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Spectral and Molecular Modifications of Hydrophilic Silica Aerogel: A Study on Doping Effects and Structural Evolution

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Abstract. Silica gel has recently become an interesting material because of its importance in many applications in several fields such as medicine and sustainable technology. It has been found that the impregnation of some materials improves the synthetic properties of silica. Silica gel was prepared via mixed TEOS: ethanol: hydrochloric acid with molar ratios of 2:10:1.5. After doping with Diethyl in different concentrations 10^{-4}, 10^{-5}, and 10^{-6}) g/cm^3, the molecular properties were studied by examining the FTIR, and the UV spectrum of the prepared samples. The synthetic and morphological properties were investigated through BET and SEM and EDX. Results showed the ultra-violet to the visible (green) region at the concentration (10^{-5}) g\cm^3, and doping was evident through the change in the absorption peaks for different concentrations. The FTIR spectrum remained unchanged, as well, the high specific surface area indicates the inner connection of the dye to the silica network, which makes it a promising material in improving the structural properties for industrial applications. Materials with a limited variety of homogeneous microporous materials except for some aggregation appear at a high concentration (10^{-4}) g/cm^3 due to diethyl particles.

Keywords: Porous Nanomaterials, Spectroscopy, Surface Modification, FTIR spectrum, Silica aerogel, Diethyl

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1. Introduction

Nanoparticles (NPs) are extremely small material with size ranging from 10 to 100 nm. It can be divided into many modules according to their characteristics, sizes, or forms[1-3]. NPs are becoming more and more important in technological progress because they can change their physical and chemical properties. These properties include better performance than their bulk equivalents in terms of melting point, wettability, electrical and thermal conductivity, catalytic activity, light absorption, and scattering

[4-6]. The advantage of nano porosity over normal porosity is its highly specific surface area. So, most researchers work on improving the structural properties of materials without compromising the surface area, which has the greatest impact on industrial and technological applications. [7, 8]. In their very porous nanostructure, aerogels are materials that are super critically dried from sol-gel and have unique characteristics, especially an excellent insulating technique [9-11]. There are various methods for preparing the aerogel, one of which is the supercritical drying process. Due to its exceptional efficacy in producing the greatest possible product qualities, this specific procedure was selected [12-14]. When supercritical drying is used, shrinkage is prevented, and the resulting aerogel's three-dimensional structure is maintained. The hydrophilic aerogel's pores instantly fill with water when it comes into contact with it, losing its structural integrity (the aerogel effect) [15]. The silanol groups on the surface or the method of synthesis are responsible for the hydrophobicity or hydrophilicity of silica aerogels [16, 17]. So that, Ashraf M. Alattar et al created composite nanoparticles of aerogel silica utilizing the doped lanthanide NaYF4 as the nanoparticle material and next examined the properties of this compound following gelation and supercritical drying to demonstrate how processing affects the final result. By attempting to create composites of silica aerogel and nanoparticles [18] . Taylor & Francis et al.'s technique showed that the use of hydrophilic silica aerogel for adsorption is a reasonably inexpensive method of extracting chemotherapeutic medicines from wastewater since it requires comparatively less expensive precursors [19]. Sekai Zong and others, proved an easy and inexpensive way to make transparent silica aerogel beads that look like macroscopic beads. These beads can be utilized as adsorbents, catalysts, or as a basis for creating new functional materials with desirable properties like high surface area, effective mass transport, and high transmittance [20]. Most studies did not shed light on the effect of doping or its use in improving the structural properties to increase the application area of silicon clusters. In this work, a nanomaterial of hydrophilic silica-monolith was prepared via super-critical drying technique and doped with Diethyl, the molecular and structural properties of the resulting material were investigated.

2. **Experimental procedures**

2.1. Materials

The aerogel was synthesized using a variety of chemical materials, including tetraethyl orthosilicate (TEOS) with a purity level of over 98 %, spectroscopic grade ethyl alcohol with a purity level of over 99 %, N, N-dimethylformamide with a purity level of over 98 %, and deionized water catalyzed by ammonium fluoride with a purity level of over 97 %. Diethyl($C_2H_5OC_2H_5$) from Sigma-Aldrich.

