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## Preparation and Characterization of High Surface Area Nanosilica from Iraqi Sand via Sol-Gel Technique

Firdews Shakir<sup>1\*</sup>, Hussein Q. Hussein<sup>2</sup> and Zeinab T. Abdulwahhab<sup>3</sup><sup>1</sup>State Company for Steel Industries /Ministry of Industry and Minerals, Baghdad, Iraq.<sup>2</sup>Chemical Engineering Department, College of Engineering, University of Baghdad, Baghdad, Iraq.<sup>3</sup>Petroleum Research and Development Center, Ministry of Oil, Baghdad, Iraq.<sup>1\*</sup>Corresponding Author E-mail: [esraa77\\_esraa77@yahoo.com](mailto:esraa77_esraa77@yahoo.com)<sup>2</sup>[huseinqassab@coeng.uobaghdad.edu.iq](mailto:huseinqassab@coeng.uobaghdad.edu.iq)<sup>3</sup>[zainabzozozo@yahoo.com](mailto:zainabzozozo@yahoo.com)

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### Abstract

The present study revealed a low-cost process for utilizing desert sand for preparing nanosilica by sol-gel technique. This work required sodium hydroxide, concentrated hydrochloric acid, distillate water as raw materials, and Iraqi sand. Nanosilica sample was characterized by X-Ray Diffraction (XRD), scanning electron microscopy analysis (SEM), atomic force microscope (AFM), surface area (BET) method, and fourier transform infrared (FTIR). The XRD result of produced sample is referred to as amorphous silica, and it has a broad peak at  $2\theta = 22^\circ - 22.5^\circ$ . SEM showed spherical, agglomerated silica particles with a diameter range of 26.57–28.93 nm. In addition, the average particle size was 76.35nm, with a dimension range of 40-110 nm, and the surface area was 510.96 m<sup>2</sup>/g. The inclusion of hydrogen-bonded silanol groups (Si–O–H) at 3437.15 cm<sup>-1</sup> and siloxane groups (Si–O–Si) at 1087.85 cm<sup>-1</sup> in the FTIR spectra.

**Keywords:** Nanosilica; Western Region sand; Particles; Hydrochloric Acid; and Sol-Gel.

### تحضير وتشخيص السليكا النانوية ذات المساحة السطحية العالية من الرمل العراقي بتقنية Sol-Gel

#### الخلاصة:

كشفت الدراسة الحالية عن عملية منخفضة التكلفة لاستخدام رمال الصحراء لتحضير السليكا النانوية باستخدام تقنية Sol-Gel. المواد الخام المستخدمة في هذا العمل هي هيدروكسيد الصوديوم، وحمض الهيدروكلوريك المركز، والمياه المقطرة، إضافة إلى الرمل العراقي. تم تشخيص عينة نانوسيليكا بحيود الأشعة السينية (XRD)، والمساح المجهر الإلكتروني (SEM)، ومجهر القوة الذرية (AFM)، وقياس المساحة السطحية بطريقة BET، وكذلك التحليل الطيفي بتقنية (FTIR). أظهرت نتيجة XRD للنموذج المنتج بأن السليكا النانوية الناتجة ذات طور غير متبلور ولها قمة واسعة عند  $2\theta = 22^\circ - 22.5^\circ$ . أظهر فحص SEM جسيمات السليكا الكروية المتكتلة التي يتراوح قطرها بين 26.57 - 28.93 نانومتر.

بالإضافة إلى ذلك، كان متوسط حجم الجسيمات 76.35 نانومتر و بمدى أبعاد 40-110 نانومتر، وبلغت مساحة السطح 510.96 م<sup>2</sup>/غم. تم تضمين مجموعات silanol المرتبطة بالهيدروجين (Si – O – H) عند 3437.15 cm<sup>-1</sup> ومجموعات (Si – O – Si) siloxane عند 1087.85 cm<sup>-1</sup> في أطياف FTIR.

الكلمات الدلالية: السليكا النانوية، رمال المنطقة الغربية، الدقائق، حامض الهيدروكلوريك، و سول-جل.

## 1. Introduction

In the last half-century, manufacturing processes, in general, have undergone a material application transformation as a result of a change from traditional bulk materials to nanoscale materials. The increase in possibilities for the manipulation of matter at the nanometer-scale has primarily led to this growth with nanomaterials at the leading edge of this fast-developing field [1]. Any substance with nanoscales in its structure is considered a nanomaterial, with dimensions ranging from one to one hundred nanometers [2].

