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Assessment of sand dunes in Missan Governorate, South-eastern Iraq: Implications for Extraction High Purity Silica

S J Al-Khafaji 1, F W Al-Halfi 2and H K Jasim 3
1,2 Department of Geology, College of Science, University of Basrah , Basrah, Iraq
3 Department of Geology, College of Science, University of Baghdad, Baghdad, Iraq
Corresponding author:khafaji52000@gmail.com

Abstract

Sand dunes are widely spread in Missan Governorate, South-eastern Iraq in many areas including; Manziliyah, Khazina, Zubadiate, and Chailat. X-ray diffraction (XRD) results of sand samples of sizes 0.063-2 mm, 0.125-0.250mm, and 0.250-0.500mm, revealed that quartz is the main mineral phase, followed by calcite, feldspar and trace of gypsum. Chemically SiO_2 is the main oxide in sand of an average 61.36 % while the rest oxides; CaO (14.08%), Al_2O_3 (6. 76%), Fe_2O_3 (3.05%), K_2O+Na_2O (2.30 %) are considered as impurities that could affect the properties of sand. XRD tests indicated the formation of crystalline phase of synthetic silica. XRF showed an increases in SiO_2 to 85.37%, while all other oxides (impurities) were reduced to their minimum rate, particularly CaO (1.06 %). Field emission scanning electron microscope (FESEM) and Brunauer-Emmett-Teller (BET) tests indicated the existence of well compiled spherical particles of synthetic silica within the size of 8.55 nm and surface area of 66.51 m^2\g. The medium sand size (0.250-0.500mm) is the appropriate size to produce high purity crystalline silica as it contains the greatest percentage of silica and the low percent of impurities compared to other sizes of sand.

Keywords: Crystalline Silica, Sand Dunes, Missan, Surface Area, Iraq.

1. Introduction:

Sand dune is an abundant natural material that occurs in many parts of the world [1]
The sand dunes have a distinct mineral content, as well as a distinct texture in both size and form of the naturally sorted grains. Many sectors can benefit from these combined properties as a low-cost raw material [2] Sand is one of the important raw materials for many different industries, whether used as a natural form or after make some treatments or improvements in order to be used in other applications [3]. Different methods for preparation silica have been applid such as emulsions [4], sol-gel [5], Stöber method [6] hydrothermal, alkaline fusion [7], coprecipitation, and electrocoagulation [8]. A type of technology known as the coprecipitation method uses chemicals to create a solute and then wait for the desired precipitate to develop., when compared coprecipitation method to other conventional methods, it is simple method to separate the production at short time and low temperature [9]. The equipment utilized is simple and inexpensive allowing for the production of a powder with a specific crystal size.. The present study aims to assess the suitability of sand dunes in Missan area, southeastern Iraq as low-cost raw materials to produce high purity crystalline silica using coprecipitation methods.

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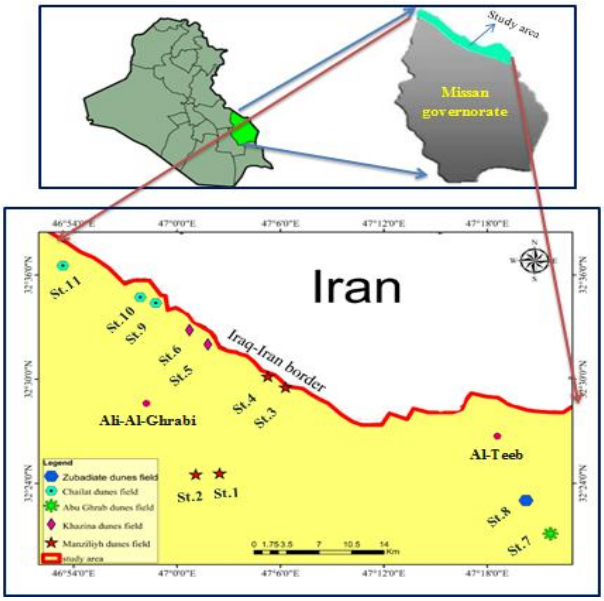


Figure 1. The study area and sample locations.

