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Effects of Cs-137 radiation on hydrophobic silica monolithic prepared by two precursors

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Article Info.	Abstract
<p><i>Article history:</i></p> <p>Received 06 February 2024</p> <p>Accepted 04 March 2024</p> <p>Publishing 30 June 2024</p>	<p>Cesium (Cs-137) radiation energy was shined on two aerogel samples made from two different starting materials at room temperature and pressure three times a day for one hour, two hours, and twenty-four hours. G1 and G2 were used as silicic acid sources to fabricate the two different types of aerogel samples. G1 represents tetraethyl orthosilicate (TEOS), while G2 represents sodium silicate (Na₂SiO₃). Field emission scanning electron microscopy analysis revealed the morphological characteristics of the molecular bonds and the impact of irradiation on them. Fourier-transform infrared spectroscopy investigated the molecular bonds and their characterization before and after exposure to radiation. The samples made from TEOS at room temperature and pressure were not affected by radiation in either the FTIR or FESEM tests. However, the samples made from Na₂SiO₃ at room temperature and pressure showed that some of the peaks were disappearing because they were exposed to radiation. Also, the peaks of the CH₃ functional group are associated with the hydrophobicity of the material. The homogeneity and uniformity for all samples are dominant, and the size of the colloidal particles is about 30-33 nanometers (nanometer structure), which is no different from the size of the particles before and after exposure to gamma rays.</p>
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1. Introduction

Aerogels, as referred to by many researchers, are porous (typically mesoporous) materials that have an amorphous structure with an irregular shape and significant characterizations like low density, high porosity, a large specific surface area, and a surface chemistry that is adaptable [1-3]. The first preparation was done by Kistler in 1931. Silica aerogels are the most common type of aerogels, which have a very low density ($\sim 0.003 \text{ g/cm}^3$) and also high thermal insulation (0.005 W/m K), in addition to many other characteristics indicated by many previous studies [4, 5]. Moreover, it can be manufactured in an easy way. Aerogels can be hydrophilic or hydrophobic, depending on how they interact with water. Silica aerogel has been found to be hydrophilic (unmodified) when it absorbs water $\sim 4-5$ times its weight over a time period of 5 min. Hydrophobic (modified) aerogels absorb $\leq 2\%$ of their weight in water even after soaking in water for 1 year. Hence, they have different properties and applications [6-8].

Silica aerogel can be prepared from different precursors. The precursors of silica, also known as silicon dioxide (SiO₂), can be various compounds that contain silicon and oxygen. Some commonly used precursors for synthesizing silica include tetraethyl orthosilicate (TEOS), tetramethyl orthosilicate (TMOS), sodium silicate (Na₂SiO₃), polyethoxydisiloxane (PEDS), and silicon tetrachloride (SiCl₄). The most commonly used precursor for silica aerogel is tetraethyl orthosilicate (TEOS), and the cheaper precursor is sodium silicate, which we used as the precursor for the two prepared samples in this study. It was found that the monolithicity of the aerogels was dependent on the type of catalyst used for each precursor. Different precursors lead to variations in the aerogel's surface chemistry, microstructure, pore size distribution, thermal conductivities, and overall performance [9-11].