Recent advancements in the field of nanotechnology have prompted several studies aimed at the synthesis and application of nanomaterials [3]. Because of their excellent physical and chemical properties, nanostructured materials have a broad range of applications, including electronics, textiles, agriculture, food, medicine, and cosmetics [1][4].

In comparison to conventional methods, nanotechnology has been used as a very effective technique for improving the flow characteristics of heavy crude oil across pipelines via reduction the viscosity[5]. The nanomaterials may have unique benefits for the solvent deasphalting process because of their high surface area to volume ratio and hence a large number of available active sites, enabling them to selectively adsorb asphaltenes onto their surfaces and thus improve asphaltene removal[6].

Silica (SiO<sub>2</sub>) has been the focus of extensive study because of the advantages it provides for a wide variety of applications. Silica can be present in nature as a major component of sand or in species such as rice husk, coffee husk, wheat husk, sugar cane bagasse, corn cob ash, and fly ash [7]. Silica is widely available in the world, accounting for 59 percent of the Earth's crust [8]. The Western Desert of Iraq has enormous amounts of silica sand deposits[9]. These deposits are found in the Ga'ara, Hussainiyat, Najmah, Nahr Umr, and Rutbah. The Rutbah area was selected for the mine's location. Ardhuma is the name of the mine, which is situated about ten kilometers west of Rutbah Town. Silica sand deposits vary in particle size from extremely fine

to coarse, although the most frequent are fine to medium grain sizes. The grains are sub-angular to sub-rounded in form, and the  $\text{Fe}_2\text{O}_3$  content varies from 0.01 to 1.5% [10].

Nanosilica (NS) has the same structure as silica but with a particle size of less than 100 nm. Nanosilica also gained popularity due to its highly reactive surface area [11], physical and chemical stability, and low toxicity [12]. In addition, silica nanoparticles (SNPs) have a large surface area and a small diameter, making them suitable for a wide range of applications, such as electronics and photonics, and energy harvesting and storage. In addition, they improve the solvent deasphalting process using nanosilica, which significantly impacts deasphalted oil and pitch yields. Finally, they have been shown to be effective at reducing the viscosity of heavy oil [5][6][13][14].

NS was made using various procedures such as chemical precipitation process, sol-gel technique, vaporization with high temperature, and speed of the vertical rotating mill and planetary and ball mill are the most common methods for synthesis SNPs from Sand [14][15][16].the raw materials used and the conditions of the process, such as temperature, time of precipitation, pH, addition of coagulants, and washing and drying methods. These Variables affect particle size, aggregation, and surface area [14].

Radhip et al [16] synthesized SNPs by using Malpe beach sand from Karnataka, India, utilizing the planetary ball mill technique at room temperature and various milling hours. The outcome was a silica nanoparticle smaller than 100 nm in size after 150 hours of milling at 250 rpm.Musić et al.[17] precipitated silica by neutralizing sodium silicate solution (water glass) with  $\text{H}_2\text{SO}_4$ . Precipitated silica in the form of powder may have a specific surface area of up to  $130 \text{ m}^2\text{g}^{-1}$ .Wahyudi et al.[18]synthesized silica nanoparticles though alkali fusion, The specific surface area of nanosilica obtained was up to  $157 \text{ m}^2\text{g}^{-1}$ .

This research aimed to investigate the synthesis of Nano silica using local Iraqi Sand as a silica precursor by chemical sol-gel and characterises amorphous SNPs properties.

## 2. Materials and Methodology

### 2.1. Materials

#### 2.1.1. Iraqi sand

local state company for mining industries/ Department of Mineral Extraction provided local sand as a precursor of Nanosilica. This sand was available in the Ardhuma location in Al-Anbar Province, Western Iraq, and The chemical composition of the sand is shown in Table (1).

**Table (1) The chemical composition of Iraqi silica sand (Department of Mineral Extraction ).**

Component	Wt. %
SiO <sub>2</sub>	98
Fe <sub>2</sub> O <sub>3</sub>	0.08
Al <sub>2</sub> O <sub>3</sub>	0.39
SO <sub>3</sub>	0.07
CaO	0.15

#### 2.1.2 .Chemical Materials

The chemicals used for Nanosilica preparation are listed in Table (2).

