol	0.1021	0.5262	0.4241	4.1538	4.3036
2			0.1019	0.5557	0.4538	4.4534	
1		Benzene	0.102	0.6037	0.5017	4.9186	4.9421
2			0.1015	0.6055	0.5040	4.9655	
1		n-Hexane	0.1001	0.478	0.3779	3.7752	3.7831
2			0.1009	0.4834	0.3825	3.7909	

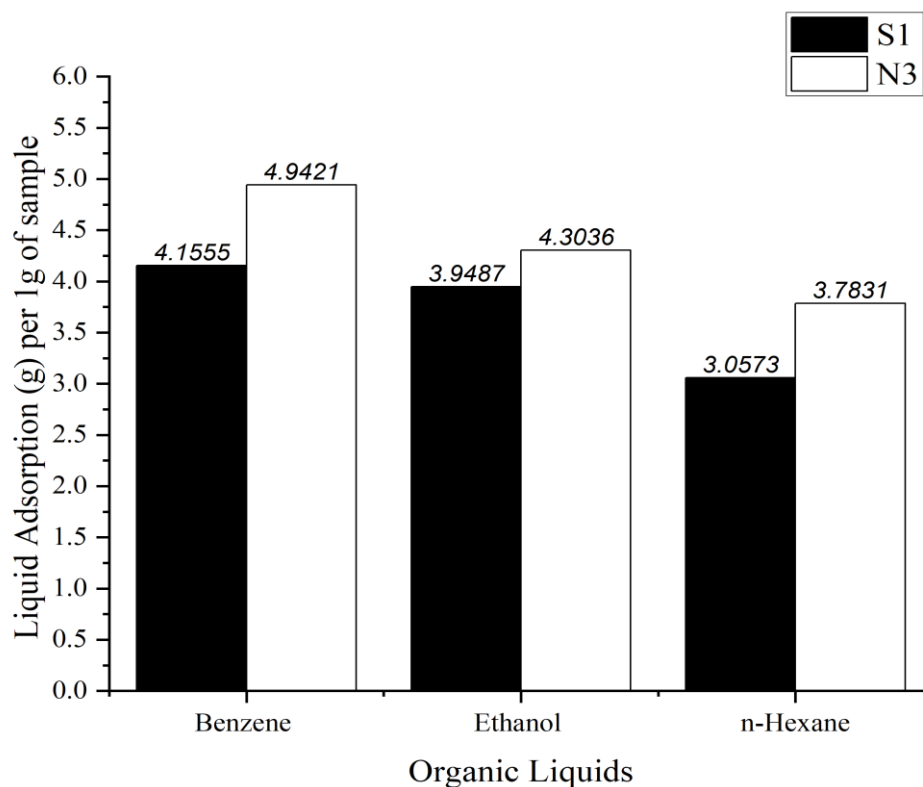


Figure 4.11: Comparison adsorption chart for S1 and N3

Further, the comparison of adsorption is made based on the surface tension of the organic liquid. Rao et al., (2007) proved that the adsorption of aerogel made of silica increases with an increase in the surface tension of the organic liquid. After experimenting with three organic liquids; benzene, ethanol, & n-hexane, the same phenomenon is observed as the surface tension is increased, and the adsorption amount by the sample also rises.

Table 4.3: Adsorption result of S1 & N3 based on organic surface tension

S.No.	Organic Liquid	Surface Tension mN/m (at 20°C)	Liquid adsorbed (g) per 1g sample	
			S1	N3
1	Benzene	28.9901	4.1555	4.9421
2	Ethanol	22.29	3.9487	4.3036
3	n-Hexane	18.5	3.0573	3.7831

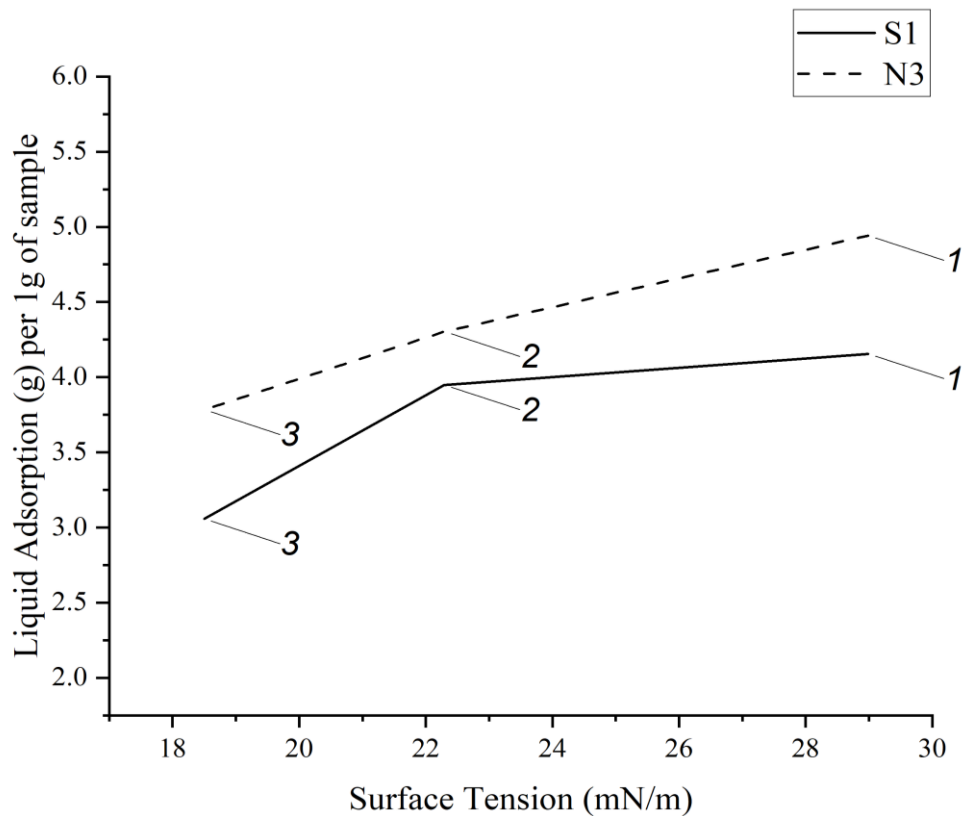


Figure 4.12: Variation of liquid adsorption by the sample based on surface tension



Figure 4.13: Sample soaked in an organic liquid

4.3.2 Using EtOH

Further, the adsorption test was also performed for the WW2 sample containing the glass wool. The dry mass of the WW2 sample was taken first and then soaked with ethanol completely. After soaking, the wet weight was taken and by subtracting it from the initial dry mass, it gave 15.1104 g of soaked ethanol. So, the unit mass (1 g) of the WW2 sample consumed around 4.3735 g of ethanol. By comparing with the N3 sample, WW2 has adsorbed approximately the

same amount of ethanol which confirmed better adsorption of organic solvent by Type-2 samples again.

Table 4.4: Adsorption test of WW2 using EtOH

Dry Weight (g)	Wet Weight (g)	Adsorbed Oil (g)	Oil Adsorbed (g) per 1g of Sample
3.4550	18.5654	15.1104	4.3735

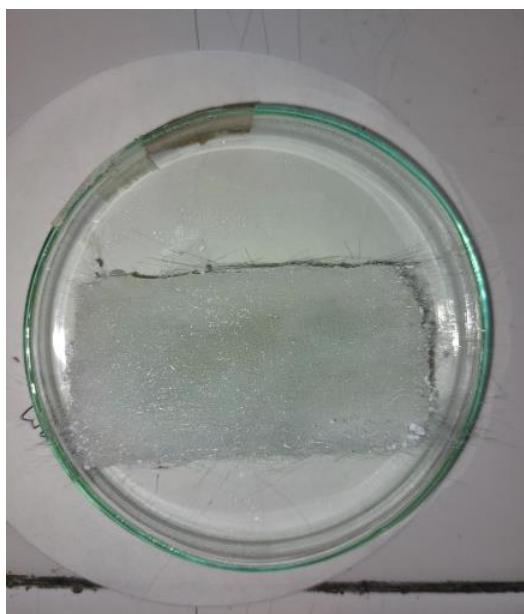


Figure 4.14: WW2 soaked with EtOH

4.3.3 Using Sunflower Oil

From the above test, the adsorption capacity of the Type-1 sample is seen better. So, the sunflower refined edible oil was used to examine the adsorption for N3, WW1, and WW2 where N3 & WW2 were placed in an oil & water mixture and WW1 was soaked in oil only. First, the N3 sample was used to remove the 2 mL oil from 10 mL distilled water. The sample N3 was placed in the mixture and after swirling for 10 minutes, the whole sample was filtered through filter paper. The extra suspended oil on the filter was drained out and the weight of the soaked sample was measured. The 5 trials were done and the average soaked weight of the oil is estimated to be 4.3250 g per 1 g of N3 sample. The surface tension of sunflower oil is 34 mN/m (Esteban et al., 2012). Based on the previous tests, it is found that the adsorption should be greater with high surface tension but it is not seen for sunflower oil as the value is less than

benzene. The reason behind this is the viscosity where sunflowers possess higher viscosity as compared to benzene so adsorption due to capillary action takes more time to adsorb its full potential. The second reason for the less adsorption is due to the loss of the sample during filtering as some of the sample powder was stuck on the glass beaker with oil. Next, the experiment of the sunflower oil adsorption was carried out with the WW1 sample as well. Here, the 3.3125 g WW1 was able to adsorb 21.028 mL of oil which gives 6.3481 g of oil per 1 g of WW1 sample. The reason behind the more oil adsorption in WW1 as compared to the N3 powder is due to the minimum loss of sample as found during filtration for the N3 sample.