2.2. The Procedures

One-step technique (acid catalysis) was synthesized to produce silica gels, in order to obtain a more stable product and to reduce the leakage of the dye during repeated washing, in the event of resorting to preparation with ambient pressure. Chemical components used were TEOS, ethanol, distal-water, and hydrochloric acid with molar ratios of 2:10:1.5: N, where N was adjusted to get a final sol with a pH < 2. The sols were mixed with Diethyl($C_2H_5OC_2H_5$) Next, 2ml of C_3H_7NO was presented as a drying control chemical additives and the mixture was stirred for a further hour. The resulting mixture forms a gel, which is then placed in plastic tube. The gel is left to age at room temperature for 48 hours. To obtain 100% purred gel, it washed in Ethanol five timed during 24 hours, the gel must undergo a washing process to eliminate any remaining unreacted monomers from the networks.

The gel was exposed to supercritical drying in a specially engineered reactor that could endure high pressures, namely at a pressure range of 1100-1200 psi and a temperature of 455 C, for a duration of 5 hours. Supercritical drying prevents cracking and gives a product with excellent specifications in terms of low density and high surface area and ensures the adhesion of the pigment within the silica network without losing it as a result of repeated washing in the case of drying under normal atmospheric pressure. It is essential to achieve optimal mixing between supercritical CO_2 and the solvent present in the gel's pores throughout this procedure. The drying process of $SCCO_2$ is nearing completion, reaching its last stage of densification. This was achieved by subjecting the material to four different temperatures: room temperature, 550°C, 650°C, and 850°C.

2.3. Characterization

To investigate the effect of different concentration of Diethyl, the transmittance of the aerogel sample was measured using a UV-VIS spectrophotometer (Ultrospec. 4300 pro). The Nicolet Is50 FT-IR spectrophotometer was utilized to get high spectral resolution data for aerogel samples in the spectral range of 400 to 4000 cm^{-1} . To study the molecular properties and the nature of the bonds of silica samples when doped with different concentrations. As is known, this examination is considered a fingerprint of the molecules when infrared rays are directed, which leads to vibration in the vibrational levels of the molecules. Pore size distribution, pore volume, and specific surface area were measured using the Brunauer–Emmitt–Teller (BET) with a micromeritics ASAP 2020 instrument. This test is important to study the structural and compositional properties of samples, where nitrogen gas is pumped at a temperature of 195.800°C under Low Pressure Dose: 5.000 cm^3/g STP and upon reaching the saturation state, it is discharged automatically, which leads to the appearance of the hysteresis loop which have four classifications H1 for the materials that have a limited variety of homogeneous mesopores H2 for pore blocking-percolation in a limited range of pore necks, H3 for macropores that aren't entirely filled with pore condensate and H4 micropore filling, there is a greater absorption at low p/p_0 [21]. Through the shape of hysteresis loop it can recognize the type of pores that belong to the silica structures[22, 23]. The morphology and microstructures of the silica aerogel specimens were examined using a scanning electron microscope (SEM, ULTRA 60) in secondary electron mode.

3. Results and Discussion

3.1. UV- Vis and FTIR analysis

Figure (1) show the absorption spectrum for aerogel doped with Diethyl in different concentrations (10^{-4}, 10^{-5}, and 10^{-6}) g/cm^3