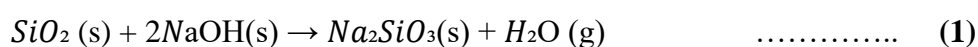
**Table (2) Chemical materials for Nanosilica preparation.**

Material	Formula	Purity%	Country of origin	The manufacturing company	Molecular weight (gram/mole)
Hydrochloric acid	HCl	36	India	CDH	36.46
Sodium hydroxide	NaOH	97	United kingdom	Choping and Williams	40
Distillate water					

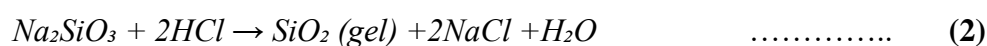
### 2.2. Procedure

A typical preparation of nanosilica using a chemical sol-gel technique that was modified from a previous study by Shakir [19], should be followed :

The sand was washed with distillate water, and the sample was left to dry for 48 hours after washing. Then 40 grams of sand was crushed for 8 hours in a planetary ball mill at the nanotechnology and advanced materials research centre/university of technology before being shaker sieved to 38 $\mu$ m; Using a pestle and mortar, 125 grams of sodium hydroxide pellets are crushed. Finally, sand and sodium hydroxide were well mixed and heated at 500 ° C. for 30 minutes to create solid sodium silicate.. The sand and sodium hydroxide were well mixed and heated at 500 °C for 30 minutes to create solid sodium silicate, as shown in equation 1.



The solid sodium silicate was cooled to room temperature and transferred to an 800 ml beaker. Next, 500 ml of distillate water was added to the beaker with vigorous stirring using a magnetic stirrer; the homogenous solution is produced when the reaction occurred. Finally, concentrated hydrochloric acid (36%) is added intermittently until pH reaches 1 to obtain a white gel, as shown in equation 2.



Continuous stirring at 400 rpm for one hour. Filter paper and a Buckner funnel fitted with a vacuum pump separated the mother liquor from the solid gel. To check the presence of chloride ions, add a few drops of 0.025 N AgNO<sub>3</sub> solution to 5 ml of the liquid filtrate; if white crystalline AgCl precipitates, continue the washing process. After washing, nanosilica powder is produced by drying the gel at 110 C° for 12 hours in an electric lab drier.

The schematic diagram of preparing SNPs is shown in Figure (1).