2. Materials and Methods

The study area includes the sand dune fields located at the north-east of Missan area, at latitude (32˚ 21' 0"- 32 ˚ 36' 00")N. and longitude (46 ˚53' 00"- 47 ˚21' 00")E, includes the following stations; Manziliyah, Khazina Zubadiate ,and Chailat, **Figure.1**, Eleven samples from sand dunes were gathered based on how they dispersed throughout the field. During October 2020, Sand samples were subjected to sieve to sand sizes 0.063-2 mm, 0.250-0.500mm, and 0.125-0.250 mm. Raw sand (total, medium, and fine size) have been analysed using X-ray diffraction technique at the Ministry of Science and Technology in Iraq uses PW3830 comprehensive X-ray devices, and chemically employs the X-ray fluoresces (XRF) technique at the University of Baghdad, Iraq. Then, sand samples were grinded by a clean electric agate mortar to 200 mesh [10].
For a full day, the powder samples were immersed in 37% of a 2M hydrochloric solution, **Figure 2** , Immersion of sand in HCl acid can lessen contaminants in raw material [9] After being filtered and rinsed with distilled water until the pH reaches neutral, the sand is dried at 100°C for two hours. [11].
The obtained sand was identified and assessed using XRD and XRF after the immersion process. Next, sand was immersed in 7M NaOH (99%) at 95 °C for 3-4 hours using a magnetic stirrer [11]; [7],Afterward, silicic acid was prepared by add 2M HCl to prepare sodium silicate solution . Ten times, distilled water was used to wash away the NaCl from the silicic acid [7]. After filtering, the silicic acid was dried at 110 °C for 2-3 hours. [12]. After that, the agglomerated silica are ground in to produce silica powder. XRD was used to identify the silica powder (**Figure. 3**) in order to look into the phase formation. Utilizing the XRF technique, the elemental composition is also assessed. Fourier transform infrared spectrometry (FTIR) Type: FTIR-8400S (SHIMADZU) at University of Basra was apply to detect the synthetic silica functional groups,. Field emission scanning electron microscopy (FESM-EDS) in Tehran University, Iran, was used to demonstrate the morphology and image of crystal form, and Bruner-Emmett-Teller (BET) technique: BEL Prep VAC II type in Tehran University, Iran, was also apply to determine the specific surface area of prepared silica.

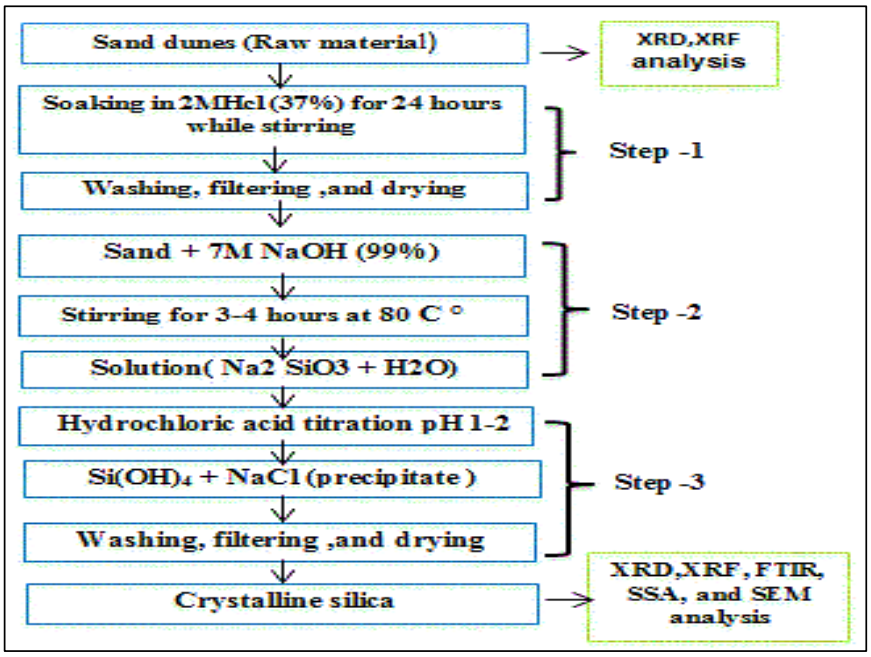


Figure 2: Diagram for the coprecipitation process of producing synthetic silica from sand dunes.