Table 4.5: Adsorption test of N3 & WW1 samples using sunflower oil

S.No.	Sample	Dry Weight (g)	Wet Weight (g)	Adsorbed Oil (g)	Oil Adsorbed (g) per 1g of Sample	
						Average
1	N3 in 2:10 mL mixture	0.1014	0.5855	0.4841	4.7742	4.3250
2		0.1065	0.5652	0.4587	4.3070	
3		0.103	0.5189	0.4159	4.0379	
4		0.1056	0.4984	0.3928	3.7197	
5		0.1034	0.5983	0.4949	4.7863	
1	WW1	3.3125	24.3405	21.028	6.3481	6.3481

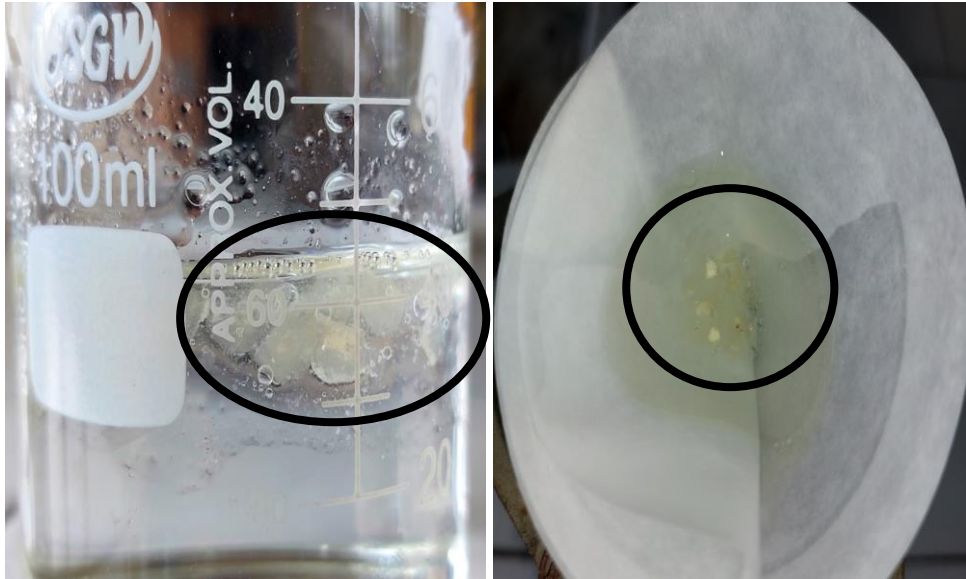


Figure 4.15: N3 placed in an edible oil-water mixture & filtration

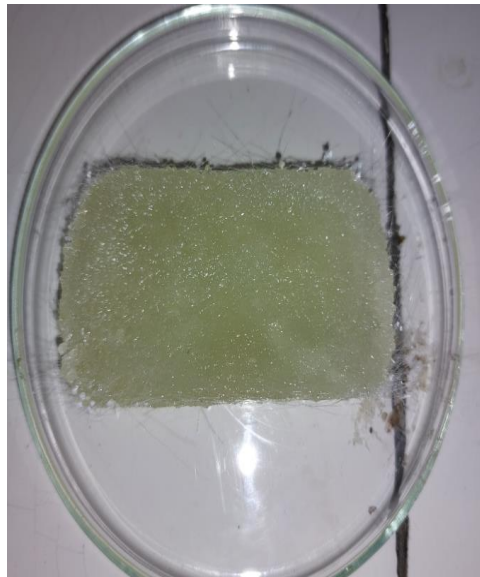


Figure 4.16: WW1 soaked with edible oil

Further, the WW2 sample was placed in the 16:100 by-volume mixture of oil and distilled water which contain 14.56 g of oil in water (oil specification: 1 litre = 910 g). After 1 minute, WW2 was removed from the water and it is found to adsorb 14.4516 g which indicated almost complete removal of oil from the water within 1 minute.

Table 4.6: Adsorption test of WW2 samples using sunflower oil

S.No.	Sample	Dry Weight (g)	Wet Weight (g)	Adsorbed Oil (g)	Adsorbed Oil (mL)
1	WW2 in 16:100 mL mixture	3.349	17.8006	14.4516	15.881

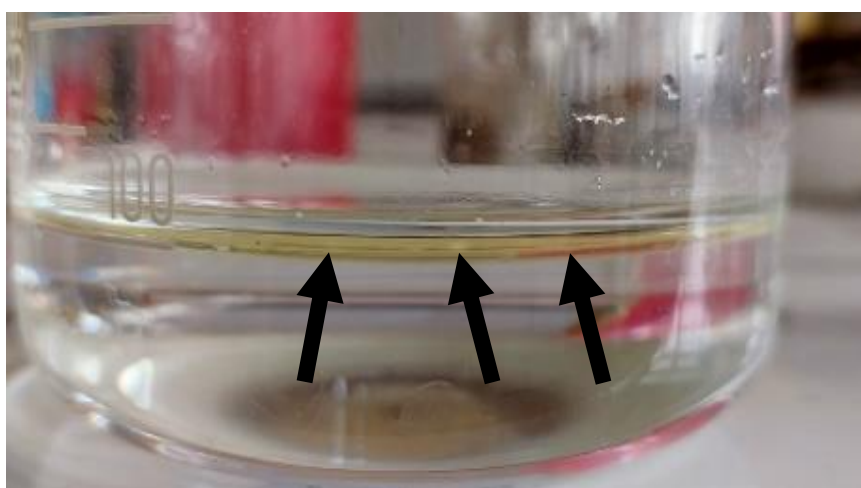


Figure 4.17: Edible oil & water mixture (16:100 ratio)



Figure 4.18: Edible oil adsorbed by WW2



Figure 4.19: Clean water after oil was removed by WW2

4.4 Desorption Test

4.4.1 Using EtOH, Benzene, & n-Hexane

The desorption test was performed to estimate the rate of removal of soaked organic liquid from the sample and the overall maximum time at the specified temperature.

4.4.1.1 For Type-1 (S1)

This test shows that the S1 sample took around 30 minutes with an average rate of evaporation of 0.0136 g/min to attend a full dry state with complete removal of ethanol at 30°C. Next, the average rate of the benzene removal is 0.0285 g/min and took 10 minutes to dry out at the same temperature. Further, the n-hexane from the S1 sample get evaporated at a rate of 0.0210 g/min and S1 became completely dry after 5 minutes. Based on these, the evaporation rate is higher for the benzene due to higher vapour pressure & weak intermolecular forces whereas the evaporation rate is intermediate for n-hexane and less for the ethanol.

Table 4. 7: Desorption result of S1 using ethanol

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.4958	0.5123	0.5040	-	
2	5	0.372	0.3382	0.3551	2.98×10^{-2}	0.0136
3	10	0.259	0.1986	0.2288	2.53×10^{-2}	
4	15	0.178	0.1076	0.1428	1.72×10^{-2}	
5	20	0.1233	0.0985	0.1109	6.38×10^{-3}	
6	25	0.0965	0.0984	0.0974	2.69×10^{-3}	
7	30	0.096	0.0984	0.0972	5.00×10^{-3}	

Table 4.8: Desorption result of S1 using benzene

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.5112	0.5323	0.5217	-	
2	5	0.2090	0.1904	0.1997	6.44×10^{-2}	0.0285
3	10	0.0960	0.0940	0.0950	2.09×10^{-2}	
4	15	0.0940	0.0938	0.0939	2.20×10^{-4}	

Table 4.9: Desorption result of S1 using n-hexane

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.3890	0.4319	0.4104	-	
2	5	0.0969	0.1030	0.0999	6.21×10^{-2}	0.0210
3	10	0.0950	0.0958	0.0954	9.10×10^{-4}	
4	15	0.0949	0.0955	0.0952	4.00×10^{-5}	

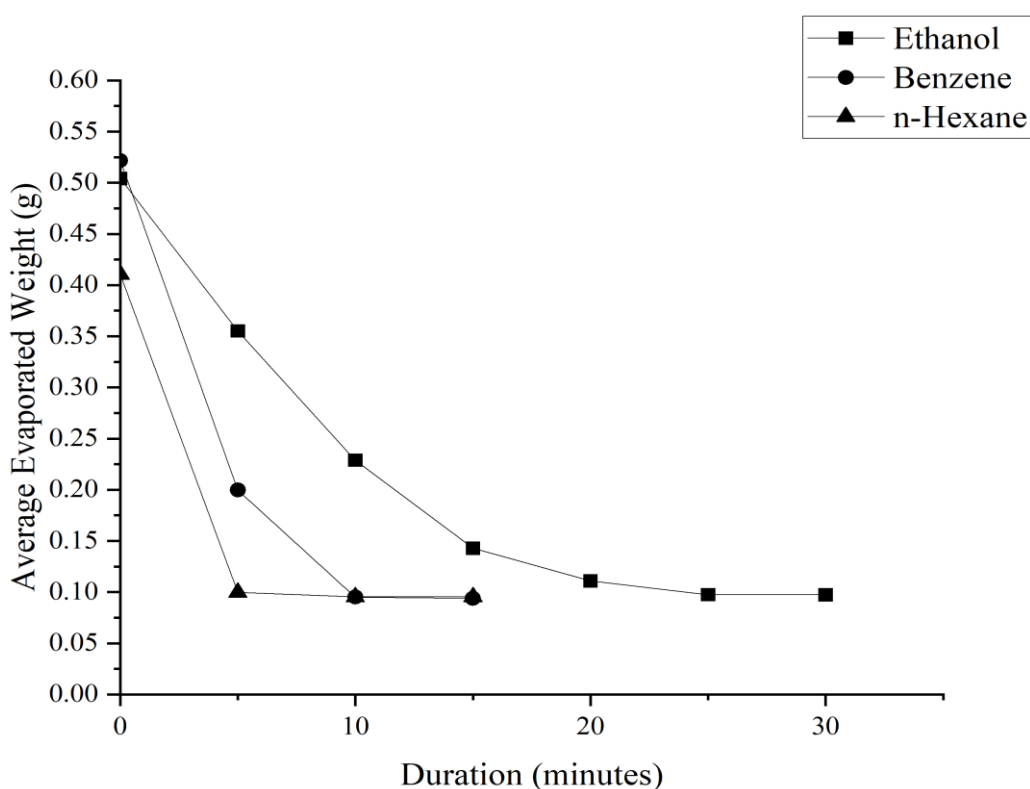


Figure 4.20: Average evaporate weight of soaked S1 over time

4.4.1.2 For Type-2 (N3)

The evaporation rate of ethanol, benzene, and n-hexane are found to be 0.0148 min/s, 0.0254 min/s, and 0.0210 min/s respectively. Here, the N3 was completely dried at 30°C from ethanol,

benzene, and n-hexane after 30 min, 15 min, and 5 min. After comparing with S1, the evaporation time for the ethanol is the same, benzene is increased, and n-hexane is the same.