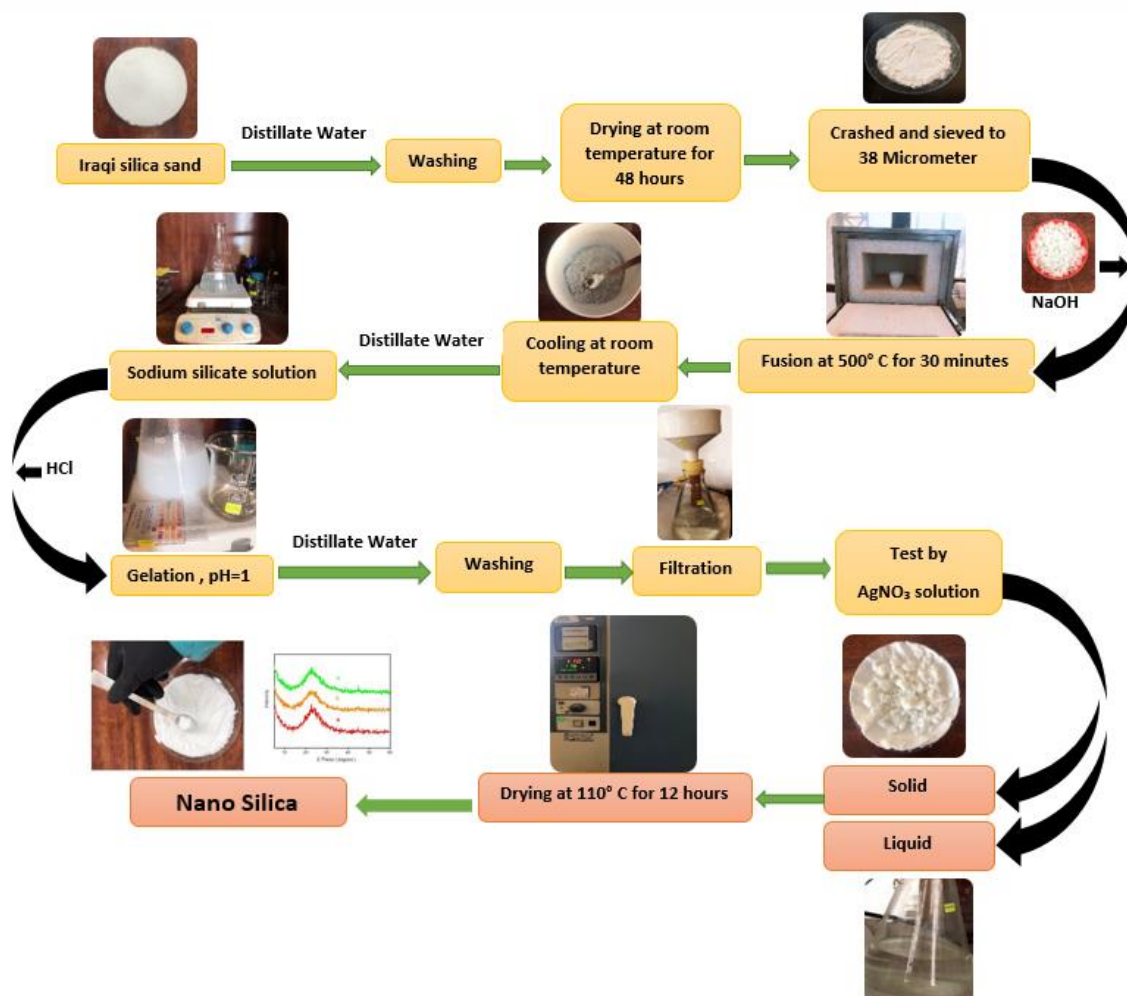


Fig. (1): The schematic diagram of preparing SNPs by sol-gel method.

### 2.3. Characterization of Silica Nanoparticles

**Structure and pattern identification:** XRD (The PANalytical, X Pert PRO) was used to identify the phase of the produced nanosilica.

**Scanning Electron Microscope (SEM):** SEM was performed on sample using (TESCAN/MIRA3 LMU model) to determine the surface morphologies of NSPs.

**Atomic Force Microscope (AFM):** An AFM was used to examine the prepared NS to measure the mean particle size and the particle size distribution. A scanning probe microscope (SPM-AA3000-Scanning Probe Microscope, Angstrom Advance Inc.,USA) was employed in this study.

**BET Surface Area:** Brunauer, Emmett, and Teller (BET) (Thermo Finnegan/USA) was used to determine the NS sample surface area, as specified by ASTM ISO 9277.2010.

**Fourier Transform Infrared Spectroscopy (FT-IR):** The FT-IR equipment (Shimadzu, IR Prestige-21) was used to identify the nanosilica's functional groups from  $4000$  to  $400\text{ cm}^{-1}$  in the wavenumber domain.

### 3. Results and Discussion

#### 3.1. X-Ray Diffraction Analysis (XRD)

When X-rays scatter in many directions, forming a big hill-like bump, with a peak in the range  $2\Theta = 15^{\circ}$ - $30^{\circ}$ , indicating the presence of amorphous structure and a highly disordered form of silica, amorphous phase would predominate[20]. Figure (2) characterizes the XRD pattern of prepared nanosilica. The absorption pattern confirms the amorphous phase of nanosilica with the absence of sharp, strong peaks, and a high-intensity broad diffraction peak at  $2\Theta = 22^{\circ}$ - $22.5^{\circ}$ ; this result is roughly similar to the findings by[14][19][20][21].