3- Results and Discussion

3-1 Reaction Mechanism

Sodium silicate solid is formed by immersion raw sand in sodium hydroxide and heating to a temperature of 95˚C.

$$2NaOH + SiO_2 \xrightarrow{95^{\circ}C} Na_2SiO_3 + H_2O \quad \text{----------(1)}$$

Sodium Hydroxide — Sand dunes — Sodium silicate — Water

Silicic acid forms when treating of sodium silicate solution with hydrochloric acid

$$Na_2SiO_3 + H_2O + 2HCl \longrightarrow Si(OH)_4 + 2NaCl\text{------(2}$$

Sodium silicate — Water — Hydrochloride acid — Silicic acid — Sodium chloride

Silicic acid is washed with distilled water to release NaCl and dried to forms high purity crystalline silica

Figure 3

$$Si(OH)_4 \xrightarrow{110^{\circ}C} SiO_2 + 2H_2O \text{ ---------(3)}$$

Crystalline silica

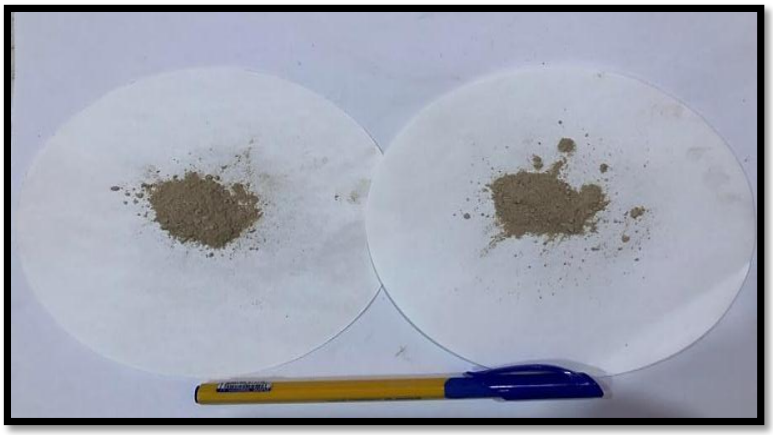


Figure 3: High-purity crystalline silica preparation

3-2 Characterization of Raw Sand

3-2-1 XRD Assessment

Samples were scanned at 2-theta (2-60°) using XRD technique, the XRD characterization of the studied sand (total, medium, and fine size) revealed that quartz, is the predominant mineral in sand followed by feldspar, calcite and gypsum, **Figure 4**. XRD data for the sample that immersed in HCl showed the presence of quartz and feldspar only, **Figure 5**. These results indicate that the immersion process is effective to reduce the amount of calcite and gypsum.