Both thermal and nuclear radiation are forms of energy transfer, and both can interact with matter and cause changes in matter. Since the material was known for its ability to provide thermal insulation [12], research and tests were also conducted to monitor the changes that could occur on the aerogel when exposed to nuclear radiation, but this topic was not addressed in a wide and detailed manner. Cesium, a highly reactive alkali metal, exists in two radioactive isotopes, Cs-134 and Cs-137, both emitting gamma and beta rays, posing radiation risks [13, 14]. The effect of this radiation on various materials, including aerogels, has been studied. In 1993, Hotaling S. P. published a report presenting new results for aerogels used as ultralight substrates for reflective coatings, in which the effects of ionizing radiation upon the aerogel heterostructures were found to be minimal up to a threshold of 35 mrad gamma ray irradiation, and an exponential relationship was found to predict the effects of radiation at high doses of gamma rays [15]. Also in 1996, S.K. Sahu, M.Z. Wang et al. assessed the harm caused by radiation to silica aerogel and found that silica aerogel with a small index of refraction ($n = 1.012-1.028$) was discovered to be radiation-resistant, at least against 9.8 Mrad of gamma radiation. Furthermore, they observed no variations in transparency or refractive index following the irradiation within the measurement error. Thus, we can employ silica aerogel without worrying about radiological harm in high-radiation situations such as B factories and space stations. [16]. In 2004, T. Bellunatoa, M. Calvi, et al. studied the aging effects on aerogel material by exposing the samples to gamma rays from a Co-60 source and to high-intensity beams of neutrons and protons. Their results showed no aging effects on aerogel when irradiated with protons and gamma rays, but when exposed to neutron radiation, there was a slight loss in clarity [17]. D.L. Perego also demonstrated in his research published in 2008 the radiative hardness of aerogel. He exposed his samples to intense (proton, neutron, and gamma) radiation, to humid air, to standard black varnish, and to C_4F_{10} and CO_2 gases. D.L. Perego did not notice any changes in the optical properties in any of the tests he conducted [18]. In his 2014 research, Benjamin wanted to know if ionizing X-rays could create free radicals when exposed to a modified aerogel sample called polyurea crosslinked silica aerogel (PCSA). It was concluded that the aerogel sample (PCSA) that had been exposed to X-rays could not be described as radiation-resistant [19]. Hydrophobic fiber-reinforced silica aerogel composites were assessed by Helena Rocha et al. for use in thermal insulation applications related to Mars exploration. The fiber-reinforced silica aerogel samples were irradiated with a Co-60 radioactive source to monitor the effect of radiation on the fiber-reinforced aerogel. Their results showed that the gamma rays used in irradiation had no effect on the physical properties of the aerogel used. Gamma rays also caused an increase in the storage modulus of the aerogel used [20].

In the presented research, TEOS and Na_2SiO_3 utilized as precursors to prepared silica aerogel samples, the exposed to Cs-137 at different times 1hr, 2hr and 24hr then investigate the effects on samples under irradiation.

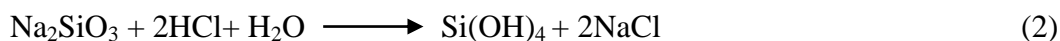
2. Experimental

2.1 Materials

Sodium silicate (Na_2SiO_3) (CAS No. 10213-79-3, purity 99.9%). Tetraethylorthosilicate (TEOS) 98.5% from Sigma-Aldrich (Germany). Trimethylchlorosilane (TMCS) > 99 % from TCI Japan, and n-Hexane (C_6H_{14}) >98% from Chemo-LAB (Belgium). Ethanol (98%) from Schariau (Spain) and Hydrochloride Acid (98%). Ammonium solution (NH_4OH) from CDH (India), distilled water, and amberlite resin to take off Na^+ from the Na_2SiO_3 solution. Finally, the radioactive source Cs-137.

2.2 Method of preparation and characterizations

Two different types of samples have been produced (G1, G2). G1 is prepared from condensed silica with a $pH > 6$ by combining TEOS as a precursor in ethanol and HCl 0.1M as an acidic catalyst, with a molar ratio of TEOS:Etho:HCl of [1:5:0.1]. G2 is made from sodium silicate (Na_2SiO_3) and using Amberlite resin (IR-120 Na) as an ion exchange resin. The solution was diluted in distilled water at a ratio of 1:5, and the resulting pH was 12. Equations (1) and (2) were used in the preparation of silicic acid [21].



After that, the solution was run through an ion exchange resin to extract silicic acid with a pH of 2-3. The G1 and G2 samples were converted to gel by adding NH_4OH [0.5M] solution drop by drop. G1 started turning into a gel after 10 min, while G2 converted to a gel during 30 min. After the two samples converted to gel, they were submerged three times for a whole day in pure ethanol. Once TMCS and n-Hexane were combined at a 1:6 molar ratio and heated to 60°C twice for two hours, the surface modification was prepared. To remove the residual TMCS, pure Hexane was washed two times, then let them soaked in hexane at room temperature for 48 hours. After that, samples were dried in an oven at 80°C–120°C gradually. Finally, samples G1 and G2 were irradiated with rays emitted from a cesium source (Cs-137) that has an activity equal to 10 μCi , represented by gamma rays. The radioactive source was placed directly a few centimeters above sample G1, which was in the form of

