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Suitability evaluation of selected sand geological formations for synthesis high purity crystalline silica, Iraq

H A Al-Saad[1], F W Al-Halfi [2], and Y I Khalaf[3]

[1,2,3]Department of Geology, College of science, University of Basrah, Iraq
*Corresponding author email: harith.sukar@uoBasrah.edu.iq ; fadia.waleed@uoBasrah.edu.iq

Abstract:

This study aims determination if the sand collected from Injana and Dibdibba Formations are suitable for use as low-cost raw materials in the manufacture of high-purity crystalline silica. Sand is distributed in vast quantities within the two Formations, which occupy large portions of Iraq's southern desert .Quartz is the predominant mineral phase, followed by calcite, feldspar, and dolomite respectively, based on mineral analysis data. SiO_2 is the main oxide in sand with an average of (82.76%), (90.24%) and the rest of oxides are CaO (6.28%), (1.64%) , Al_2O_3 (4.02%),(3.19%), Fe_2O_3(2.73%), (0.86%), K_2O+ Na_2O (2.05%), (2.51%),SO_3(0.05%), (0.47%) , MnO (0.01%), (0.03%) P_2O_2 (0.23%) ,(0.03%) , and MgO (1.75%), (1.01%) in Injana and Dibdibba Formations respectively ,which are considered as impurities impact the manufacturing properties of sand . The coprecipitation method, which produces high purity crystalline silica at a low cost, involves three steps: the first involves soaking raw sand powder in 2M HCl for 24 hours; The second involves mix purified sand with NaOH while stirring and heating it to 95C° to produce Na_2SiO_3; and the third involves adding 3M HCl to sodium silicate solution to produce $Si(OH)_4$ and drying it to produce high purity of silica .The prepared silica got mineral analysis and chemical analysis. The synthesized silica was in the crystalline phase, according to the results of the mineral characterization. In synthetic silica of Injana and Dibdibba sand, The amount of SiO_2 increased according to chemical analysis, reaching 96.62% , and 97.69% respectively, while all other oxides (impurities) were lowered to their lowest levels. FE-SEM revealed the existence of spherical, well-agglomerated synthetic silica particles within the size 28.6 nmand 24.76nm in synthetic silica particles of Injana sand and Dibdibba sand respectively .

Keywords: Iraq , Sand ,Injana Formation ,Dibdibba Formation, Crystalline Phase , Co precipitation method.

1-Introduction

Iraq is one of many countries which it has a wide desert, it consists of plenty amounts of sand, particularly distributed in the Mesopotamian plain, the Tigris and Euphrates riverbanks, and its branches, geological Formations such as the Dibdibba and Injana Formations which is exposed in the middle and south of Iraq[1] , Injana Formation consists of three layers ,it sandstone, siltstone and claystone, The layers of sandstone are less compact and thicker, Coarsening of sandstone towards the higher Formation, This Formation separates from the above Formation (Mukdadiya Formation) by a layer of pebbly sandstone, The layers of claystone are brown, cracked and contain lenses of siltstone and lenses of sandstone, It contains a veins of secondary gypsum [2],The age is the upper Miocene[3] . The surface-exposed Dibdibba Formation is composed of silt and clay lenses, as well as sandy and sandy-pebble layers[4] ,Sand is spread in economical quantities in Dibdibba Formation(Pliocene), which covers vast

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areas in the southern desert of Iraq, starting from Basrah and passing through in Nasiriya and ending with Najaf and Karbala, these sediments cover an estimated 500 km^2 in the Basrah governorate [5].