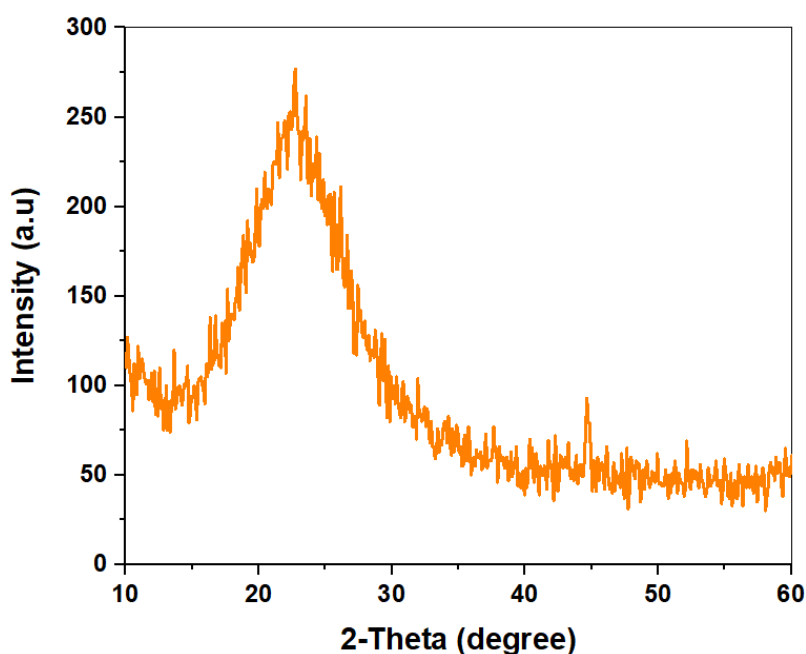
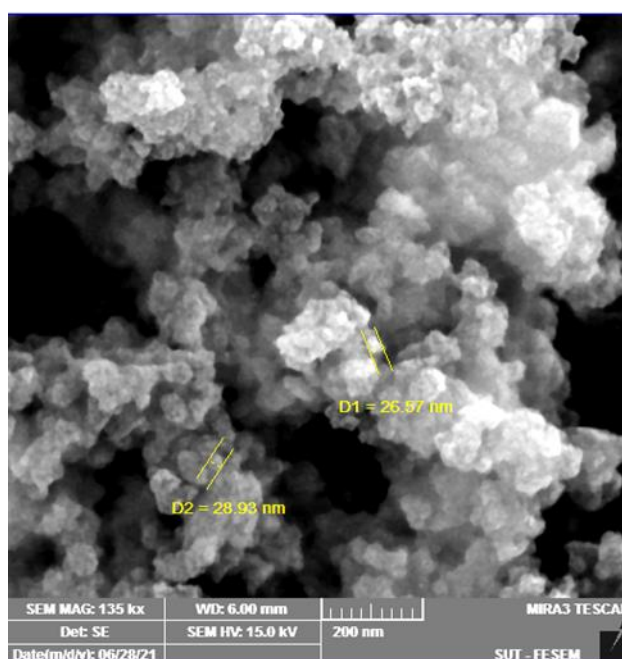


Fig. (2): X-ray diffraction pattern series of prepared SNPs.



### 3.5. Scanning Electron Microscopy Analysis (SEM)

Figure (3) shows a scanning electron micrograph of precipitated silica. The micrograph image clearly showed the amorphous nanosilica components are uniformly dispersed. However, it is essential to notice that agglomeration occurs and has an amorphous nature. Comparing the SEM micrograph analysis findings and the XRD pattern analysis reveals that the silica is amorphous nanosilica[22]. And the particles have a spherical shape with an average size of 26.75-28.93 nm and a porous appearance .this finding is in agreement with [13][17][23].



**Fig. (3): SEM for prepared nanosilica.**

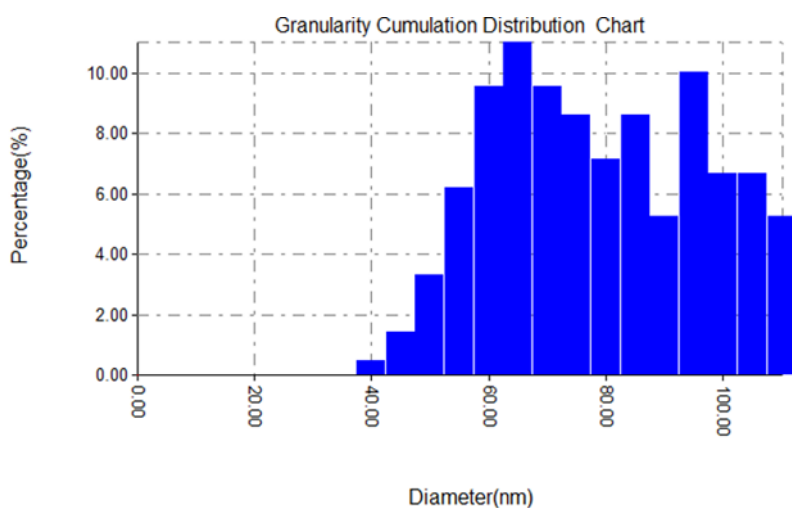
### 3.3. Atomic force microscopy and Average particle size