small, solid pieces. After that, G1 was exposed to the radiation emitted by the radioactive source for one hour, and part of the pieces exposed to radiation were taken for the purpose of conducting the test represented by FTIR spectroscopy (Bruker-Tensor 27 with ATR unit, Germany) to monitor the changes that may occur in the sample when irradiated and to ensure the success of the process of modifying the aerogel and making it hydrophobic. Making the aerogel hydrophobic allows us to maintain the properties of the aerogel for a longer period without worrying that the sample may undergo changes in its structure or composition or even damage as a result of exposure to the surrounding environment. Thus, it allows us to complete the irradiation process and continue testing. The irradiation process did not stop until a small part of the first sample was taken to conduct its first FTIR examination, where the radioactive source was removed and part of G1 was taken and then returned to its original position. The irradiation process was continued for another hour, and a second FTIR examination was performed two hours after the sample was placed under a radioactive source. A third irradiation was completed for the same sample, but the time period since irradiation for sample G1 was 24 hours. During the latter, FTIR and FESEM (TESCN MIRA3 FRENCH) tests were performed, and the purpose of using the latter test was to monitor the topography of the aerogel after it was exposed to radiation. The above steps were repeated for sample G2. Table (1) contains all the information about the radiation source.

Table 1. Information for Cesium-137 source

Isotope	Activity	Date of Manufacture	$t_{1/2}$	E(Kev)	I(%)
$^{137}_{55}\text{Cs}_{82}$	10 μCi	1978	30.07Y	661.66 513.97	$I_{\gamma}=85.10$ $I_{\beta}=94.40$ $I_{\alpha}=13.0$

3. Results and discussion

3.1 FTIR analysis

Figure 1. Shows the absorption spectra (transmittance versus wavenumber) of aerogel for the G1 sample before irradiation from a gamma-ray source (Cs-137).

FTIR analysis of the G1 sample before irradiation shows that at around $\sim 3500\text{ cm}^{-1}$ and 1600 cm^{-1} , the bands are weak due to O-H stretching or Si-OH stretching, which indicates that the presence of -OH groups is very scarce, and this is consistent with the successful hydrophobic modification of aerogel samples. At $(2959.50, 2925.46)\text{ cm}^{-1}$, it is associated with asymmetric and symmetric stretching vibrations of C-H bonds, indicating the successful modification of TMCS/n-hexane and thus the presence of hydrophobic organic groups $-\text{CH}_3$. The peak at $(1458.17, 1377.40)\text{ cm}^{-1}$ corresponds to the bending vibrations of C-H bonds, at $(1254.23)\text{ cm}^{-1}$ Si- CH_3 stretching Vibrations, at $(843.75)\text{ cm}^{-1}$ Si- CH_3 rocking Vibrations, at $(691.23, 441.52)\text{ cm}^{-1}$ associated with Si-O bending vibrations within the silica aerogel structure, at $(1160.54, 1060.07)\text{ cm}^{-1}$ Si-O-Si stretching vibrations, at $(797.70, 756.24)\text{ cm}^{-1}$ indicate Si-O-Si bending vibrations, at $(541.56)\text{ cm}^{-1}$ associated with lower-energy vibrational modes of Si-O [22-28]. The FTIR of sample G2 is not much different from sample G1 which is clearly shown in Figure 2. Both shows hydrophobic character when modified with TMCS/n-hexane, with peaks at $(2958.64, 2925.15)\text{ cm}^{-1}$ back to C-H band, at $(1253.93)\text{ cm}^{-1}$ for Si- CH_3 , and at $(1061.91)\text{ cm}^{-1}$ to Si-O-Si which represents the silica framework [29, 30].