Table 4.10: Desorption result of N3 using ethanol

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.5262	0.5557	0.5409	-	
2	5	0.3758	0.4170	0.3964	2.89×10^{-2}	0.0148
3	10	0.2295	0.2779	0.2537	2.85×10^{-2}	
4	15	0.1466	0.1568	0.1517	2.04×10^{-2}	
5	20	0.1097	0.1060	0.1078	8.77×10^{-3}	
6	25	0.0967	0.0978	0.0972	2.12×10^{-3}	
7	30	0.0966	0.0977	0.0971	2.00×10^{-5}	

Table 4.11: Desorption result of N3 using benzene

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.6037	0.6055	0.6046	-	
2	5	0.3224	0.2864	0.3044	6.00×10^{-2}	0.0254
3	10	0.1390	0.1141	0.1266	3.56×10^{-2}	
4	15	0.0973	0.0970	0.0972	5.88×10^{-3}	
5	20	0.0972	0.0969	0.0970	2.00×10^{-5}	

Table 4.12: Desorption result of N3 using n-hexane

S.No.	Time (minutes)	Trial 1	Trial 2	Average Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
		Weight (g)				
1	0	0.3890	0.4319	0.4104	-	
2	5	0.0969	0.1030	0.0999	6.21×10^{-2}	0.0210
3	10	0.0950	0.0958	0.0954	9.10×10^{-4}	
4	15	0.0949	0.0955	0.0952	4.00×10^{-5}	

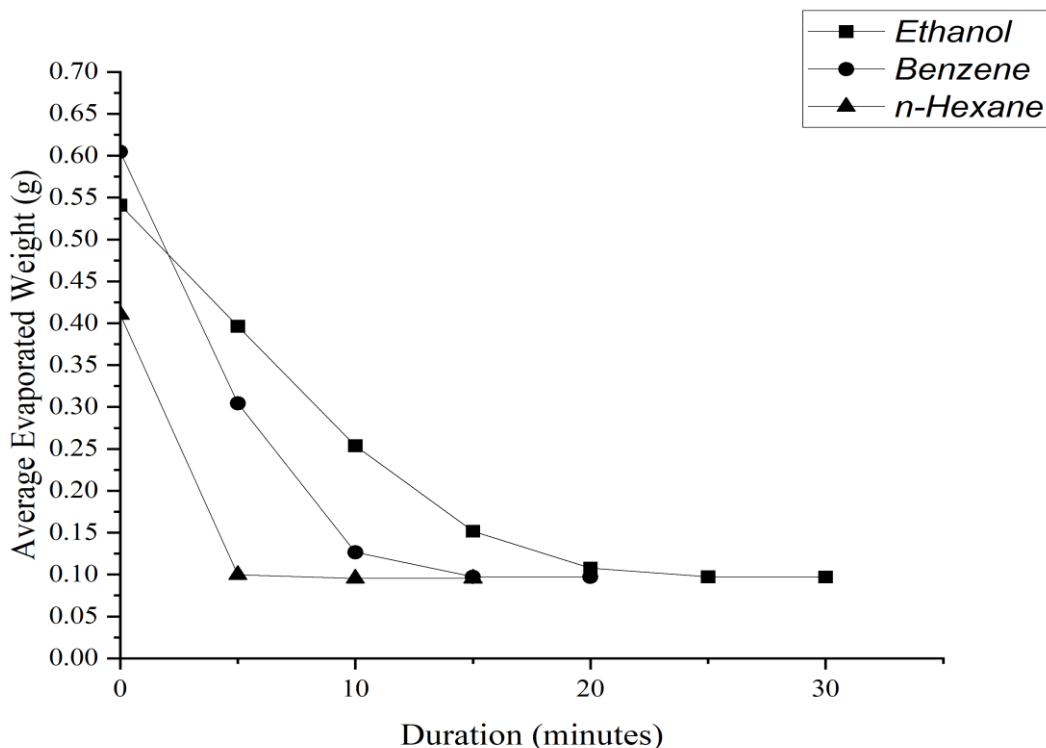


Figure 4.21: Average evaporate weight of soaked N3 over time

4.4.1.3 For Type-2 (WW2)

The desorption of the WW2 sample having 1.5 g glass wool was also examined. The drying process was conducted at 30°C in the oven and weight was noted every 10 minutes until the initial weight was achieved. A total time of 3 hours was taken to completely dry the WW2 sample from ethanol. The average rate of evaporation is estimated to be 0.08432 g/min. The sample WW2 took a long time to dry up from ethanol due to the large mass of the sample and low evaporating temperature. The sample after drying can be again used for adsorbing the organic liquid. The evaporation rate can be increased by rising temperature to 50°C and 100°C which allowed the sample to dry quickly for reapplication. Hence from this test, the prepared sample can be used again for adsorbing the organic liquid.

Table 4.13: Desorption result of WW2 using ethanol

S.No.	Time (minutes)	Evaporated Weight (g)	Evaporation Rate (g/min)	Average Evaporation Rate (g/min)
1	0	18.5654	-	
2	10	17.5906	0.09748	0.08432
3	20	16.2416	0.1349	
4	30	15.0082	0.12334	
5	40	14.4439	0.05643	
6	50	13.2062	0.12377	
7	60	12.006	0.12002	
8	70	10.8361	0.11699	
9	80	9.5999	0.12362	
10	90	8.3623	0.12376	
11	100	7.0905	0.12718	
12	110	6.0223	0.10682	
13	120	5.1562	0.08661	
14	130	4.4738	0.06824	
15	140	3.9701	0.05037	
16	150	3.6232	0.03469	
17	160	3.4357	0.01875	
18	170	3.388	0.00477	
19	180	3.3879	10 ⁻⁵	

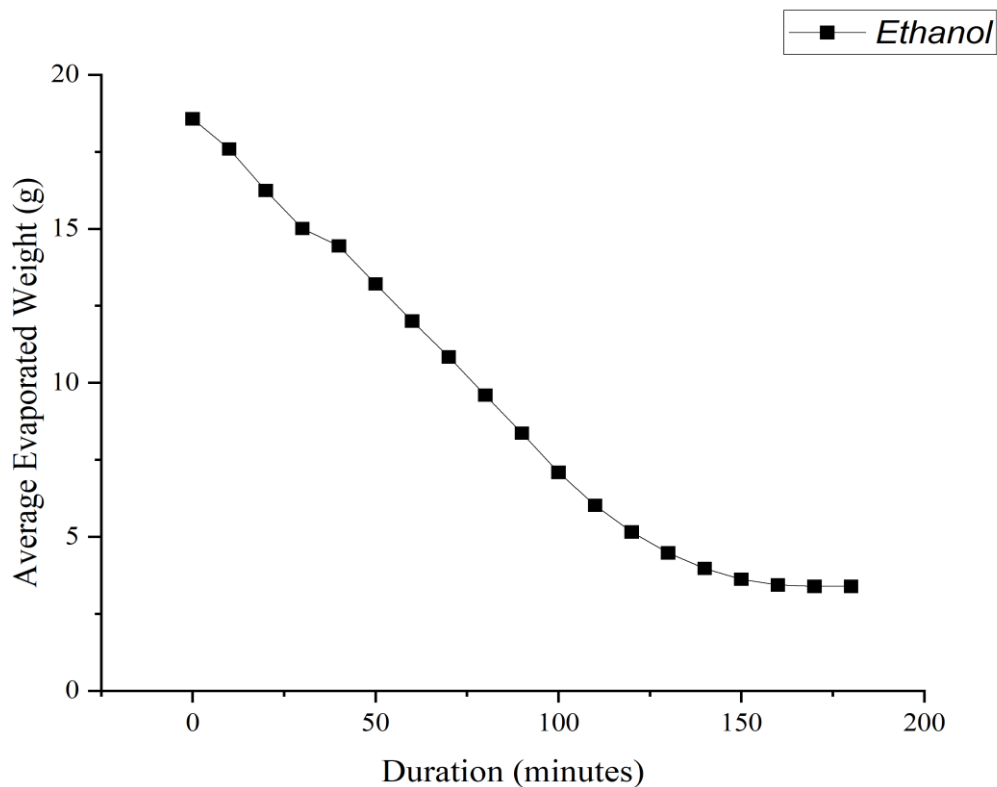


Figure 4.22: Average evaporate weight of soaked WW2 over time

4.5 Optical & Electrical Properties

4.5.1 UV-Vis Spectral Analysis

The behaviour of the S1 and N3 were examined under the ultraviolet spectrum (0 to 400 nm) and visible light spectrum (400 to 700 nm). According to the plotted graph, the N3 shows maximum transmittance in the visible light region and starts to decline under the wavelength below 400 nm which is the UV reason. The behaviour of N3 under the UV & visible light spectrum is similar to the silica aerogel prepared by Werfa et al., (2008). Further, this shows that N3 is capable of absorbing UV light effectively. Next, the behaviour S1 are also evaluated which also shows the same behaviour. Moreover, S2, S3, & A1 of Type-1 and all Type-2 could show the same UV-Vis behaviour as their properties are similar to S1 and N3 respectively which were confirmed in the previous analysis.