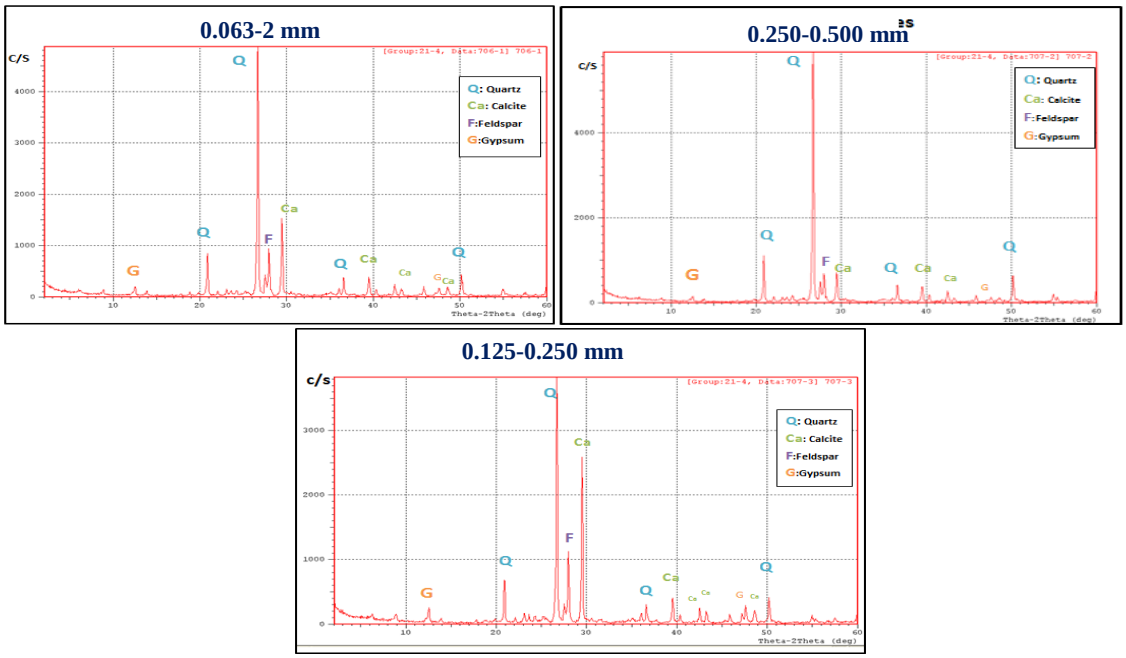


Figure 4: XRD pattern of the raw sand

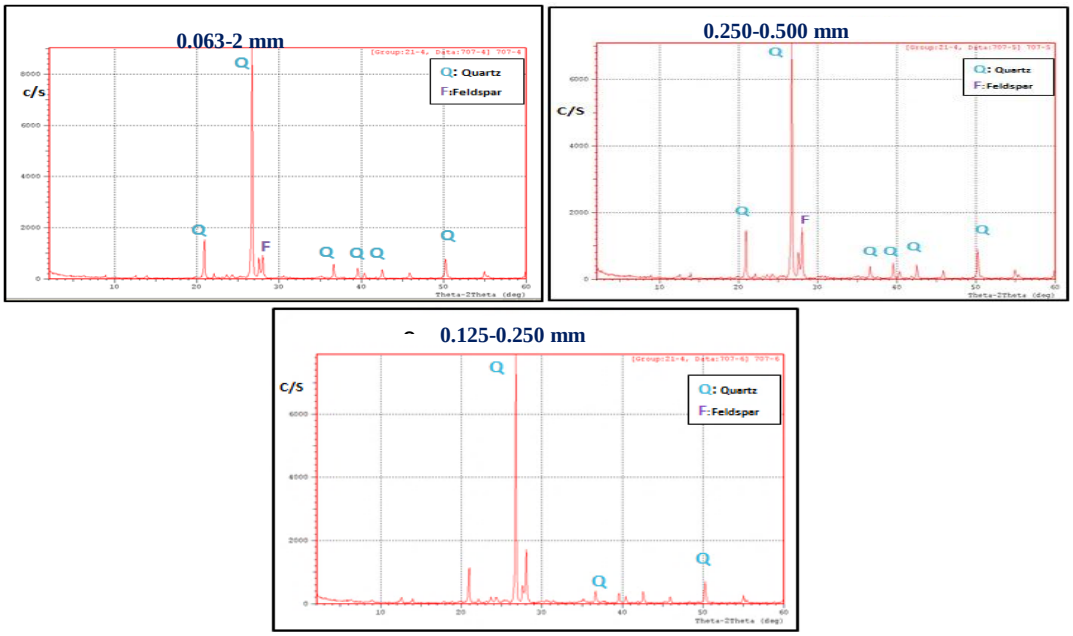


Figure 5: XRD pattern of sand after immersion process

3-2-2 Chemical Assessment

The result of XRF analysis of the raw sand is listed in **Table 1**, **Figure 6**, It indicates that the predominant oxides, are SiO_2, CaO, Al_2O_3, Fe_2O_3, and alkali (K_2O+Na_2O) of 60.81 %, 13.58 %, 6. 81%, 3.05 %, and 2.22 % respectively. While the other oxides tend to be concentrated in the fine sand fractions, except of SiO2 and P2O5 which are abundant in the medium sand fractions. **Table 2**. These statistics indicate that there is an increase of about 17.04 % of SiO_2 content in comparison to its concentrations in raw sand materials (60.81%) after immersion by HCl **Table 3 and 4, Figure 7**, which clearly indicates the dissolved of some oxides (impurities) as a result of the immersion process especially CaO which reduced by 13.04% from the initial content.

Table 1: Raw sand samples chemical analysis (0.063-2 mm).

Name	Oxides (Wt.%)									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	Na_2O+ K_2O
Manziliyah	63.87	7.16	3.16	12.7	0.04	0.09	0.57	0.05	1.47	2.36
Khazina	64.81	5.91	3.07	9.95	0.02	0.10	0.51	0.03	0.99	1.99
Zubadiate	56.76	6.10	3.01	12.95	0.05	0.08	0.29	0.05	0.02	2.01
Chailat	57.81	8.08	2.97	19.1	0.06	0. 09	0.70	0.08	1.70	2.55
Min	56.76	5.91	2.97	9.95	0.02	0. 08	0.29	0.03	0. 02	1.99
Max	64.81	8.08	3.16	19.1	0.06	0.10	0.70	0.08	1.70	2.55
Average	60.81	6.81	3.05	13.58	0.04	0.09	0.517	0.05	1.04	2.22