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 suitable materials to use [5] , Various methods for obtaining silica have been developed such as emulsions [6] , sol-gel method [7] , Stöber method [8] hydrothermal method [9] , alkaline fusion [10] and co-precipitation, electrocoagulation [11] , The coprecipitation is a technological chemical method containing a solute down until the desired precipitate is formed [12] . When compared coprecipitation method to other conventional methods, this method has the following advantages: It has a high level of purity, the deposition process is straight forward, and it is easy to separate at low temperatures. The amount of time required is relatively short. The equipment utilized is simple and inexpensive, allowing for the production of a powder with a specific crystal size, [13]

2- Study area and sample collection

The fieldwork was carried out on 1-3 August 2022, Topographic (Basic map ,Scale 1.100000) and global positioning system (GPS) , type: (Garmin GPS Map 64s) were used to determining the coordinate of the studied location,The study area were include the Injana and Dibdibba Formations Figure 1.
The Dibdibba Formation samples were taken from three specific sections in the Safwan and Al-Zubair area to the southwest of the Basrah governorate between latitudes (30º 21' 68") N and (30º 09' 15") N, and longitudes (47º 32' 55") E and (47º 40' 25") E. Meanwhile, The Injana Formation samples were taken from three specific sections east of Ali Al-Gharbi city, the Missan governorate, between latitudes (32 °35' 30.11") N and (32 °36' 31.837") N, and longitudes (46° 52' 22.670") E and (46° 53' 20.5") E. The samples were stored in plastic bags labeled with the field data (Sample number and Formation name).

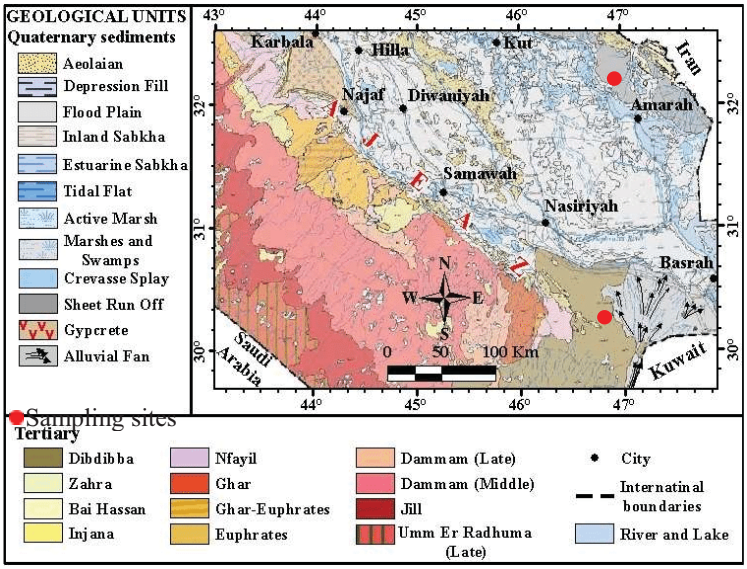


Figure 1: Geological map of the study's area developed by the Iraqi Geological Survey[14].

3-Materials and Methods

The raw material that is used in this study were Injana and DibdibbaFormations sands , 37% hydrochloric acid (HCl), Sodium hydroxide pellets with purity 99% (NaOH) , distilled water. The equipment were baker glass with the size of 250 ml; measuring glass, pipette , funnel, mortar grinder RM 200 , PH meter, thermometer, filter paper (Whatman No 41), oven for drying, sensitive balance and furnace

Six samples were subjected to grain size analysis according to [15] , The sandy portion with size 2-0.063mm was separated from the gravel and mud (silt and clay) using a shaker machine and a set of standard sievesin the Department of Geology / College of Science/ University of Basrah,Iraq .The sand of each sample was analyzed mineralogical by using XRD technique type : PW3830 comprehensive x-ray generator instruments (Department of physical / College of Science/ University of Basrah, Iraq) and chemically by using XRF technique type: (Ed-XRF Instrument Spectro -Xepos of Ametek Company) at Australian Laboratory Services (ALS).