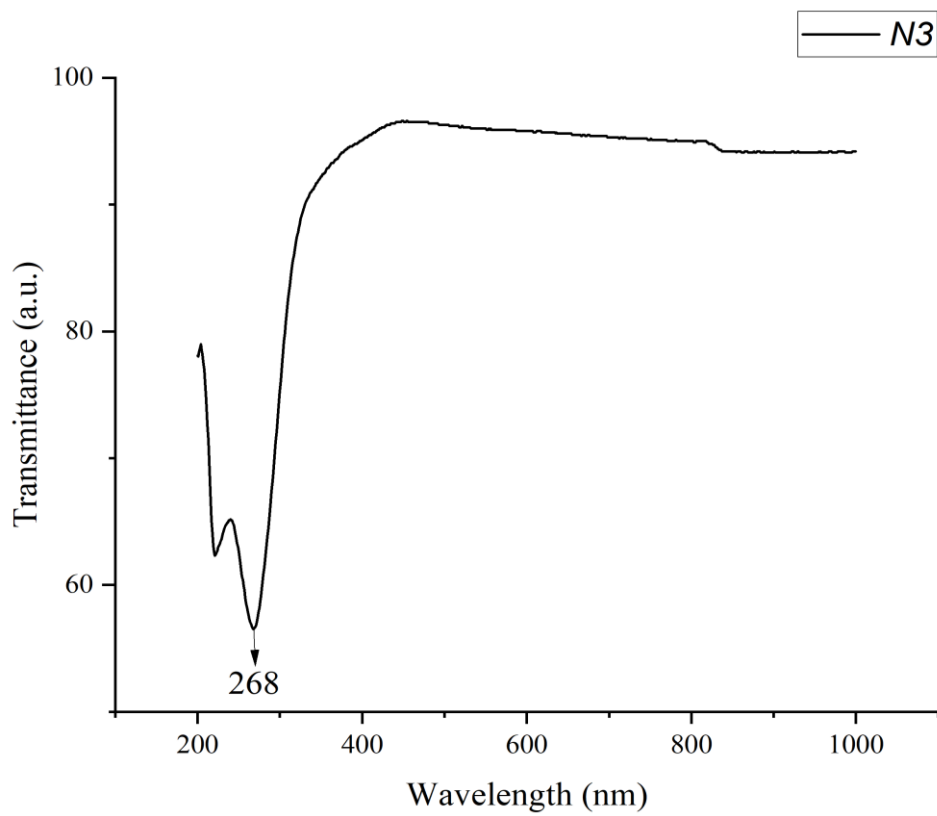


Figure 4.23: Transmittance spectrum for N3

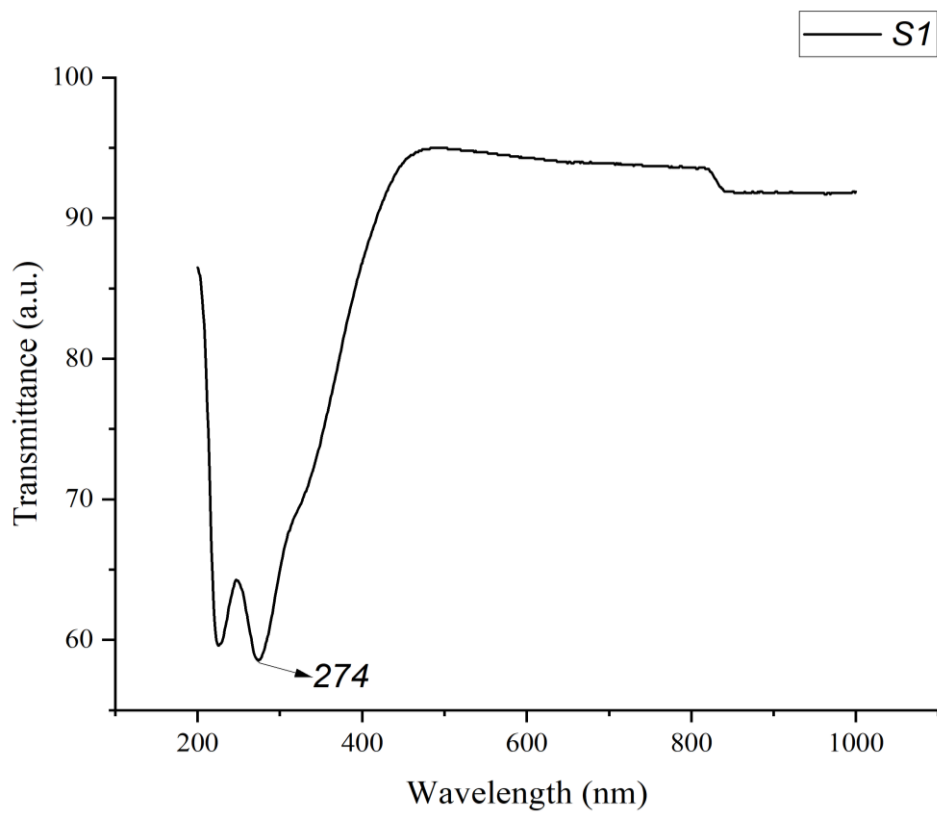


Figure 4.24: Transmittance spectrum for S1

4.5.2 Bandgap Energy

The bandgap energy of the Type-1 (S1) and Type-2 (N3) was evaluated by measuring reflectance against wavelength. Using Kubelka Munk's relation, the $(\alpha h\nu)^2$ was plotted against $h\nu$. Here, the bandgap energy of the S1 is found to be 4.4847 eV which is greater than 4.25 eV as presented by Khedkar et al., (2019). Sample N3 exhibits 4.1499 eV which is close to 4.25 eV. Based on their band gap energy, the S1 and N3 show the property of an electrical insulator.

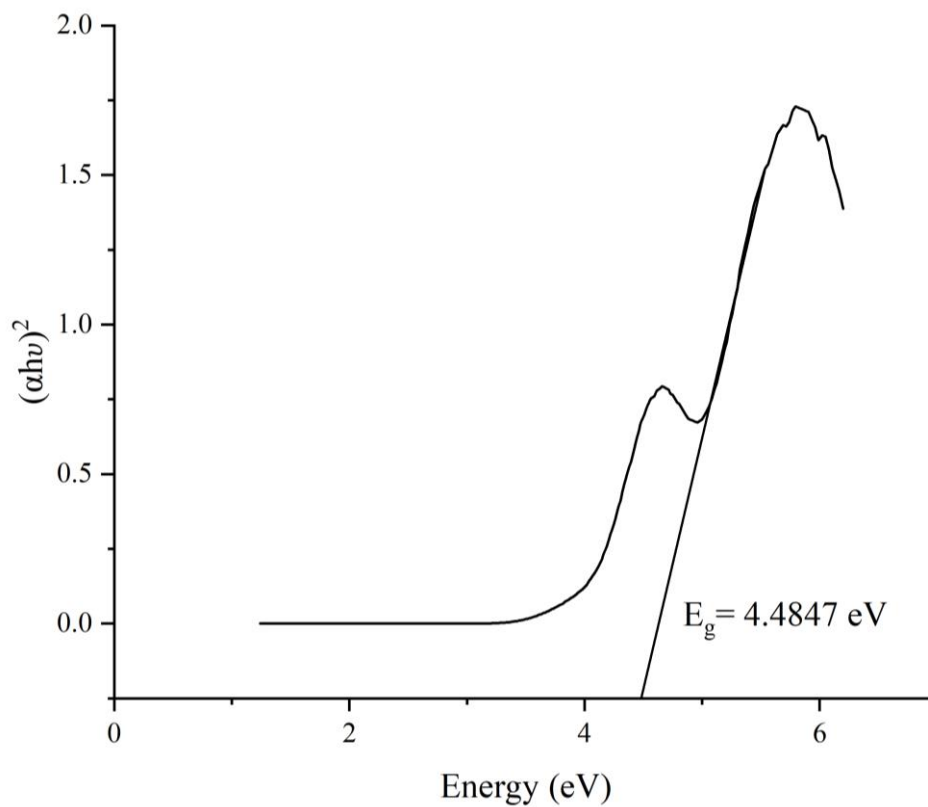


Figure 4.25: Bandgap energy of S1

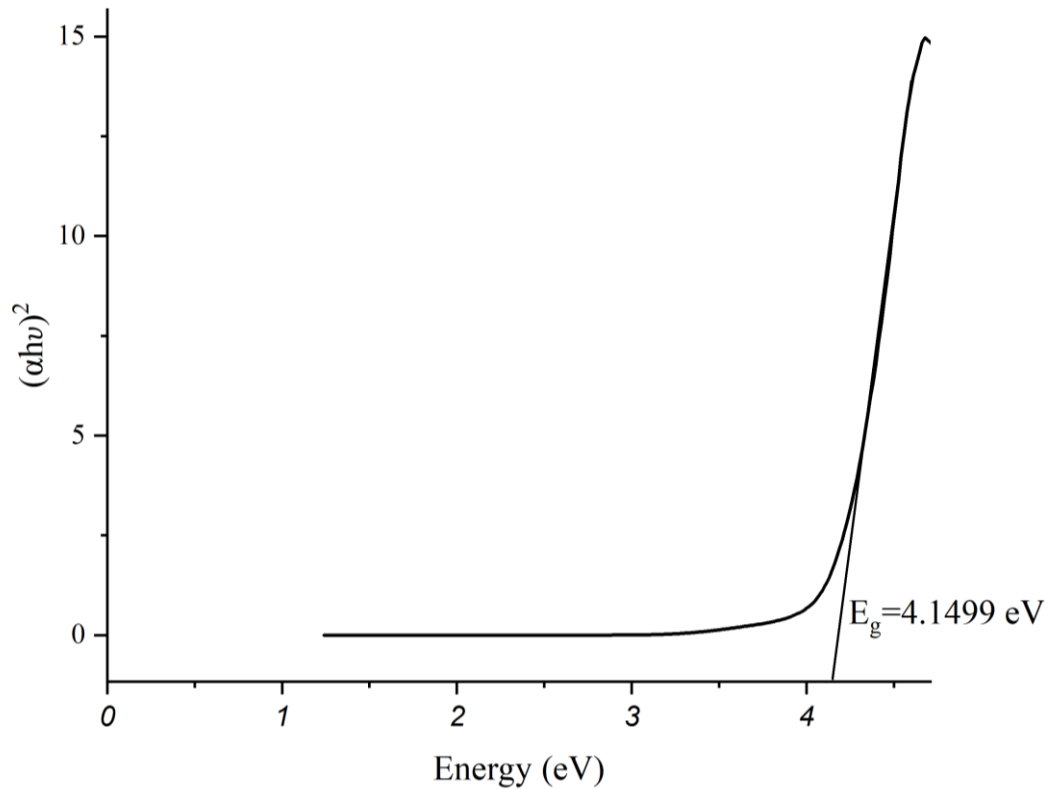


Figure 4.26: Bandgap energy of N3

4.6 XRD

The Type-2 samples; N3 and W3 were examined under the XRD test. Here, the broad peak is seen between 12.55° and 19.9° which indicates that N3 is amorphous in nature (Khedkar et al., 2019). But the two sharp peaks are also noted at 31.58° and 45.32° which describes the presence of the NaCl having Miller indices (h k l) of (200) and (220) respectively. Next in W3, the broad peak is noted between 13.83° and 20.71° which explains the amorphous property. In this also, the sharp peaks are seen at 31.74° , 45.48° , 56.51° , & 75.37° which are due to the presence of NaCl with different crystal planes. The presence of NaCl in the final sample is not desired so it is considered as an impurity. Further, it confirms that NaCl was not completely washed out by doing normal washing of silica gel for several days using distilled water as mentioned in the previous section.