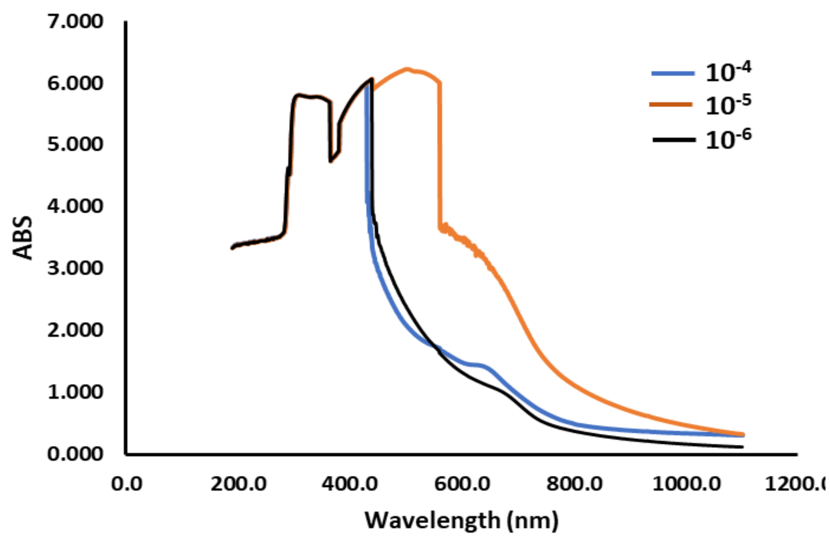


Figure 1. Absorption spectrum for silica aerogel doped with Diethyl in different concentrations.

The maximum absorption peaks with different concentrations at the wavelengths between 300 – 600 nm which indicate the Ultra-Violet to Visible (green) region at the concentration (10^{-5}) g/cm^3. In cases of ordinary silica (without doping), the absorbance falls in the ultraviolet region [[24, 25], but doping was evident through the change in the absorption peaks for different concentrations in fact diethyl ether does not have a UV spectrum because of π* molecular orbital does not exist because there are no π-bonded electrons in diethyl [26] , this leads us to the fact that mixing Diethyl with silica enables to obtain a material with different properties that can benefit from for practical purposes. FTIR spectra for silica aerogel in different concentrations are depicted in figure (2)

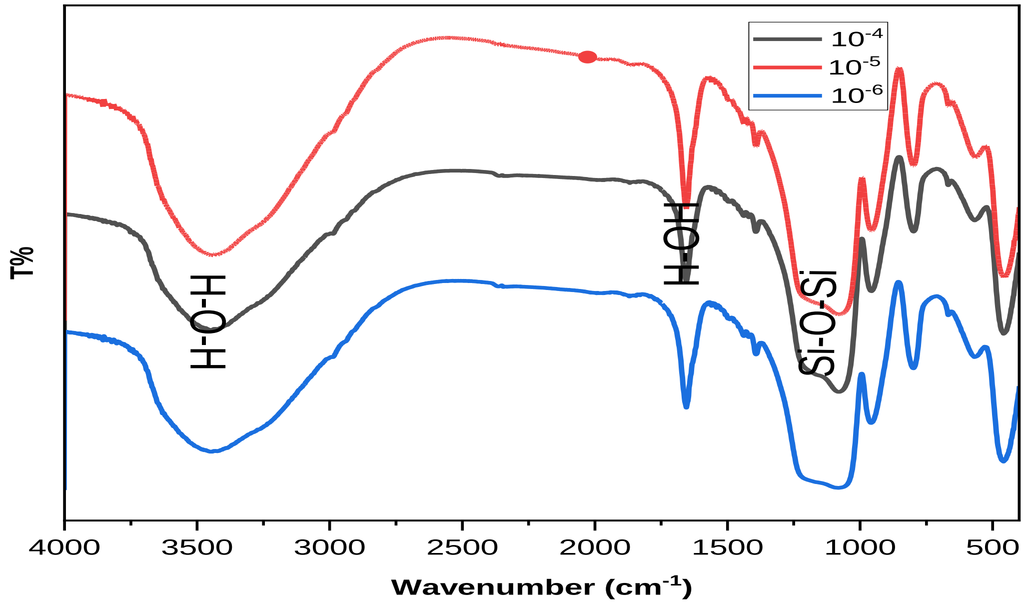


Figure 2. FTIR spectrum for silica aerogel doped with Diethyl in different concentrations.