The powder of sand samples were immersed in a 37% solution of 2M HCl for 24 hours. According to [12] , this procedure can help remove contaminants in raw materials. Sand is purified, rinsed with distilled water to neutralize the pH, and then dried at 100 C°for 2 hours , Sand was heated at 95 C° for 3–4 hours using a magnetic stirrer after soaked in 7M NaOH (99%).After that, silicate sodium solution was treated with 2M HCl to produce silicic acid.At pH 1-2, the addition of HCl solution had been stopped [16], Wash the silicic acid with distilled water to remove (NaCl) for (10) times [10] , After filtering, the silicic acid is dried at 110 C° for 4 hours [16] . Then mortar grinder RM 200 using to ground the silica agglomerates into silica powder . In order to analyze the phase formation of the produced silica powder, mineral analysis was used by using XRD technique . Additionally, the X-ray fluorescence technique was used to

determine the elemental composition of synthetic silica . In order to display the morphology and image of the crystal form of produced silica , FESEMs (Field Emission Scanning Electron Microscopes) of the type: FEI NOVA Nano SEM 450 EDS: BRUKER X FLASH 6I30 were used , in Tehran University.

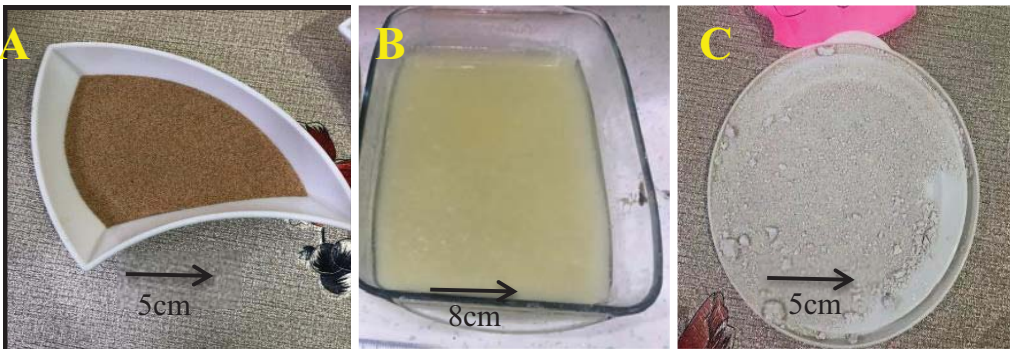


Figure 2: (A; Raw sand , B: Sodium silicate solution ,C: Synthetic crystalline silica) of Dibdibba sand

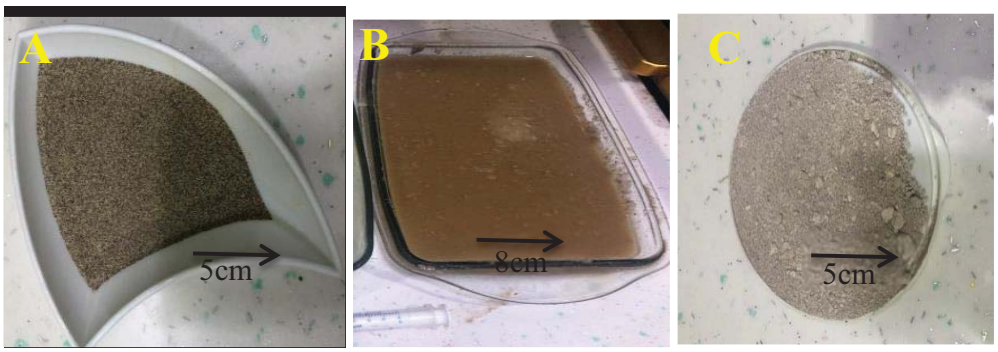


Figure 3: (A; Raw sand , B: Sodium silicate solution ,C: Synthetic crystalline silica) of Injana sand

4-Results and Discussion

4-1 Raw material

A- Grain size analysis

Grain size analysis is mainly used to identify and understand the grain size characteristics and texture of sediments [17], The grain size analysis results of three samples of each Injana and Dibdibba Formations shows , The percentage of the sand (0.063- 2 mm) is within an average 78.74%,82.44% respectively .

B- Mineral analysis

Samples were scanned for a 2 theta (2 -60)° using XRD, quartz is the dominated mineral followed by calcite, and feldspar in sand samples of Dibdibba and Injana Formations ,in addition to presence of small percentage of dolomite mineral in Injana Formation(Figures 4 and 5).