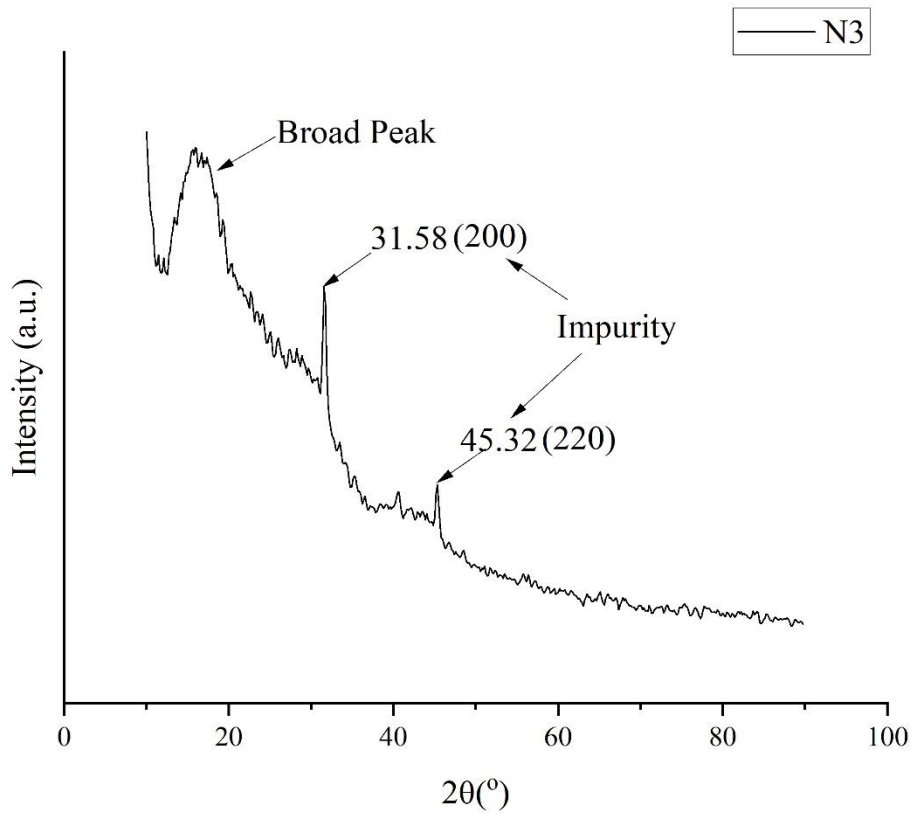


Figure 4.27: XRD of Type-2 sample N3

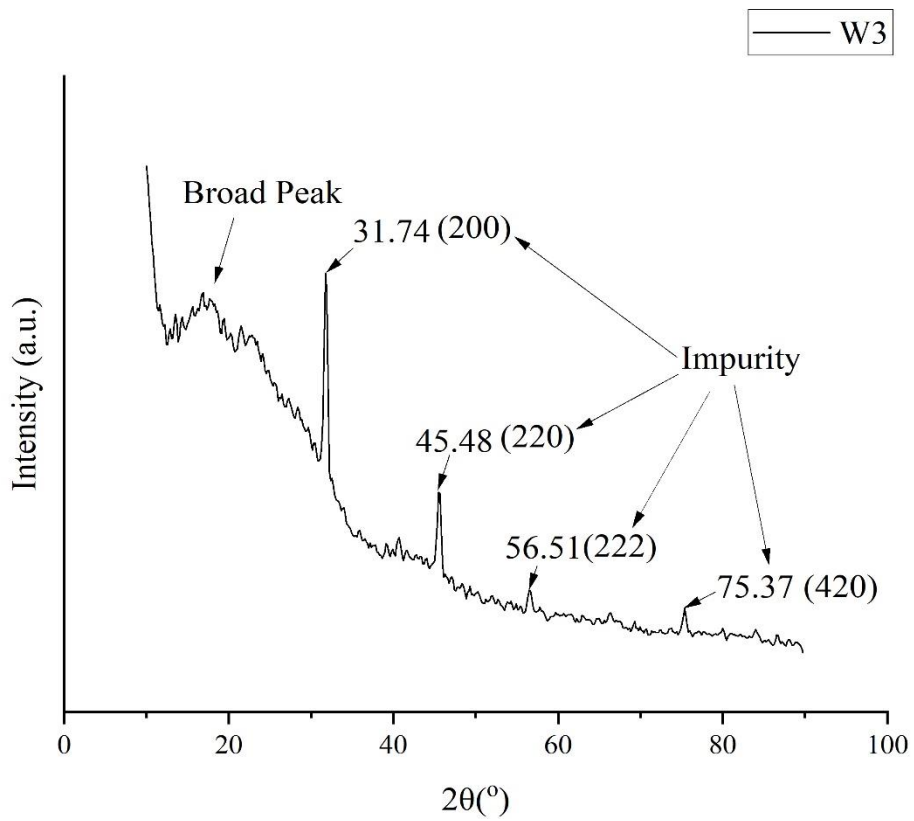


Figure 4.28: XRD of Type-2 sample W3

4.7 SEM Analysis

The SEM images are presented below to study the presence of the pore in the prepared sample. Here, the three images are shown for 2000, 5000, and 10000 times zoom with the scale of 10 μm and 1 μm of S1 and S4. In the S1 sample, the presence of the pore is not clearly seen in these images because the pore size may be smaller to be noticed under 10000 times zoom. Although, it is seen that the surface of the sample is rough which indicates the presence of a pore in it. As per previous analysis, it is found that S1 is able to repel the water and adsorb the organic liquid. But in the case of the S4 sample, the surface of the sample seems to be smooth as compared to the S1 which indicates the crystalline nature and ability to attract the water molecules. Based on this analysis, the Type-2 sample can have better porosity because it is similar to the S1 sample but has more organic liquid adsorption capacity.