Table 2: Medium and fine sand size chemical analysis

Name	Fraction	Oxides (Wt.%)									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	Na_2O+K_2O
Manziliyah	**Medium**	66.50	7.03	3.00	8.90	0.02	0.08	0.50	0.03	1.40	1.95
	Fine	50.10	6.91	4.01	18.78	0.05	0.09	0.71	0.08	2.01	1.76
Khazina	**Medium**	70.01	5.99	3.05	7.50	0.03	0.07	0.49	0.01	0.66	2.00
	Fine	45.02	7.07	4.10	20.23	0.04	0.9	0.59	0.06	2.02	2.41
Zubadiate	**Medium**	59.81	6.02	1. 99	15.93	0.03	0.99	0.41	0.99	0.03	2.02
	Fine	47.01	7.01	2.98	21.96	0.03	0.07	0.61	2.01	0.08	2.41
Chailat	**Medium**	66.34	6.98	3.11	9.33	0.04	0.083	0.52	0.02	1.40	2.63
	Fine	49.29	6.76	4.02	18.09	0.029	0.1	0.67	0.05	1.59	2.50

Table 3: Chemical examination of the entire sand particle following immersion

Name	Oxides (Wt.%)									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	$Na_2O+ K_2O$
Manziliyah	84.2	7.0	3.01	0.5	0.02	0.04	0.6	0.03	0.90	2.41
Khazina	76.3	6.9	2.99	0.53	0.02	0.03	0.49	0.02	1.00	2.35
Zubadiate	72.4	6.3	2.6	2.30	0	0.5	0.39	0.04	0.7	1.29
Chailat	78.8	9.1	2.9	0.71	0.02	0.05	0. 71	0.03	1.6	2.73
Min	72.4	6.3	2.6	0.5	0	0.03	0. 39	0.02	0. 7	1.29
Max	84.2	9.1	3.01	2.30	0.02	0.5	0.71	0.04	1.6	2.73
Average	77.92	7. 32	2.87	1.01	0.015	0.15	0.54	0.03	1.05	2.19

Table 4: Chemical examination of medium and, fine sand upon immersion

Name	Fraction	Oxides (Wt.%)									
		SiO_2	$A\ l_2O_3$	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	$Na_2O + K_2O$
Manziliyah	**Medium**	83.4	9.10	3.1	0.70	0.01	0.02	0.49	0.03	0.88	2.6
	Fine	78.41	9.85	5.7	1.00	0.001	0.06	0.79	0.04	1.73	2.91
Khazina	**Medium**	72.30	1.99	2.67	7.21	0.02	0.06	0.41	0.02	1.01	2.31
	Fine	63.7	7.91	4.76	6.8	0.04	0.2	0.69	0.04	1.60	2.83
Chailat	**Medium**	81.30	2.8	2.85	0.59	0.002	0.03	0.48	0.01	0.89	2.65
	Fine	74.20	7.50	3.78	1.51	0.001	0.09	0.70	0.04	1.11	2.46

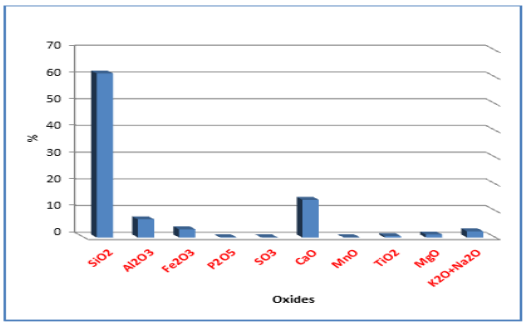


Figure 6: Major oxide average in sand dunes

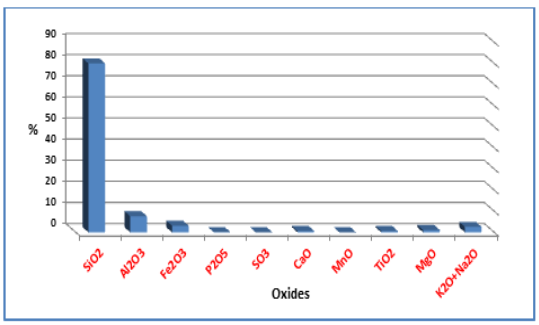


Figure 7: Major oxides after immersion

3-3 Characterization of Crystalline Silica

3-3-1 XRD Assessment

XRD results of synthetic silica **Figure 8** demonstrates that there is no discernible difference between the examined samples' mineralogical composition and their composition following the immersion process. The new phases formed are crystalline silica.