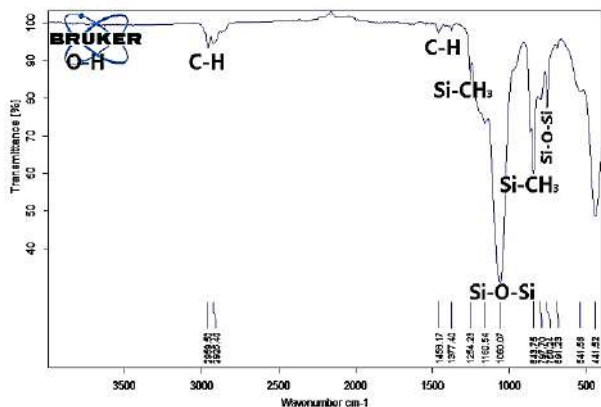


Fig. 1. FTIR spectrum for G1 silica aerogel sample

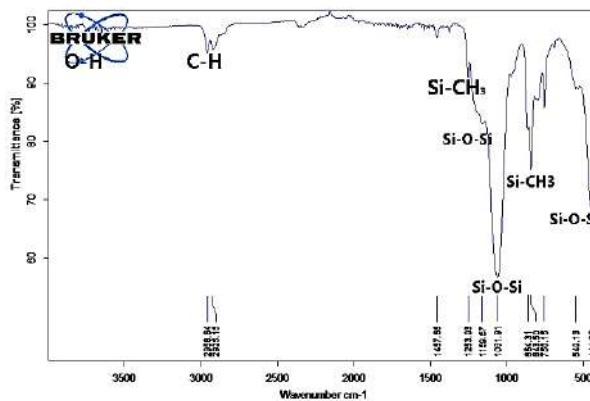
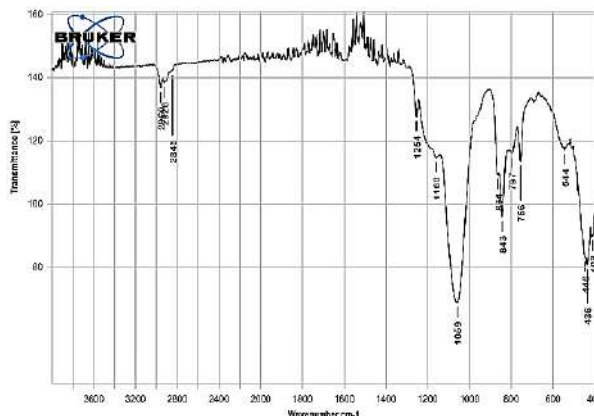
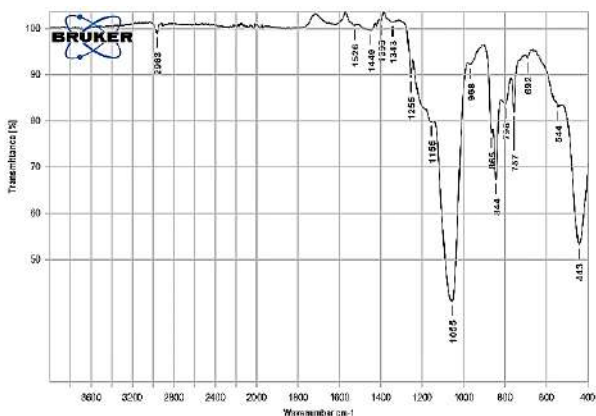
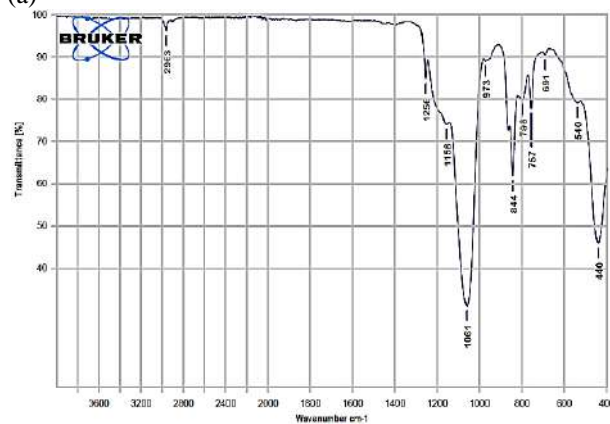


Fig. 2. FTIR spectrum for G2 silica aerogel sample



(a)

(b)



(c)

Fig. 3. FTIR spectra of sample G1 after irradiation for (a) 1 hour, (b) 2 hours, (c) 24 hours

In the figure 3(a). There are no prominent shifts in the positions of the peaks, such as a noticeable increase or decrease in the wave number when compared to the positions of the peaks before irradiation of sample G1, which indicates that there is no increase or decrease in the strength of the bonds, and no noticeable changes in the width of the absorption peaks (wider or narrower peaks) compared to sample G1 before irradiation, indicating no changes in molecular vibrations and thus no increase in disorder or increase in order or crystallinity. As the CH₃ functional group remained

unchanged at 1255 cm^{-1} , 2963 cm^{-1} , and 844 cm^{-1} . All this indicates that no changes have occurred in the sample. No oxidation or reduction reactions occurred, as the material maintained its hydrophobicity. The weak bands associated with Si-OH at 1400 cm^{-1} and 3500 cm^{-1} are still present. The presence of Si-O-Si at 1055 cm^{-1} proves that the material still contains silica, which is the main component of the aerogel.