The broadband at 3445-3448 cm^{-1} refer to O-H band which confirm the hydrophilicity property because the O-H band in hydrophobic silica is very weak or sometimes disappear in case of superhydrophobic property[27, 28] because of modification prosses which leads to dismantling the H-O-H bands. While in case of hydrophilic structure there are large quantities of water in hydrogel. The broad band in region 1072- 1228 cm^{-1} it indicates the presence of large amounts of silica in the samples; as the bonds go back to the silicon oxide network, besides at 1654 cm^{-1}. The band between 954-958 cm^{-1} return to C-O stretching [29] while at 798-800 cm^{-1} refer to C–H out-of-plane [2, 30]. Upon comparing all samples at varying concentrations, we discovered that the FTIR spectrum remained unchanged which instead provides fingerprints to the molecular vibrational modes.
This is because an increase or decrease in Diethyl does not alter this spectrum. The main reason for the dye not appearing in the IR spectrum is due to its complete dissociation and integration with the silica network this is agreement with *Das.* [31, 32] .

3.2. BET Analysis

Figure (3) shown the hydrogen adsorption- desorption isotherm and average pore volume distribution (BJH-Plot) under Low Pressure Dose: 5.000 cm³/g STP, Analysis Bath Temp.: -195.800 °C, the Sample Density: 1.000 g/cm³, mass 0.0987 g, and different concentrations. From figures it clear that the type of hysteresis loop is H1 which occurs in materials that have a limited variety of homogeneous microporous, this classified is depended by the International Union of Pure and Applied Chemistry (IUPAC) [21, 33]. Besides this type of hysteric loop was found in materials which display a narrow range of uniform mesopores, as for instance in templated silicas, also it epitomizes cylindrical pore in which both end is open and capillary in the middle of the qualified pressure. The maximum pore size is categorized as Mesopores, characterized by pore diameters mostly below 50 nm, interspersed with occasional Micropores measuring less than 2.0 nm. Mesopores are often found in materials like silica gels. [34, 35]. Additionally, the variation of pore size, pore volume, particle size, and surface area confirmed that the doping the internal structure of the silica network was penetrated as evident from the decrease in pore volume, which led to a decrease in the surface area with increasing concentration within the nanoscale limits of the particles, as shown in the table (1).