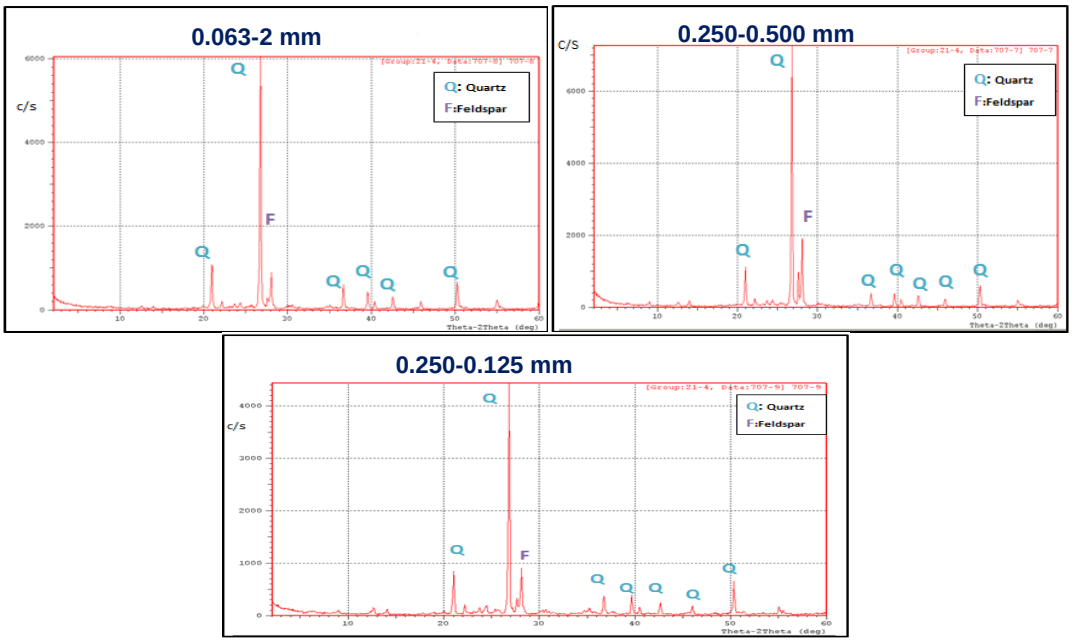


Figure 8: XRD pattern of synthetic silica

3-3-2 Chemical Assessment

Table5, **Figure 9** lists the synthetic SiO2 content. The average SiO2 concentration is 85.37%. With indications of some partially dissolved impurities, the silica in the raw sand increased by 24.01% relative to its average. The most common impurities that are partially released during the purification process are Al2O3 (6.51%), MgO (0.94%), Fe2O3 (2.82%), K2O+Na2O (2.18%), and CaO (1.06%). The highest percentage of synthetic silica was found in Manziliyah medium sand size (87.61%), associated with the impurities Al2O3 (7.1%), Fe2O3 (2.67%), K2O+Na2O (2.44%), CaO (0.62%), TiO_2 (0.61%), and MgO (1.01%). In contrast, Khazina sand had the lowest percentages of extracted silica (83.50%), associated with other oxides (impurities), **Table 6**.

Table 5: Synthetic silica chemical analysis based on the overall size of the sand

Name	Oxides (Wt.%)									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	$Na_2O+ K_2O$
Manziliyah	87.75	6.95	2.88	0.79	0.01	0.03	0.5	0.02	0.91	2.47
Khazina	83.03	8.28	3. 51	0.71	0.01	0.05	0.75	0.03	1.1	2.71
Zubadiate	90.45	1.72	1.17	0.44	0	0.67	0.53	0.01	0	0.45
Chailat	80.25	8.9	3.7	2.3	0.02	0.14	0. 72	0.03	1.7	3.1
Min	80.25	1.72	1.17	0.44	0	0.03	0.5	0.01	0.034	0.45
Max	90.45	8.9	3.7	2.3	0.5	0.67	0.75	0.03	1.7	3.1
Average	85.37	6.51	2.82	1.06	0.14	0.22	0.62	0.02	0.94	2.18

Table 6: Analyzing synthetic silica made from medium-sized fine sand chemically

Name	Fraction	Oxides (Wt.%)									
		SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	TiO_2	MnO	MgO	Na_2O+K_2O
Manziliyah	**Medium**	87.61	71	2.67	0.62	0.03	0.04	0.61	0.03	1.01	2.44
	Fine	78.92	9.73	5.7	1.08	0.03	0.03	0.87	0.04	1.63	2.87
Khazina	**Medium**	83.5	6.1	3.4	0.6	0.02	0.34	0.46	0.03	0.67	2.34
	Fine	68.25	7.92	3.6	1.4	0.02	0.13	0.79	0.05	1.4	2.61
Chailat	**Medium**	86.28	6.99	2.7	0.53	0.03	0.04	0.49	0.03	0.89	2.73
	Fine	78.78	8.98	4.21	1.28	0.03	0.12	0.66	0.05	1.39	2.91