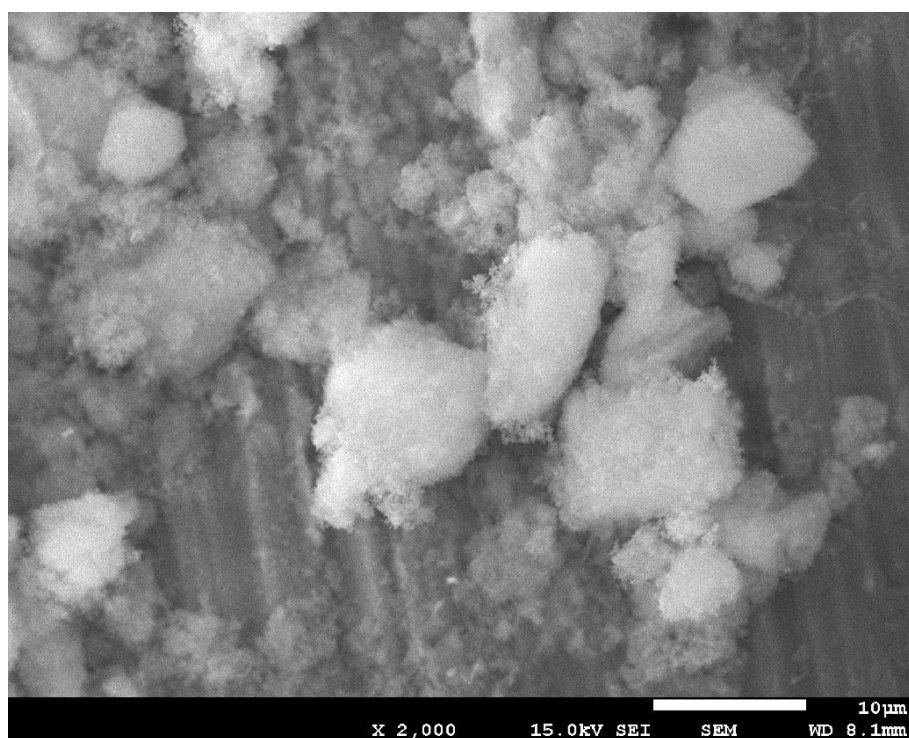


Figure 4.29: 2000 times zoom SEM image of S1

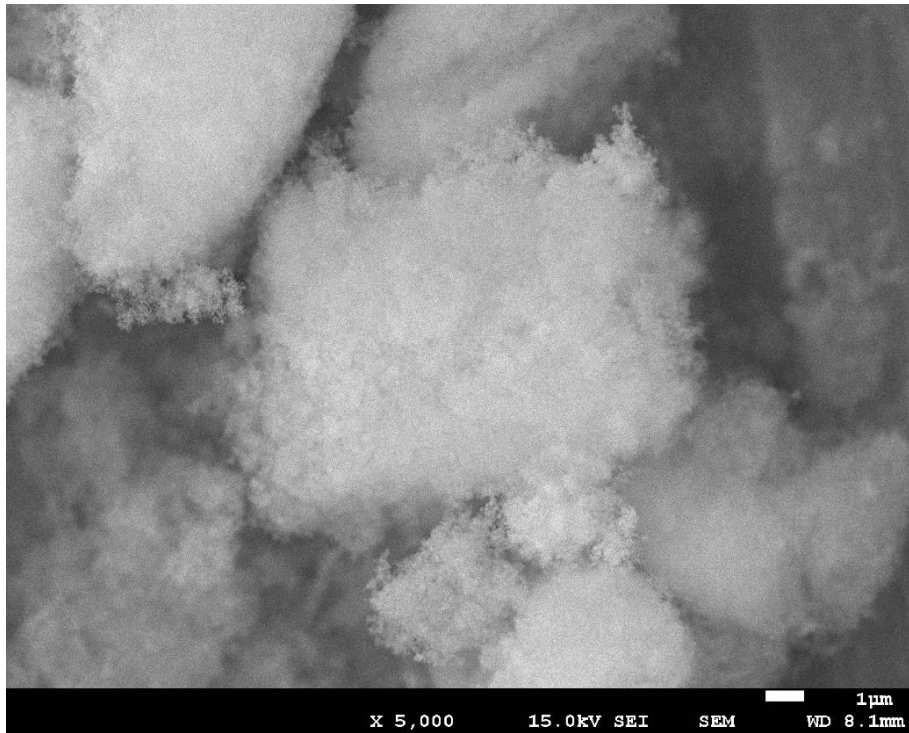


Figure 4.30: 5000 times zoom SEM image of S1

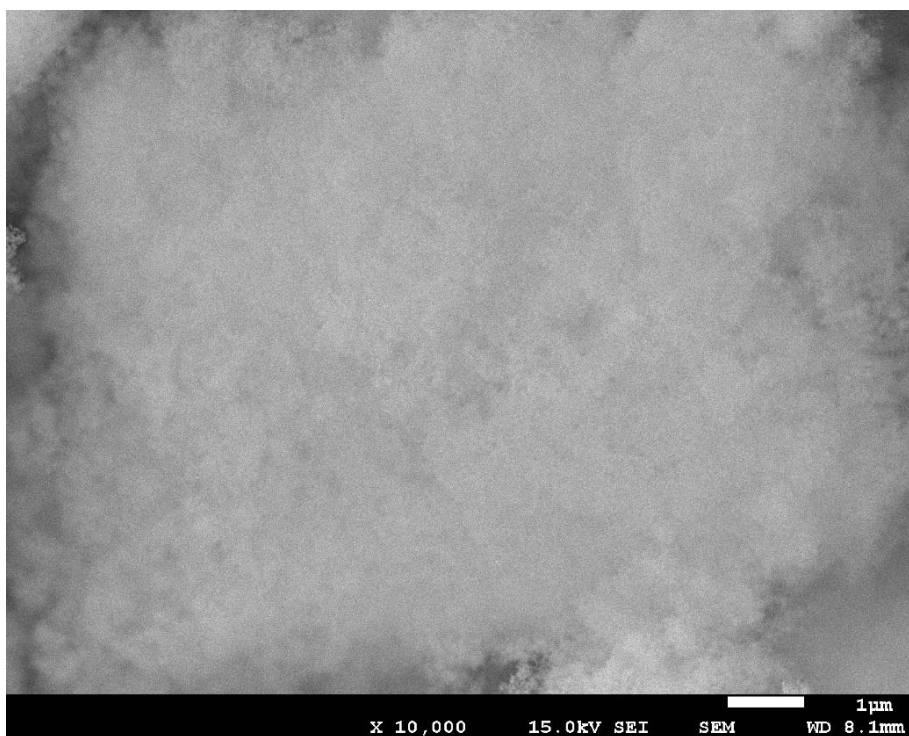


Figure 4.31: 10000 times zoom SEM image of S1

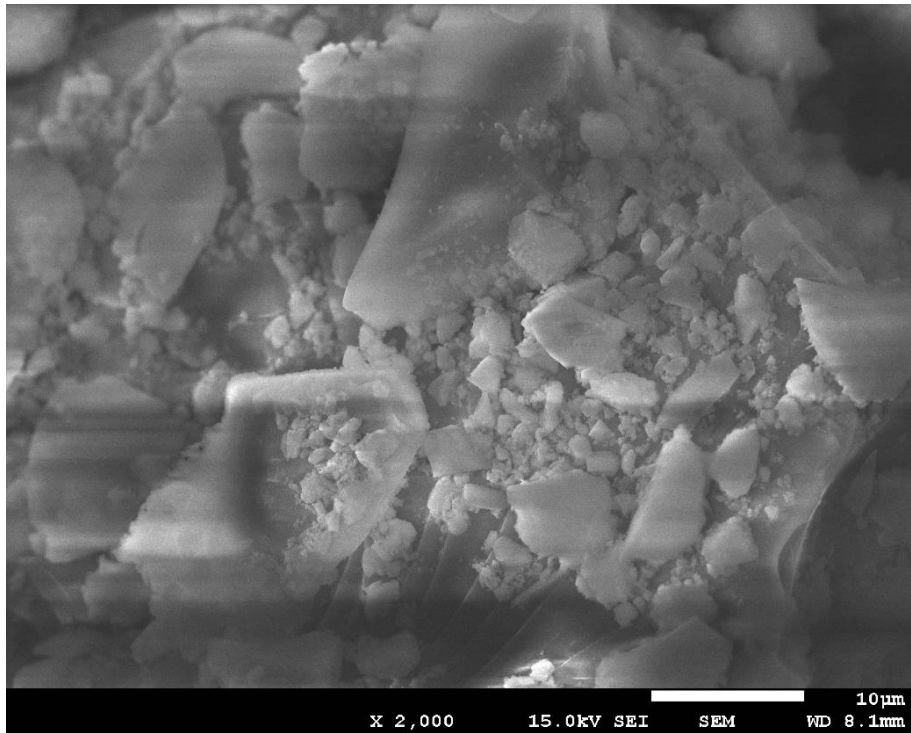


Figure 4.32: 2000 times zoom SEM image of S4

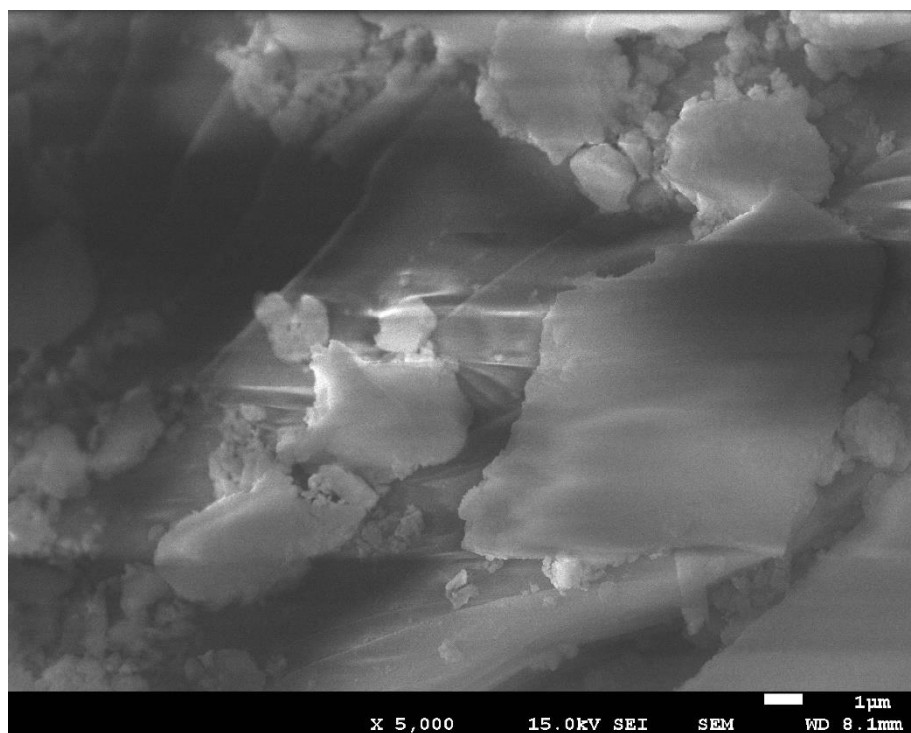


Figure 4.33: 5000 times zoom SEM image of S4

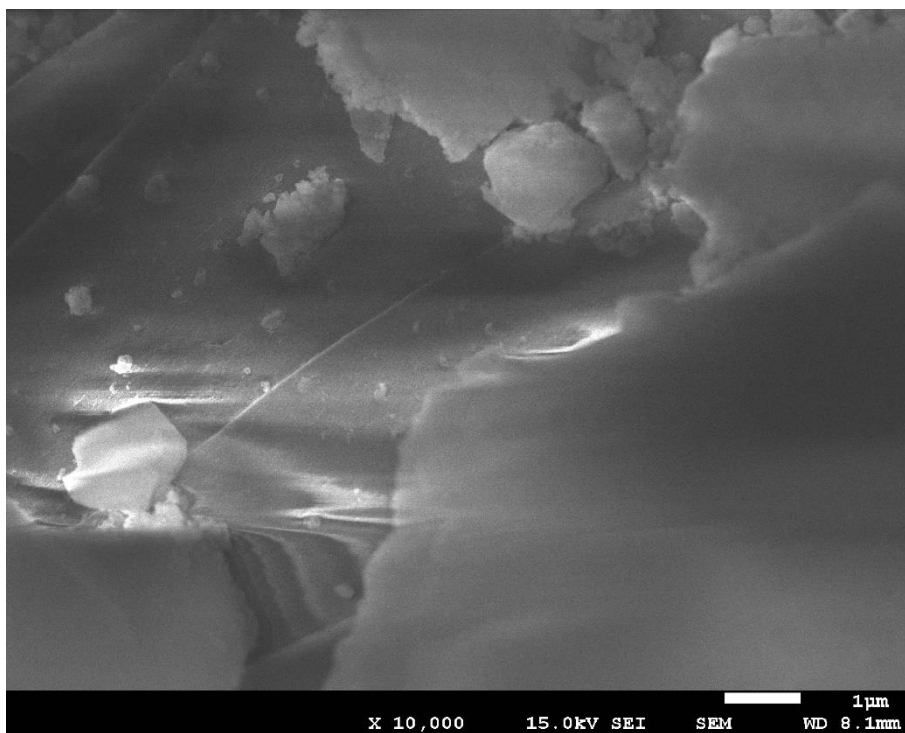


Figure 4.34: 10000 times zoom SEM image of S4

4.8 Thermal Stability Test

The thermal stability of the sample is mainly done using TG-DTA but due to unavailability, the approximate stability was performed using a box oven and tube furnace. Here, the WW2 sample was placed in a box oven for 1 hr at 200°C. N3 powder was placed in a glass tube and heated at 300°C in the presence of inert gas i.e., Nitrogen for 1 hr. After heating, the hydrophobicity of both samples was checked by placing the water droplet on them. The image of the spherical water droplets on the surface of the sample is presented below which indicates that they still possess hydrophobic nature. Based on this, the maximum heat resistance of the Type-2 sample is considered to be 300°C but the heat resistance of the sample may be more because the critical temperature at which hydrophobicity will lose is not determined.