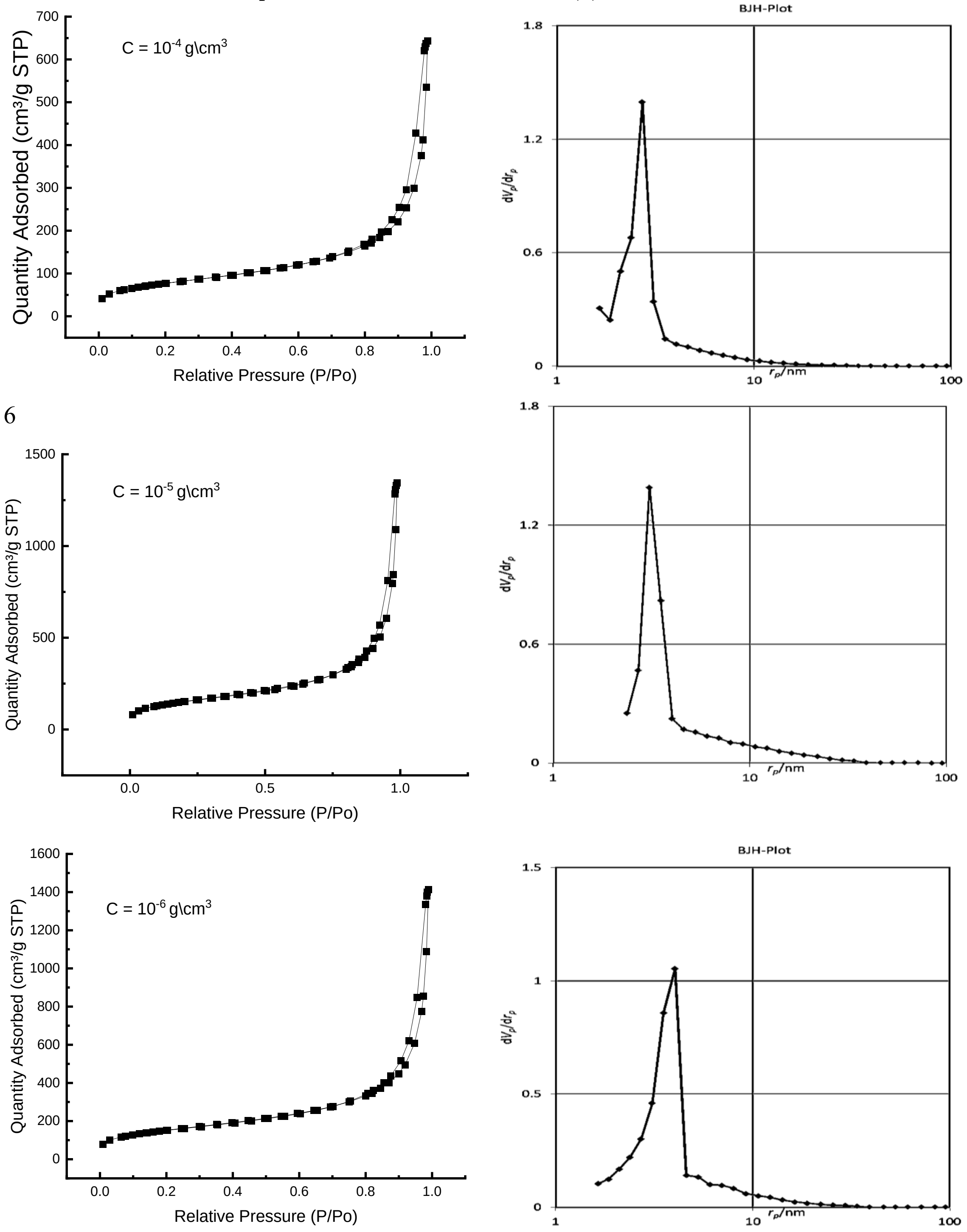


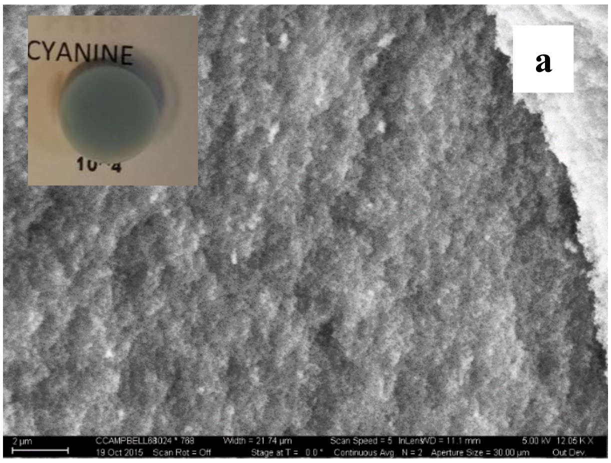
Figure 3. Hydrogen adsorption- desorption isotherm and average pore volume distribution for silica aerogel doped with Diethyl in different concentrations

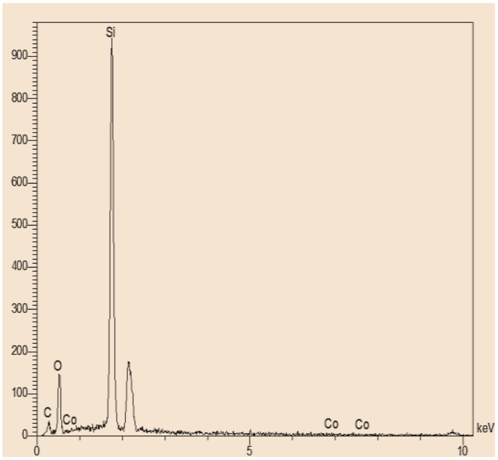
Table 1. Summarizes the nitrogen adsorption measurements and density for the silica aerogel doped with Diethyl in different concentrations