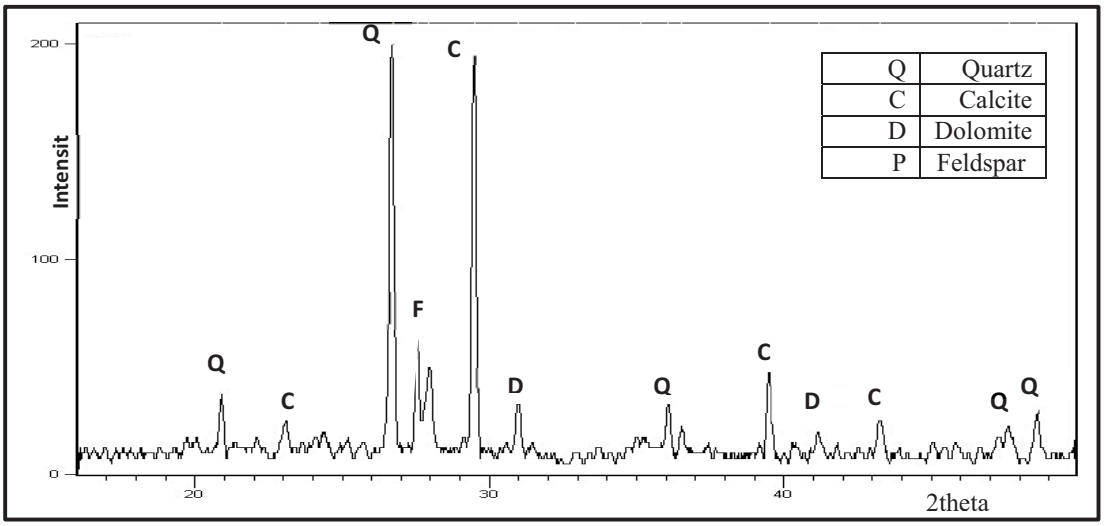


Figure 4: XRD pattern for mineral analyses of sand of Injana Formation.

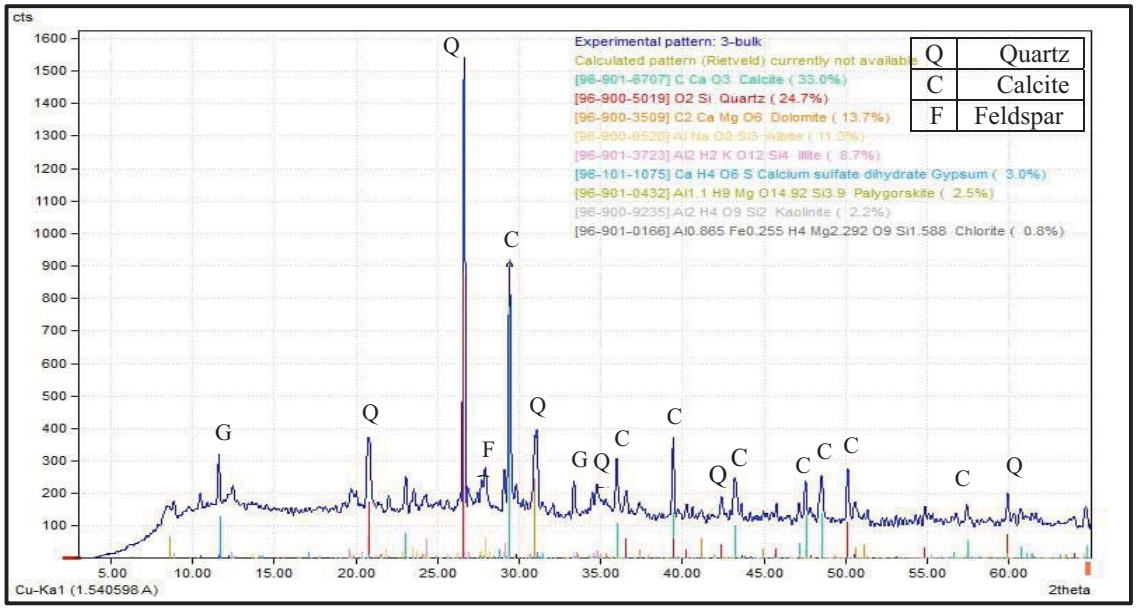


Figure 5: XRD pattern for mineral analyses of sand of Dibdibba Formation.