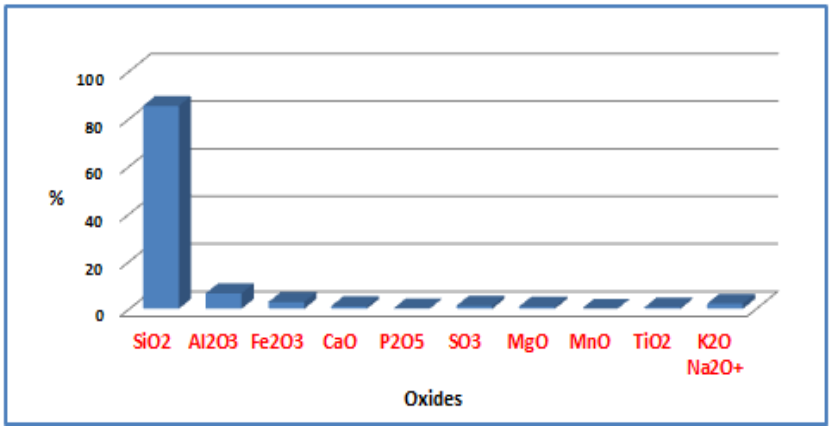


Figure 9: Average of synthesized silica's oxides

3-3- 3 FTIR Assessment

With the aid of FTIR **Figure 10**, the functional groups of the isolated SiO2 were identified. Stretch OH vibrations are characteristic at wave numbers 3425.69–3452.70 cm-1, while bend OH vibrations are typically absorbed at wave numbers 1631.83–1697.41 cm-1 [13]. The symmetrical stretching of Si-OH (Silanol) bonding is the cause of the prominent absorption peak detected at 1080.7 cm-1 [14], The creation of Si-OH bonds, which are left behind when the precipitated silica is heated, indicates the presence of water that binds to silica. The absorption band at wave number 466.1 cm-1 is brought on by Si-O bond tension (siloxy), whereas the Si-O-Si bond (siloxane).detected at 790.8 cm^{-1} [15 and 16].

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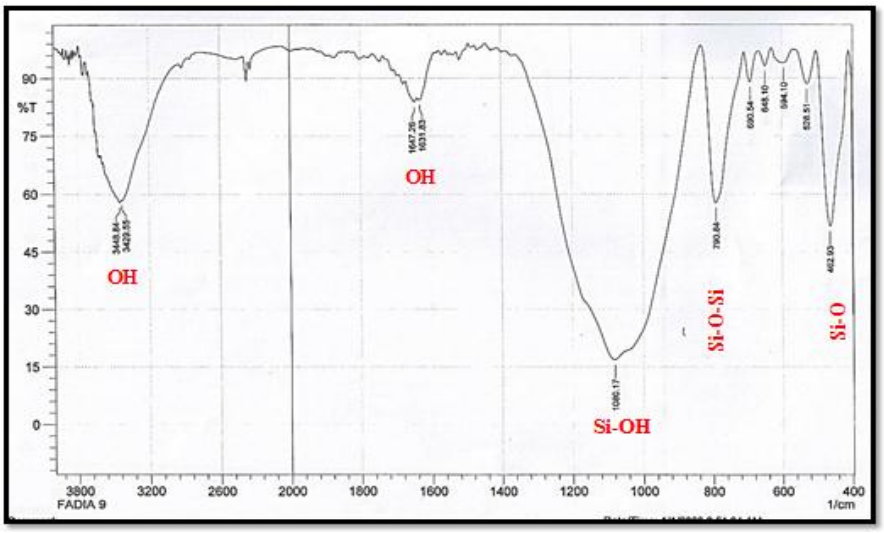


Figure 10: FTIR chart of synthetic SiO_2

3-3-4 Specific Surface Area Assessment

Based on nitrogen gas adsorption at 77 K, the BET method indicates that the synthetic crystalline silica has average specific surface area of 66.51 m^2/g, and average particle size of 8.55 nm, an average monolayer volume of 15.66 cm^3/g, and an average total pore volume of 0.125 cm^3/g, **Table 7**

The specific surface area, total pore volume, and monolayer volume was highest in the synthetic crystalline silica of Zubadiate sand, lowest in the synthetic crystalline silica of Manziliyah and Chailat sand respectively. **Table 7**.