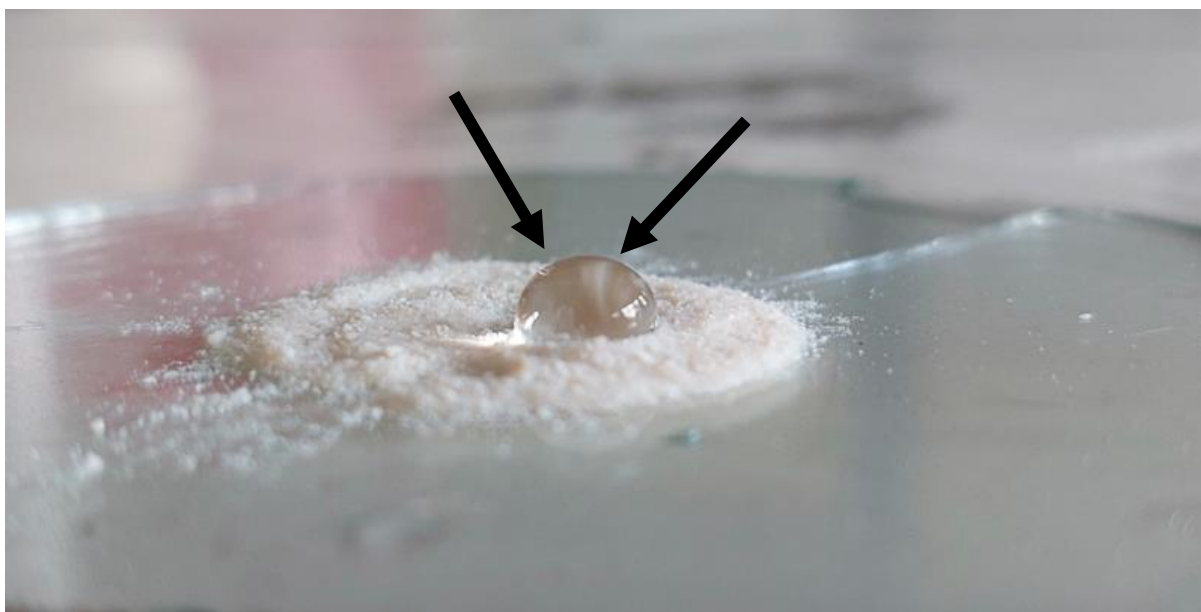


Figure 4.35: Visual inspection of N3 for hydrophobicity after heating at 300°C



Figure 4.36: Visual inspection of WW2 for hydrophobicity after heating at 200°C

4.9 Mechanical Test

Mechanical tests like flexural & tensile are not feasible for the samples prepared in this research. All Type-1 samples and two samples (N3 & N4) of Type-2 were in powder form. Further, the sample of Type-2 (W3, W4, WW1, & WW2) containing wool glass contains flaw like the silica network is not properly bonded by the glass wool as seen in the photos and the powder from the sample keeps falling while handling them. The glass wool samples already have cracks and gaps which will not allow to give the exact load at which the initial crack will be seen. Although, it is seen that the sample with glass wool was able to maintain the moulded structure as compared to the non-wool samples. So, it is clear that glass wool was able to provide the reinforcement structure upto some extent.



Figure 4.37: Presence of cracks

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The attempt of synthesizing the silica aerogel from commercially available water glass (sodium silicate) was done in this work. The silica gel was prepared in the water by following the sol-gel method. Then, the water was replaced by ethanol which gave alco-gel and the surface of the alco-gel was modified by TMCS chemical. The Type-1 (S1, S2, S3, S4, A1, A2, A3) & Type-2 (N3, N4, W3, W4, WW1, WW2) samples were made based on the modification ratio of chemicals TMCS: n-Hexane: EtOH as 4:30:1 and 20:30:10 by volume respectively. After that, the modified sample was dried in the oven at ambient pressure at different steps. The final sample was characterized by FTIR, XRD, UV-Vis spectroscopy, & SEM and further, the sample was tested for adsorption, desorption, & heat resistance properties.

- The presence of the Si-C was indicated by FTIR analysis in samples; S1, S2, S3, & A1 for Type-1 and all samples of Type-2 which confirmed that the -OH group was replaced by the -CH₃ group from TMCS during modification due to which samples were hydrophobic in nature.
- The minimum time for modification was found to be 24 hours in the air time container and modifying twice by maintaining the temperature at 50°C, also played a better role in the replacement of -OH with -CH₃ in Type-2 as compared to Type-1.
- The contact angle of the S1 and N3 sample was noted to be 124° & 146.7° respectively which indicated the samples were hydrophobic.
- The adsorption of organic liquid was greater for the N3 sample than for S1 which confirmed that the porosity of the N3 sample was better. Further, the adsorption of the organic liquid in grams per 1g sample was found to be increasing with an increase in the surface tension of the organic liquid.
- The 1g N3 was able to adsorb 4.3250g of sunflower oil from a 2:10 ratio of oil & water and 3.349g WW2 was able to remove around 15.881 mL of oil from water containing 16 mL of oil.
- Based on desorption data, the evaporation time can be reduced by increasing the temperature and after that dry sample is again available for the adsorption task.
- The sample showed low transmittance in the UV spectrum and high transmittance in the visible spectrum. S1 and N3 were found to be electrical insulators due to high band gap energy.

- The amorphous nature of the Type-2 was confirmed by XRD analysis but found NaCl as an impurity that did not wash away completely during the washing of silica gel using distilled water.
- From SEM images, the rough texture on the surface was seen in the S1 sample which indicated the porous nature. Based on the S1 SEM image, it is concluded that Type-2 samples had better porosity as confirmed by the adsorption test than Type-1.
- The sample was confirmed to resist upto a maximum temperature of 300°C examined in a tube furnace.
- The mechanical tests were not performed due to flaws in the samples as some of them were in powder form and other samples with glass wool contained cracks & gaps. But the samples with glass wool were able to hold the structure as compared to the sample without glass wool.

In conclusion, Type-2 shows properties close to silica aerogel as compared to the Type-1 sample excluding SEM and XRD analysis which were not done for both samples. Here, the main reason for inefficient silica aerogel synthesis is due to the use of commercial sodium silicate as a precursor which contains impurities. Further, the Type-2 samples had NaCl as an impurity in the final product.

5.2 Recommendations

- In the present work, commercial sodium silicate was used for making silica aerogel which contains impurities so a pure form of sodium silicate can be used for synthesis work.
- The process of replacing the water with ethanol can be done by increasing the surrounding temperature.
- The 4 mL and 20 mL volumes of TMCS were used for the modification process so the better replacement of -OH by -CH₃ can be studied by increasing its volume further.
- The mechanical test can be done after enhancing the proper bonding of glass wool with the silica network.

REFERENCES

- Abdullahi, S. S., Güner, S., Koseoglu, Y., Musa, I. M., Adamu, B. I., & Abdulhamid, M. I. (2016). Simple Method For The Determination of Band Gap of a Nanopowdered Sample Using Kubelka Munk Theory. *Journal of the Nigerian Association of Mathematical Physics*, 35, 241-246.
- Bobrowski, A., Stypuła, B., Hutera, B., Kmita, A., Drożyński, D., & Starowicz., M. (2012). FTIR Spectroscopy of Water Glass - The Binder Moulding Modified by ZnO Nanoparticles. *Metalurgija* 51, 477-480.
- Çok, S. S., Koc, F., & Gizli, N. (2021). Lightweight and Highly Hydrophobic Silica Aerogels Dried in Ambient Pressure for an Efficient Oil/Organic Solvent Adsorption. *Journal of Hazardous Materials*, 408.
- Dutrow, B. L., & Clark, C. M. (2022, May 22). *X-ray Powder Diffraction (XRD)*. Retrieved from Nanotechnology in STEM: www.serc.carleton.edu/207663
- Esteban, B., J.-R. R., Baquero, G., & Rita Puig, A. R. (2012). Characterization of the surface tension of vegetable oils to be used as fuel in diesel engines. *Fuel*, 231–238.
- Fox, P. J. (2013). *University of Delaware* . Retrieved from Index of /chem/fox/Chem333/Fall2013/Chem333Fall2013/Welcome_files: https://www1.udel.edu/chem/fox/Chem333/Fall2013/Chem333Fall2013/Welcome_files/IR%20handout.pdf
- FTIR Analysis*. (2022, May 23). Retrieved from RTI Laboratories: <https://rtilab.com/techniques/ftir-analysis/>
- Gurav, J. L., Jung, I.-K., Park, H.-H., Kang, E. S., & Nadargi, D. Y. (2010). Silica Aerogel: Synthesis and Applications. *Journal of Nanomaterials*, 1-11.
- Gurav, J. L., Rao, A. V., Rao, A. P., Nadargi, D., & Bhagat, S. (2009). Physical properties of sodium silicate based silica aerogels prepared by single step sol–gel process dried at ambient pressure. *Journal of Alloys and Compounds*, 476(1-2), 397-402.
- Hilgedick, A. (2020, February 25). *Introduction to freeze drying*. Retrieved from Labconco: <https://www.labconco.com/articles/introduction-to-freeze-drying>
- Hrubesh, L., Coronado, P., & Satcher, J. (2001). Solvent removal from water with hydrophobic aerogels. *Journal of Non-Crystalline Solids*, 328-332.