AFM was used to detect the average diameter of SNPs, a high resolution of (444 \* 452) pixels and a scanned area (1622\*1651) nm<sup>2</sup> were used to obtain a sharp topography of NS. As a result, the average diameter of nanosilica prepared was 76.35 nm with a dimension range of 40-110 nm, as shown by the granularity cumulation distribution chart and particle size distribution as shown in Figure (4-a) and Table (3). The topography of the nanosilica surface can be seen very clearly in this research in two-dimensional and three-dimensional images, which displayed an aspherical shape and agglomerated tiny grains of nanosilica particles, as shown in Figure (4 b and c), respectively. This result agrees with [20][24].

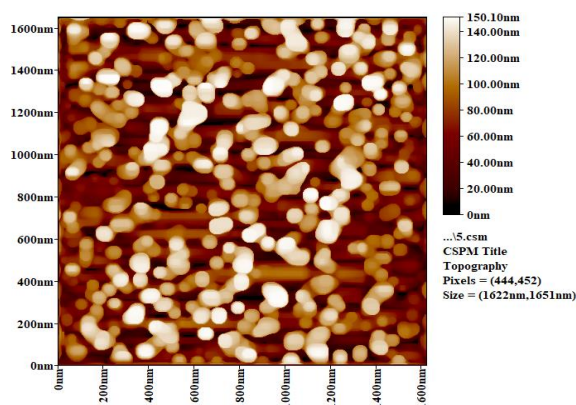


**Table (3) Particles size distribution of prepared nanosilica**

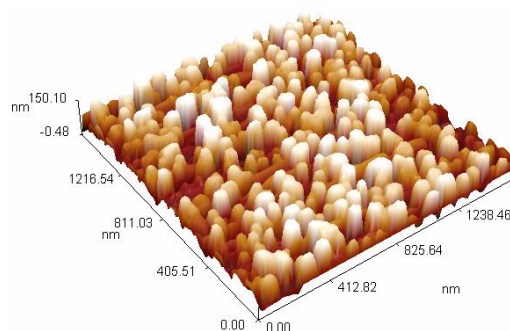
Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)
40.00	0.48	0.48	65.00	11.00	32.06	90.00	5.26	71.29
45.00	1.44	1.91	70.00	9.57	41.63	95.00	10.05	81.34
50.00	3.35	5.26	75.00	8.61	50.24	100.00	6.70	88.04
55.00	6.22	11.48	80.00	7.18	57.42	105.00	6.70	94.74
60.00	9.57	21.05	85.00	8.61	66.03	110.00	5.26	100.00



(a)



(b)



(c)

**Fig. (4): (a) Particle size distribution of prepared NS, (b) 2-D and (c) 3-D images of prepared NS.**

### 3.4. BET surface area analysis

The surface area was calculated using the BET procedure of physical nitrogen adsorption at liquid nitrogen temperature. The study found that silica nanoparticles prepared had surface areas of 510.96 m<sup>2</sup>/g. The BET surface area obtained by this study is higher than the surface area obtained by [14][17].

### 3.2. FT-IR analysis

Figure (5) depicts the FT-IR spectrum of NS for each sample obtained from Iraqi silica sand using the chemical precipitation method (125g NaOH + 40g sand) by adding HCl to the sample. The finding reveals the chemical compositions of nanosilica product. The spectrum shows a broad and more intensive band at 1087.85 cm<sup>-1</sup>, which is typical of the Si-O-Si asymmetric stretching vibration, in agreement with [12][23][25][26][27]. This band is less intense, but on the other side of the package, for the range 470.63 cm<sup>-1</sup> - 802.39cm<sup>-1</sup>, which is due to symmetric stretching vibrations (Si-O-Si), these are the distinctive solid lines of silica, and these are in agreement with [13][20][21][25]. The high concentration of silanol groups (Si-OH) at 956.69 cm<sup>-1</sup> was seen in the results; this agrees with [23][25][26][27][28]. Adsorbed water molecules on the SiO<sub>2</sub> surface cause -OH peak broadening, and there is a peak at 3437.15 cm<sup>-1</sup>. The bending vibration of the -OH bonds in adsorbed water molecules on the Nanosilica surface is responsible for the stretching frequency of the hydroxyl group and the peak at 1634.35 cm<sup>-1</sup>, the bound water in the precipitated silica that is not removed during oven heating. These are in agreement with [22][23][27][29]. All the above results are consistent with chemical analysis as shown in Table (4).