For figure 3(b) when G1 was irradiated with a cesium source after two hours, silica is still present at 1059 cm^{-1} and was not affected by radiation. The functional group is still present at 1254 cm^{-1} , 2960 cm^{-1} , 843 cm^{-1} , and finally, the material remains hydrophobic. For figure 3(c) when G1 irradiated with a cesium source after twenty-four hours, there is no significant change from its predecessors regarding irradiation after one and two hours.

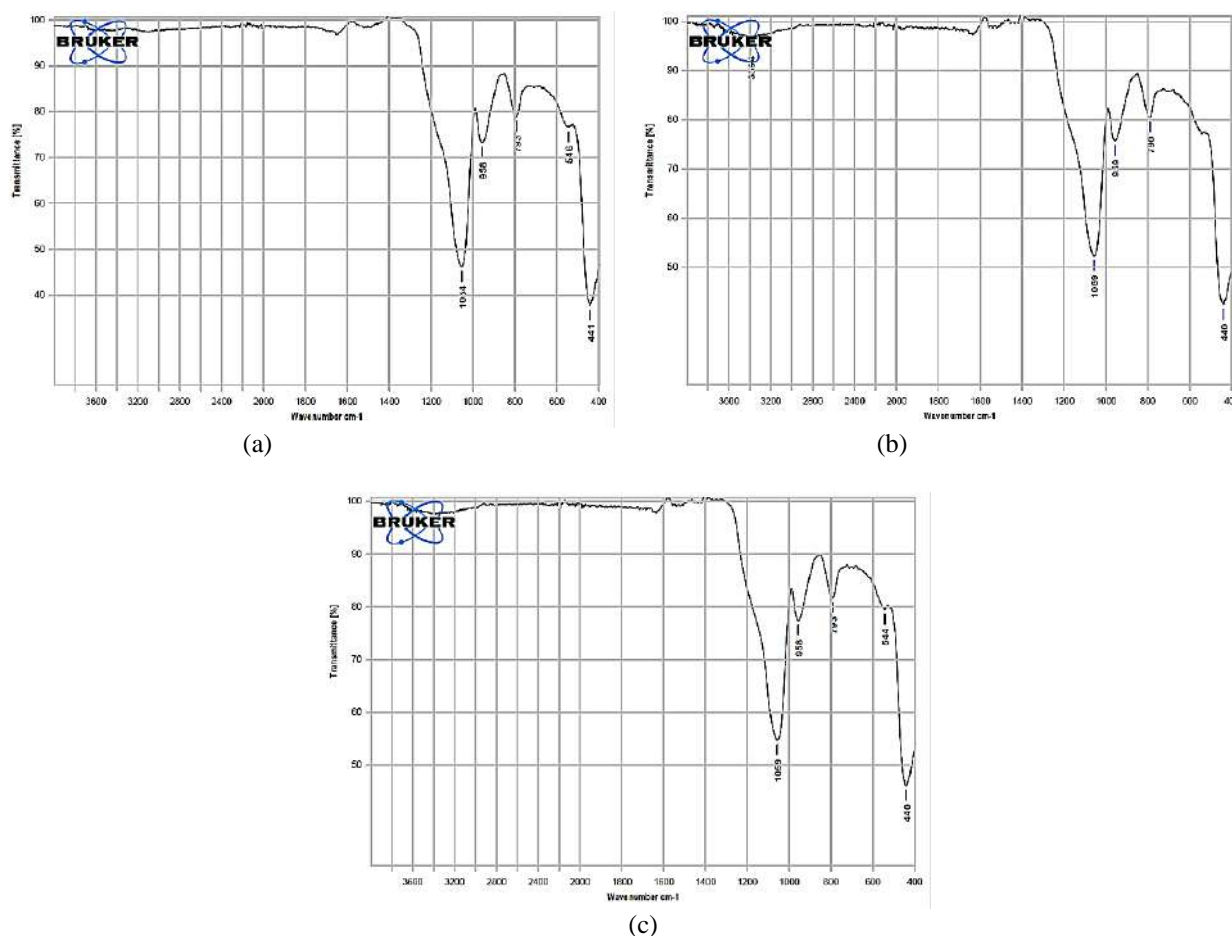


Fig. 4. FTIR spectra of sample G2 after irradiation for (a) 1 hour (b) for 2 hours (c) 24 hours

Similar to sample G1, sample G2 was irradiated with the same source for the same time periods and measured by FTIR. We will also summarize the most prominent results in the following lines. In figures 4(a), 4(b) and 4(c) the CH₃ functional group disappears, which can be attributed to one, all or possibly another of the following reasons.

Firstly, the disappearance of the CH₃ functional group may be attributed to Radiation-Induced Chemical Reactions: Ionizing radiation, such as gamma radiation emitted by Cs-137, can break chemical bonds and induce chemical reactions on the surface of materials. This can result in the cleavage or modification of functional groups that contribute to hydrophobicity. For example, radiation can lead to the degradation or oxidation of organic or hydrophobic moieties, causing them to lose their hydrophobic properties. Also, may be the Cross-Linking and Polymerization: Radiation exposure can also promote cross-linking or polymerization reactions on the surface of materials. This can lead to the formation of new chemical structures that may not be hydrophobic, thereby altering the surface properties. Secondly, it is possible due to the formation of radical species: Ionizing radiation generates

highly reactive species, such as free radicals, on the surface of the material. These radicals can initiate chemical reactions with functional groups, leading to their transformation or removal. In the case of hydrophobic groups, their susceptibility to radiation-induced reactions can result in changes in surface chemistry. Third, surface cleaning: Radiation can effectively "clean" or remove contaminants and organic residues from the surface of the material. This cleaning action can expose the underlying silica structure, which is inherently hydrophilic, thus changing the surface from hydrophobic to hydrophilic. Fourth, depolymerization: In some cases, radiation can stimulate the depolymerization of surface functional groups, leading to the removal of hydrophobic components. As well as peaks at (1054, 958, 959, 790, 793, 440, 441) cm^{-1} are attributed to Si-O-Si vibration modes.

3.2. FESEM analysis

A micrograph of sample G1 before exposure to gamma radiation is shown in figure (5) with two different magnifications (200nm, 1 μm).