- Huang, Y., He, S., Chena, G., Daia, H., Yuana, B., Chena, X., & Yangc, X. (2019). Fast preparation of glass fiber/silica aerogel blanket in ethanol & water solvent system. *Journal of Non-Crystalline Solids*, 286-291.
- Hunt, A., & Ayers, M. (1983). *A Brief History of Silica Aerogels*. Retrieved from The Norris Research Group : <https://pamelanorris.wordpress.com/resources/history-of-silica-aerogels/>
- Jones, S. M., & Paik, J.-A. (2013). *Ambient Dried Aerogels*. NASA Tech Briefs. Retrieved from <https://ntrs.nasa.gov/citations/20140002270>
- Karamikamkar, S., Abidli, A., Behzadfar, E., Rezaei, S., Naguib, H. E., & Park, C. B. (2019). The Effect of Graphene-Nanoplatelets on Gelation and Structural Integrity of a Polyvinyltrimethoxysilane-Based Aerogel. *The Royal Society of Chemistry 2019*, 9(20), 11503–11520.
- Khedkar, M. V., Somvanshi, S. B., Humbe, A. V., & Jadhav, K. (2019). Surface Modified Sodium Silicate Based Superhydrophobic Silica Aerogels Prepared Via Ambient Pressure Drying Process. *Journal of Non-Crystalline Solids*, 411, 140-146.
- Kistler, S. S. (1931). Coherent Expanded-Aerogels. *The Journal of Physical Chemistry*, 52–64.
- Launer, P. J., & Arkles, B. (2013). Infrared Analysis of Organosilicon Compounds. *Silicon Compounds: Silanes & Silicones*, 175-178.
- Lebedev, A., Suslova, E., Troyankin, A., & Lovskaya, D. (2021). Investigation of Aerogel Production Processes: Solvent Exchange under High Pressure Combined with Supercritical Drying in One Apparatus. *Gels*.
- Lee, M., Nam, K. S., & Hwang, H. (2021). Thermal Gelation for Synthesis of Surface-Modified Silica Aerogel Powders. *Gel*, 7(4), 1-10.
- Maleki, F. H., Nemati, A., & Joya, Y. F. (2015). Synthesis of C–N–Y Tri-Doped TiO₂ Photo-Catalyst for MO Degradation. *Materials Research Express*, 2(10), 1-12.
- Maleki, H., Duraes, L., & Portugal, A. (2014). An overview on silica aerogels synthesis and different mechanical reinforcing strategies. *Journal of Non-Crystalline Solids*, 55-74.

- Manzocco, L., Mikkonen, K. S., & García-González, C. A. (2021). Aerogels as Porous Structures for Food Applications: Smart Ingredients and Novel Packaging Materials. *Food Structure*, 28(11), 1-11.
- Mendes, A. L., Pontinha, A. D., Santos, P., & Durães, L. (2022). Aerogel Composites Produced from Silica and Recycled Rubber. *Materials*, 1-14.
- Motahari, S., & Abol, A. (2015). Silica Aerogel–Glass Fiber Composites As Fire Shield for Steel Frame Structures. *Journal of Materials in Civil Engineering*, 27(10), 04015008 (1-7).
- Olalekan, A. P., Dada, A. O., & Adesina, O. A. (2014). Review: Silica Aerogel as a Viable Absorbent for Oil Spill Remediation. *Journal of Encapsulation and Adsorption Sciences*, 122-131.
- Perdigoto, M., Martins, R. C., Rocha, N., & Quina, M. J. (2012). Application of hydrophobic silica based aerogels and xerogels for removal of toxic organic compounds from aqueous solutions. *Journal of Colloid and Interface Science*, 134-40.
- Pierre, A. C., & Rigacci, A. (2011). SiO₂ Aerogels. In M. A. Aegerter, & M. Prassas, *Advances in Sol-Gel Derived Materials and Technologies book series* (pp. 21-44). Springer.
- Rao, A. V., Hegde, N. D., & Hirashima, H. (2007). Absorption and Desorption of Organic Liquids in Elastic Superhydrophobic Silica Aerogels. *Journal of Colloid and Interface Science*, 305(1), 124–132.
- Rapier, R. (2020, June 25). *Fossil Fuels Still Supply 84 Percent Of World Energy — And Other Eye Openers From BP's Annual Review*. Retrieved from EnergyNow.ca: <https://energynow.ca/2020/06/fossil-fuels-still-supply-84-percent-of-world-energy-and-other-eye-openers-from-bps-annual-review/>
- Salimian, S., & Zadhoush, A. (2019). Water-Glass Based Silica Aerogel: Unique Nanostructured Filler for Epoxy Nanocomposites. *Journal of Porous Materials*, 26(8), 1-11.
- Shao, Z., He, X., Niu, Z., Huang, T., Cheng, X., & Zhang, Y. (2015). Ambient Pressure Dried Shape-Controllable Sodium Silicate Based Composite Silica Aerogel Monoliths. *Materials Chemistry and Physics*, 1-8.

- Shi, H.-x., Cui, J.-t., Shen, H.-m., & Wu, H.-k. (2014). Preparation of Silica Aerogel and Its Adsorption Performance to Organic Molecule. *Advances in Materials Science and Engineering*, 2014(1), 1-8.
- Slosarczyk, A. (2021). Carbon Fiber—Silica Aerogel Composite with Enhanced Structural and Mechanical Properties Based on Water Glass and Ambient Pressure Drying. *Nanomaterials*, 1-14.
- Steiner, S. (2022, May 22). *About Aerogel*. Retrieved from Aerogel.org: <http://www.aerogel.org/?cat=21>
- Thomas, G. (2012, August 22). *What is Aerogel? Theory, Properties and Applications*. Retrieved from AZO Materials : <https://www.azom.com/article.aspx?ArticleID=6499>
- Wang, D., McLaughlin, E., Pfeffer, R., & Lin, Y. (2012). Adsorption of oils from pure liquid and oil–water emulsion on hydrophobic silica aerogels. *Separation and Purification Technology*, 28-35.
- Werfa, I. v., Palmisano, F., Leob, R. D., & Marrone, S. (2008). Chemical Analyses of Silicon Aerogel Samples. *Nuclear Experiment (nucl-ex)*, 1-11. <https://doi.org/10.2172/955880>
- Woignier, T., Primera, J., Alaoui, A., Despetis, F., Etienne-Calas, S., Faivre, A., . . . Etienne, P. (2020). Techniques for characterizing the mechanical properties of aerogels. *Journal of Sol-Gel Science and Technology*, 6-27.
- Yi, Z., Tang, Q., Jiang, T., & Cheng, Y. (2019). Adsorption Performance of Hydrophobic/Hydrophilic Silica Aerogel for Low Concentration Organic Pollutant In Aqueous Solution. *Nanotechnol Reviews*, 8(1), 266–274.
- Zhou, T., Cheng, X., Pan, Y., & Congcong. (2018). Mechanical Performance and Thermal Stability of Glass Fiber Reinforced Silica Aerogel Composites Based on Co-Precursor Method By Freeze Drying. *Applied Surface Science*, 437, 321-328.
- Zimnoch, L. B. (2015). Fourier Transform Infrared and Raman Characterization of Silica-Based Materials. In M. T. Stauffer, *Applications of Molecular Spectroscopy to Current Research in the Chemical and Biological Sciences*. IntechOpen.

APPENDIX

1. Preparation of 1 M HCl Solution

- Ass% = 37%
- Specific density = 1.182
- Molecular weight (MW) = 36.46
- Concentration (C1) = (Ass% × Sp. density × 10) / MW = (37 × 1.182 × 10) / 36.46 = 11.995 ≈ 12M
- Initial concentration (C1) of HCl = 12 M
- Initial volume of HCl (V1) = X
- Final concentration (C2) = 1 M
- Final volume (V2) = 250 mL
- So, V1 = (C2 × V2) / C1 = (1 × 250) / 12 = 20.83 mL

2. TMCS Grade Certification



河北冠朗生物科技有限公司
Hebei Guanlang Biotechnology Co., Ltd.
Add: No.108 Zhongshan West Road, Qiaoxi District, Shijiazhuang, Hebei, China
TEL: 0311-66562153 Fax: 0311-66562153

CERTIFICATE OF ANALYSIS

Product Name	Chlorotrimethylsilane	CAS	75-77-4
Lot Number	20220205-1	Quantity	1000kg

Items	Standard	Results
Appearance	Colorless liquid	Colorless liquid
Trimethyl monochlorosilane %	≥99.5	99.53
Methyltrichlorosilane %	≤0.10	0.035
Silicon tetrachloride %	≤0.05	0.00
High-boiling residues %	≤0.20	0.13



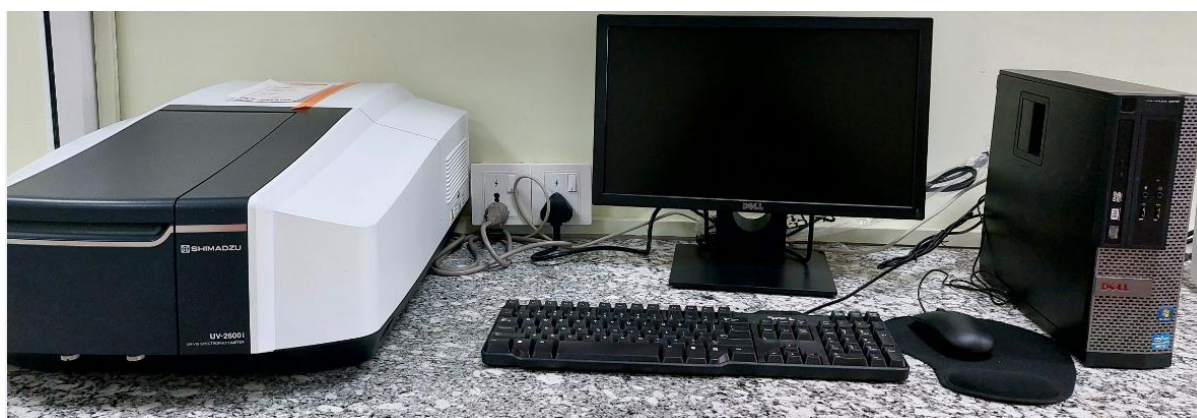
Q/A Manager: Li Meng

Checked by: Quan Mi

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3. Instruments



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FTIR Spectrophotometer



Tube furnace

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