Table7: properties of synthetic silica

Name	Total pore volume (cm^3/g)	Monolayer volume (cm^3/g)	Average particle size (nm)	Specific surface area (m^2/g)
Manziliyah	**0.10**	**10.23**	**10.3**	**45.43**
Khazina	**0.09**	**13.34**	**9.08**	**54.43**
Zubadiate	**0.19**	**24.94**	**7.99**	**108.59**
Chailat	**0.12**	**14.16**	**6.86**	**57.6**
Min	**0.09**	**10.23**	**6.86**	**45.43**
Max	**0.19**	**24.94**	**10.3**	**108.59**
Average	**0.125**	**15.66**	**8.55**	**66.51**

It is practical to categorize pores sizes in the context of physisorption based on the International Union of Pure and Applied Chemistry's (IUPAC) recommendation on their size [17].

1. Micro pores are defined as pores whose diameters do not surpass around 2 nm.
2. Meso pores are defined as pores with a width of two to 50 nm.

3-Macro pores are those whose diameters are greater than or equal to 50 nm.

Mesopores are present in synthetic silica. Adsorbents with pores have a higher selectivity for both adsorption and reactions in addition to having a larger surface area. [18].

3-3-5 Scanning Electron Microscopy Assessment

The SEM images show that the crystalline silica has defined boundaries and a spherical form. The average particle size is 7.86 nm. **Figure 11** Van Der Walls forces act to aggregate the particles, resulting in the agglomerated silica particles. This is consistent with the findings of [9].

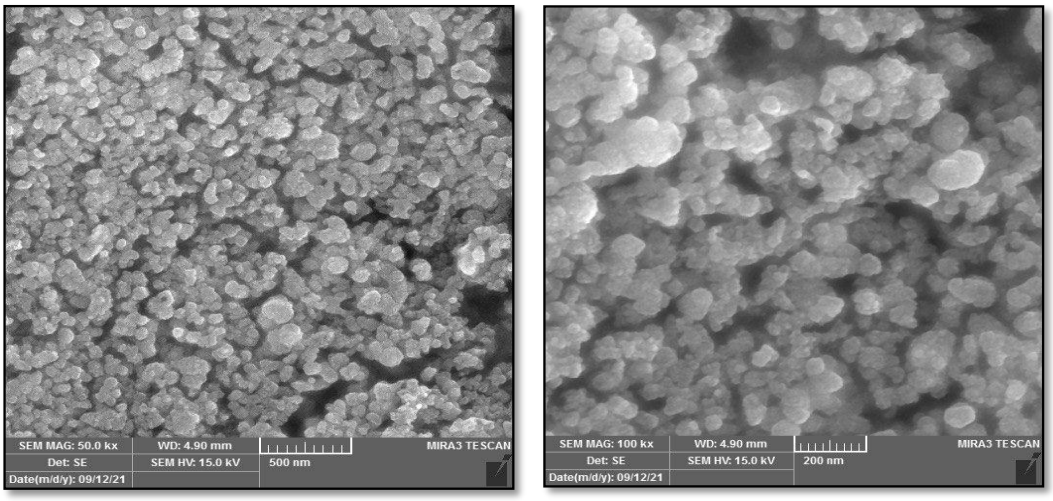


Figure 11: SEM images of synthetic silica.

4-Conclusions

The main conclusions of this study are:

1-The immersion of raw sand materials in HCl is an effective process to dissolved and reduce the impurities especially the CaO. In addition to reduce other oxides to a minimum amount.

2-It is possible to extracted crystalline silica form sand dune, the Si-O-Si and Si-O groups of synthetic silica are distinguished by their typical absorption bands of 790.84 cm^{-1} and 451.36 cm^{-1}, respectively.

3- Agglomerated and spherical crystalline silica particles measuring 7.86 nm were detected by the SEM test, and 66.51 m^2 \ g of specific surface area was revealed by the BET method.

4- The percentage of silica in the fine sand size is low and the percentage of impurities is high so it cannot be relied upon to produce high purity silica whereas the medium sand size is the appropriate size to produce high purity crystalline silica.

5- Manziliyah sand is the best sand for the synthesis of crystalline silica because of low impurities, followed by zubaidate, Chailat and Khazina sand.

6- High purity crystalline silica has been successfully synthesized from four different locations from Missan dune fields, South-eastern of Iraq, including Manziliyah, Khazina, Zubadiate, and Chailat by means of coprecipitation method using NaOH (The percentage of SiO_2 in sands of the study area were increased by the coprecipitation method from 60.81% to 85.37%).

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