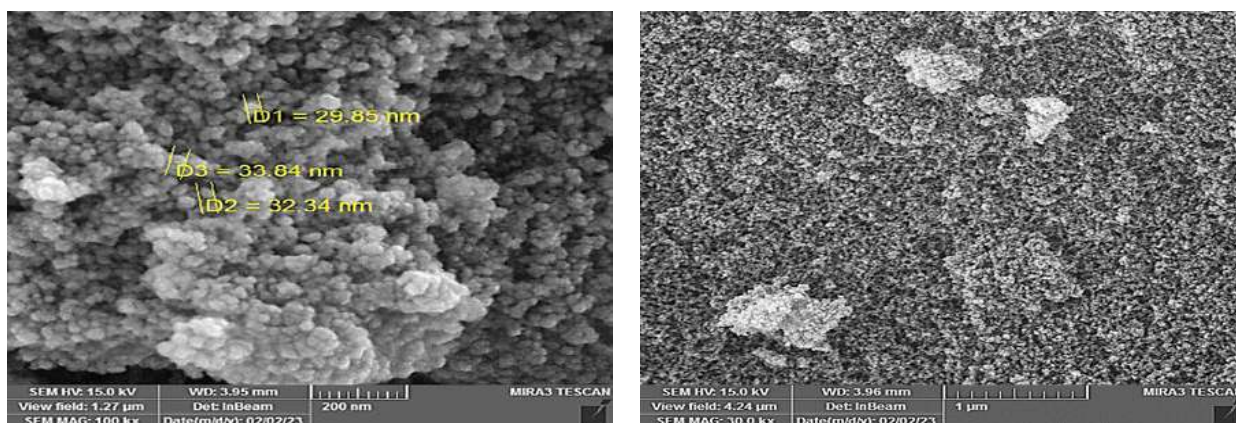


Fig. 5. FESEM image for aerogel G1 sample

From the figure 5. the fine and well-defined microstructure of sample G1 can be seen with homogeneity and uniformity for SiO_2 particles and porous network. Which supports the test results derived from previous studies [23, 24]. The size of the colloidal particles is about 33 nanometers, which is consistent with the results of previous studies, which confirms the success of the synthesis process. While the micrographs of sample G2 before irradiation are shown in figure (6) below, they are also present at two different magnifications (200nm, 10 μm).

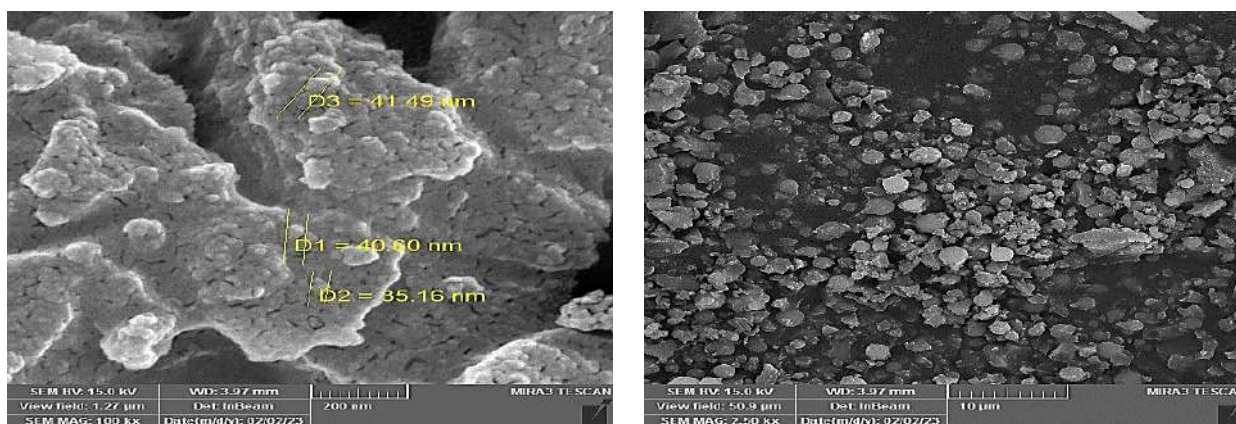


Fig. 6. FESEM image for aerogel G2 sample

In sample G2, the rough surface with an open pore network appears at magnification (10 μm), and the size of the colloidal particles is about 41 nanometers, and this is also consistent with the results of

previous studies[31]. A micrograph of sample G1 after exposure to gamma radiation is shown in figure (7) below with also two different magnifications (200nm, 1 μ m).