Con. (g/cm^3)	surface area (m^2/g)	pore size (nm)	pore volume (cm^3/g)	pore radius (nm)	Particle size (nm)
10^{-4}	888.31	8.911	3.100	2.02	33.09
10^{-5}	800.9	7.054	2.591	1.85	42.11
10^{-6}	769.55	6.119	2.222	1.40	48.04

3.3. Scanning electron microscopy (SEM)

The morphological property and the nature of surface for all microporous samples are represented by scanning electron microscopy are illustrated in figure (4) , where the homogenous structure and soft surface are domain in most regions although the variation in concentration the image of SEM can describe the figure at 10^{-6} g/cm^3 and 10^{-5} g/cm^3 as resembles grains of sand scattered on a smooth surface, except some aggregation seems at high concentration 10^{-4} g/cm^3 because of Diethyl particles are more so the surface of the sample appears undulating, These results are consistent with most researchers who prepare a homogeneous structure, and there is no agglomeration in the silica aerogel even when doped with dyes. All these changes are evident in the main component diagram of the samples in the DLS test as shown in figure (4). And percentage of the atomic and weight of C, O, CO and Si atoms is shown in table (2). The larger rate was return to Si atoms; because of its high quantity in the basic material is TEOS and the high content of the O element is attributed to adsorbed oxygen species present on the nano surface material.