C-Chemical analysis

Table 1 lists the results of the chemical analysis of the Injana Formation's raw sand sample, which show that the main oxides are SiO_2 (82.76%), CaO (6.28%) , Al_2O_3 (4.02 %) ,Fe_2O_3 (2.73%) , Na_2O+K_2O (2.05%), SO_3(0.05%) ,MnO (0.01%) , P_2O_2 (0.23%) , MgO (1.75%). The results of the chemical analysis of the Dibdibba Formation's raw sand sample, which show that the main oxides are SiO_2 (90.24%), CaO (1.64%) , Al_2O_3 (3.19%) ,Fe_2O_3 (0.86%) ,K_2O +Na_2O (2.51%), SO_3(0.47%) ,MnO (0.03%) ,MgO (1.01%) and P_2O_2 (0.03%).

Table 1 :XRF analysis of raw sand sample

Sample NO		Oxides (Wt.%)										
		SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	MnO	MgO	Na_2O	K_2O	+Na_2O K_2O
Injana	1	82.65	4.02	2.75	6.62	0.04	0.03	0.01	1.92	0.97	1.05	2.02
	2	83.09	3.9	2.56	6.25	0.07	0.07	0.02	1.72	1.01	1.08	2.09
	3	82.55	4.14	2.88	5.98	0.04	0.06	0.01	1.61	0.99	1.07	2.06
Average		82.76	4.02	2.73	6.28	0.05	0.23	0.01	1.75	0.99	1.06	2.05
Dibdibba	1	90.33	3.44	0.82	1.29	0.33	0.02	0.03	0.91	1.13	1.32	2.45
	2	90.45	3.09	0.91	1.87	0.65	0.04	0.04	1.09	1.26	1.35	2.61
	3	89.95	3.06	0.85	1.76	0.43	0.03	0.02	1.03	1.19	1.30	2.49
Average		90.24	3.19	0.86	1.64	0.47	0.03	0.03	1.01	1.19	1.32	2.51

4-2Prepared silica

A- XRD Assessment

XRD results of synthetic silica shows that the new phases formed are crystalline silica , Figure 6

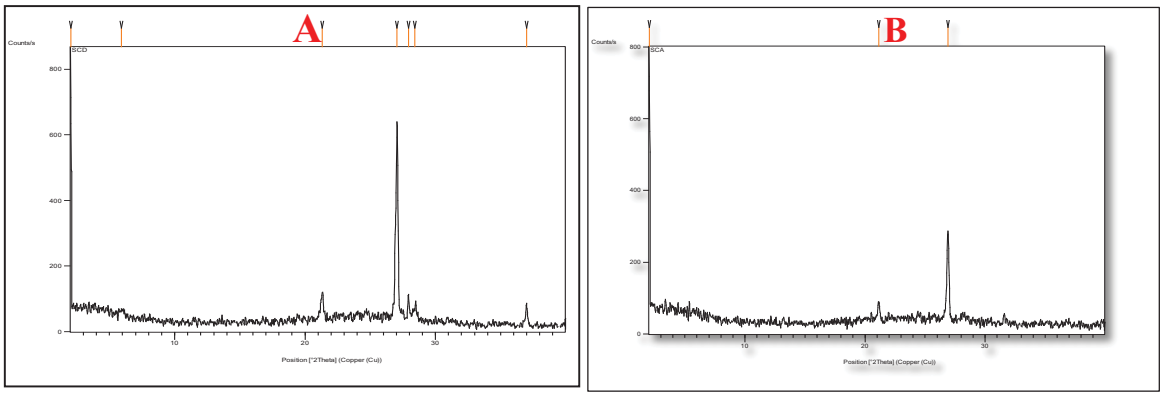


Figure 6: XRD pattern for mineral analyses of prepared silica of **A**: Sand of Dibdibba Formation , **B**: Sand of Injana Formation.