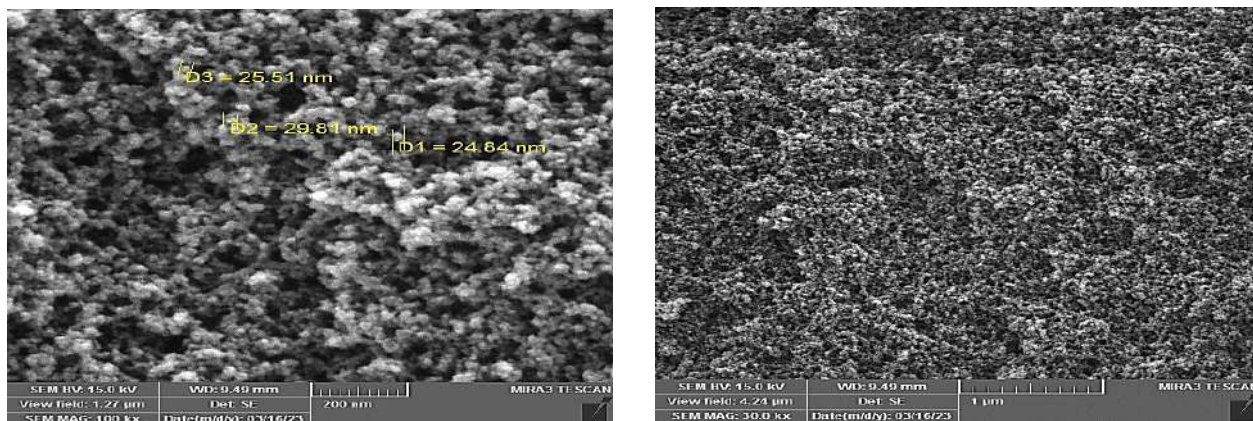


Fig. 7. FESEM image for aerogel sample G1 after exposure to gamma radiation

From figure (7), the three-dimensional porous structures of the silica aerogel still exist. The smooth and uniform surface remains unchanged. The nanopores still have a homogeneous distribution. The size of the colloidal particles is about 30 nanometers, which is no different from the size of the particles before exposure to gamma rays. A micrograph of sample G2 after exposure to gamma radiation is shown in figure (8) below with also two different magnifications (2 μ m, 1 μ m).

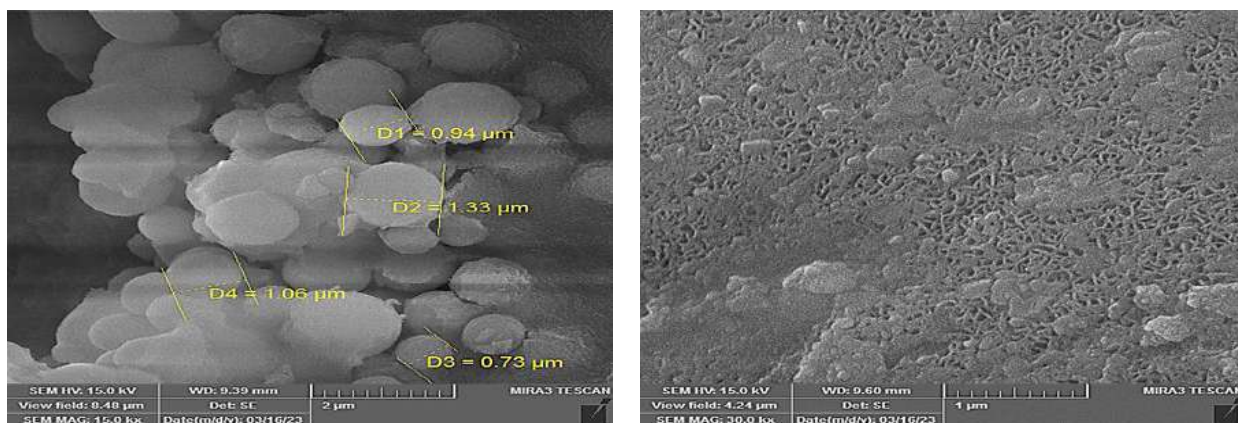


Fig. 8. FESEM image for aerogel sample G2 after exposure to gamma radiation

The micrograph at 1 μ m shows that the 3D porous structures of the silica aerogel are also still present and are not affected by the radiation dose and still maintain the aggregate. The particle size of sample G2 is approximately 1 μ m after irradiation. We must note here that the particle size measurement shown in the microscopic image at 2 μ m magnification depends on the available data provided by the testing center.

4. Conclusions

This research paper presents the effect of radiation from a Cesium source on the properties of a hydrophobic aerogel made from two different precursors (TEOS, Na₂SiO₃) at ambient pressure drying. FTIR and FESEM measuring devices were used to measure changes in the aerogel. The results presented in the research paper indicated that there were no changes to sample G1 (prepared from TEOS) in both FTIR and FESEM measurements, as no prominent and clear changes appeared in the absorption peaks displayed after irradiation, nor did any change appear in the three-dimensional porous gel structure and particle size of sample G1 after irradiation. For sample G2 (prepared from Na₂SiO₃), the results showed changes in the absorption peaks after irradiation. The CH₃ functional group associated with the substance's hydrophobicity disappeared and was not replaced by another functional group. This may be due to a reason or group of reasons or another reason from the reasons described in the results section. Also, no changes were observed in the network 3D of sample G2 after irradiation.

However, this study represents a preliminary step to study how gamma nuclear radiation affects the properties of aerogels prepared from different precursors to select the best precursor for the best radiation isolation.

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