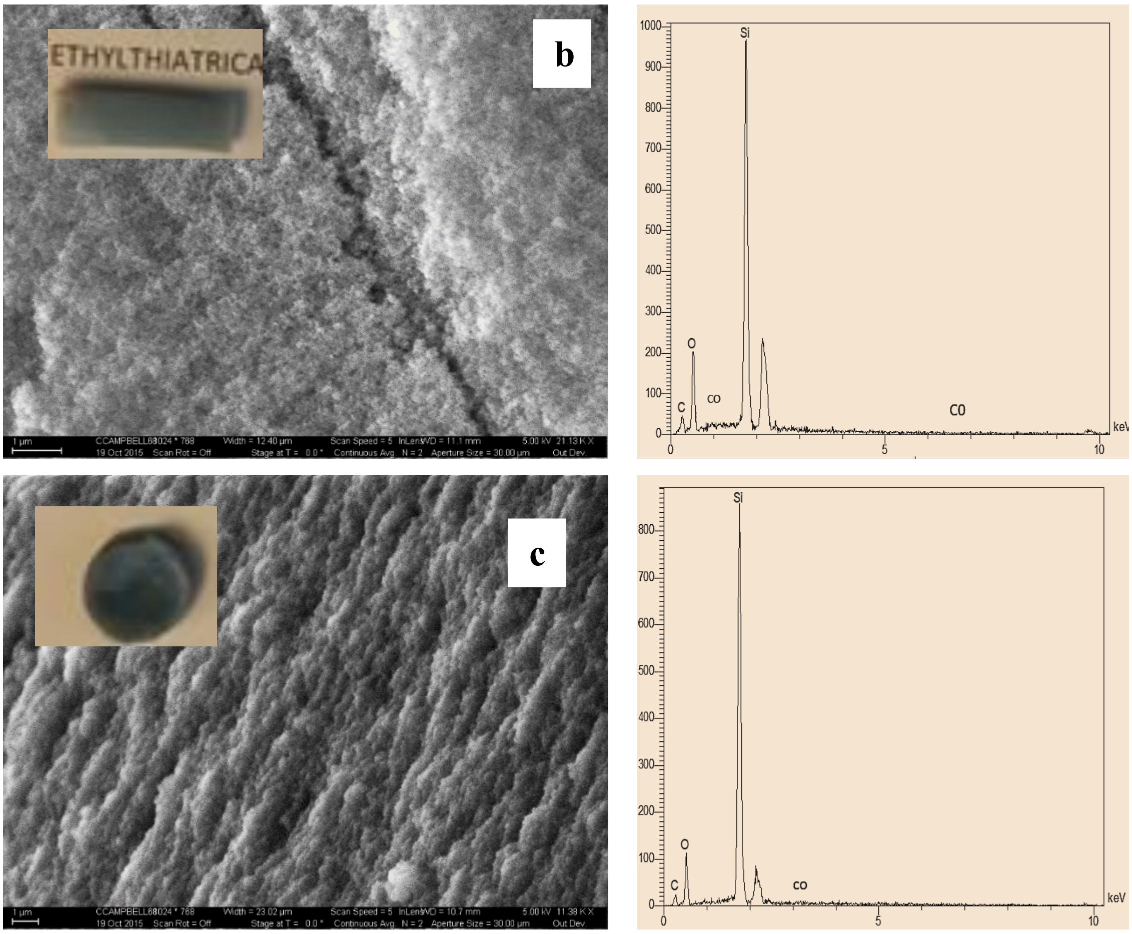


Figure 5. SEM images and DLS test for silica aerogel doped with Diethyl with concentrations (a: 10^{-6}), (b: 10^{-5}), and (c: 10^{-4}) g/cm^3.

Table 2. The elemental analysis of the silica aerogel

Element	weight %	Rate in sample%
Si	41.76	56.52
O	20.77	23.58
C	16.88	19.07
CO	11.5	14.11

4. Discussions

1. The maximum absorption peaks with different concentrations at the wavelengths between 300 – 600 nm which indicate the Ultra-Violet to Visible (green) region at the concentration (10^{-5}) g\cm^3.

2- The weak band appears, indicating that the OH groups are disappearing; as a result of modification prosses. And the FTIR spectrum remained unchanged. This is because an increase or decrease in Diethyl does not alter this spectrum.

3- hysteresis loop type is H1 which occurs in materials that have a limited variety of homogeneous microporous, besides this type found in materials which display a narrow range of uniform mesopores, as for instance in templated silicas, also it epitomizes cylindrical pore in which both end is open and capillary in the middle of the qualified pressure. The maximum pore size is categorized as Mesopores.

4- The variation of pore size, pore volume, particle size, and surface area confirmed that the doping the internal structure of the silica network was penetrated as evident from the decrease in pore volume, which led to a decrease in the surface area with increasing concentration within the nanoscale limits of the particles.
5- The homogenous structure and soft surface are domain in most regions although the variation in concentration.

5. Conclusion

In the ultra-violet to visible (green) region at the concentration of 10^{-5}g/cm^3, doping was evident through the change in the absorption peaks for different concentrations this means that mixing Diethyl with silica enables to obtain a material with different properties that can advantage from for practical purposes in many applications. The FTIR spectrum remained unchanged. This is because an increase or decrease in diethyl does not adjust this spectrum, which instead provides fingerprints for the molecular vibrational modes; with comparing with another researcher, it found the dye not appearing in the IR spectrum is due to its complete dissociation and integration with the silica network. Materials with a limited variety of homogeneous microporous materials, such as templated silicas, exhibit the H1 type of hysteresis loop. The good results after doped with Diethyl such as pore size 8.911nm, pore volume 3.100 cm3/g, particle size 33.09 nm, and high surface area 888.31g/cm^2 confirmed that the doping the internal structure of the silica network was penetrated as evident from the decrease in pore volume, which led to a decrease in the surface area with increasing concentration. It also signifies cylindrical pores, where both ends are open and capillary reduction occurs in the middle of the qualified pressure, with the homogenous structure and soft surface dominating in most regions. However, the variation in concentration, except for some aggregation, appears at a high concentration 10^{-4}g/cm^3 due to diethyl particles, resulting in an undulating surface. This makes doping with Diethyl alkyl silica a better choice for industrial applications, such as self-cleaning or anti-fog windows. We suggest preparing Diethyl-doped silica from less expensive precursors, such as sodium silicate, or from natural materials, such as rice husks, and comparing this with what we have obtained in this paper.

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