B- Chemical Assessment

The percentage of main oxides in silica prepared from Injana sand is shown in Table 2, The average SiO_2 content is 96.72%, which is approximately 13.9% higher than the concentrations of 82.76% in the raw sand materials. Other oxides, or impurities, are almost entirely dissolved., The average SiO_2 content is 97.69% in silica synthetic from Dibdibba sand , There an increase of roughly 7.45 % compared to the SiO_2 concentrations in raw sand materials (90.24 %), whereas, other oxides (impurities), are nearly completely dissolved.

Table 2 :Chemical analysis of synthetic silica

Sample NO		Oxides (Wt.%)										
		SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	P_2O_5	MnO	MgO	Na_2O	K_2O	$+Na_2O$ K_2O
Injana	1	96.89	0.96	2.03	0.05	0.003	0.001	0	0.03	0.02	0.01	0.03
	2	96.61	1.15	2.08	0.04	0.007	0.001	0	0.02	0.01	0.02	0.03
	3	96.65	1.21	1.95	0.07	0.003	0.002	0	0.01	0.01	0.03	0.04
Average		96.72	1.11	2.02	0.05	0.004	0.001	0	0.02	0.01	0.02	0.03
Dibdibba	1	98.01	1.35	0.31	0.12	0	0	0	0	0. 03	0.02	0.05
	2	97.21	1.83	0.21	0.11	0.01	0	0	0	0.02	0.02	0.04
	3	97.85	1.26	0.12	0.09	0.01	0	0	0	0.03	0.01	0.05
Average		97.69	1.48	0.21	0.11	0.006	0	0	0	0.025	0.017	0.045

C- Scanning electron microscopy

A clear, spherical, and agglomerated form of crystalline silica prepared by coprecipitation method according to SEM images(Figure 7). The estimation indicated the particle size with (28.6 , 24.76) nm in synthetic silica particles from Injanasand and Dibdibba sand respectively. The grain borders were also highly distinct.

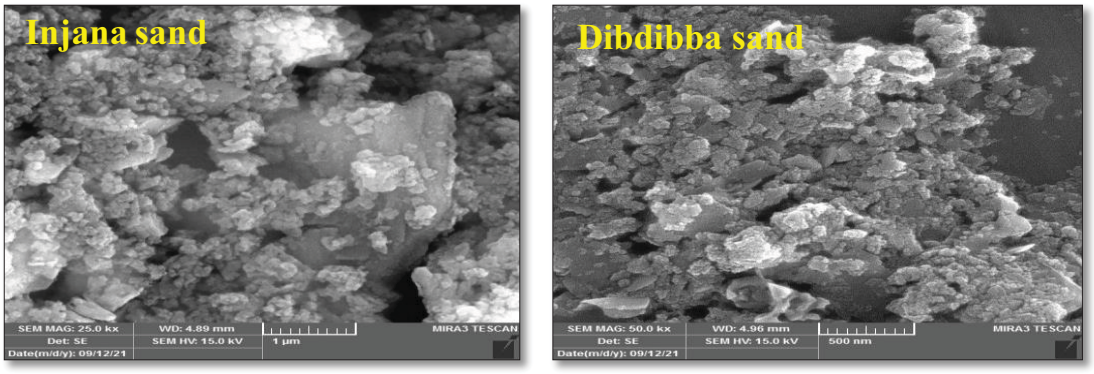


Figure 7: SEM image of prepared silica

5-Conclusion

The present study conclude ;
1-The mineral analysis reveals the prepared silica have crystalline phase.

2- Based on chemical analysis, The percentage of SiO_2 reached of 96.72% and97.69% in prepared silica from Injana sand and Dibdibba sand respectively with the less percentage of impurities.

3- Extracted silica tested by the SEM method showed the presence of agglomerated , spherical particles with a size (28.6 , 24.76) nm in synthetic silica particles from Injana sand and Dibdibba sand respectively.

4- High purity crystalline silica has successfully synthesized from sand of selected sections of Injana and Dibdibba Formations by coprecipitation method using NaOH.

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