**Table (4) Assignments of the FTIR for studied sample.**

Wavenumber(cm <sup>-1</sup> )	Assignment	References
470.63 -802.39	(Si-O-Si)symmetric stretching (siloxane group)	[13][20][21][25]
956.69	Si-OH bending vibrational absorption (silanol group)	[23][25][26][27][28]
1087.85	Si-O-Si asymmetric stretching	[12][23][25][26][27]
1634.35	H <sub>2</sub> O	[22][23][27][29]
3437.15	-OH bending vibration	[22][23][27][29]

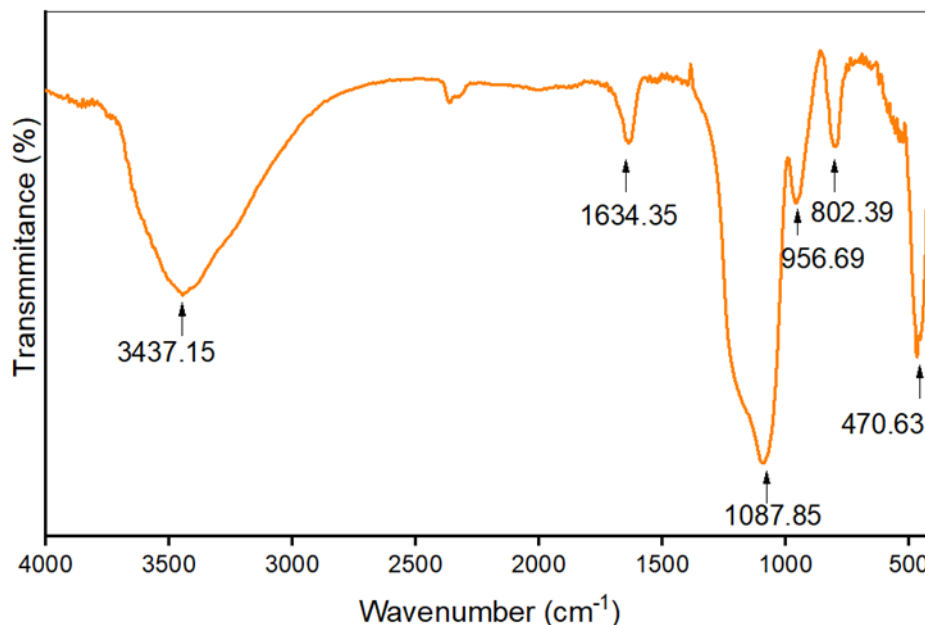


Fig. (5): The FT-IR spectra of prepared nanosilica.

#### 4. Conclusion

With the availability of high-quality sand in the western region of Iraq with a silica content of up to 98 %, the synthesis of silica nanoparticles from local raw sand is regarded as an eco-friendly and cost-effective process. Silica sand is a good alternative precursor that may be used as a silica source and chemically treated to produce silica nanoparticles. The surface area of chemically formed silica nanoparticles was quite high as compared to other methods of producing nanoparticles. The tests of the atomic force microscopy AFM prepared sample revealed that the production of silica nanoparticles in the average diameter of particles less than 100 nm, confirming the validity of the orientation in the selection of methods of preparation of nanosilica from the silica peak that resulted in the XRD experiments revealed that the silica nanoparticles was amorphous in shape. SEM scan identifies the spherical shaped morphology of produced nanosilica with a size ranging from 26.57 to 28.93 nm. The FTIR also show the NS spectrum at 1087.85 cm<sup>-1</sup>. When the SNPs spectrum is compared and matched with the scientific literature, it is discovered to be entirely applicable with SNPs under study to ensure the validity of prepared nanomaterials; the FTIR spectra confirmed amorphous silica nanoparticles produced coinciding with the standard.

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