

INSIGHT: PREPARATION AND CHARACTERIZATION OF SILICA FROM IRAQI SAND AS AN ECO-FRIENDLY CORROSION INHIBITOR BY USING NANOTECHNOLOGY TECHNIQUE

Samer Noaman Shattab ¹	Kadhim F. Alsultani ²
eng435.sammer.noaman@student.uobabylon.edu.iq	<u>finteelalsultani@gmail.com</u>
^{1,2} Chemical Engineering Department, College of Engineerin	ng, University of Babylon, Iraq.

ABSTRACT

Corrosion is a major challenge in all industrial sectors, prompting the scientific community to search for effective solutions that can stop or reduce corrosion attacks. Inhibitors (both synthetic and natural) have emerged as the ideal solution to this challenge. In this context, nano silica has been found to be a promising material due to its unique environmentally friendly properties and high surface area, capable of forming interconnected protective layers. In this study, nano silica was prepared from Najaf sand, Iraq, using the sol-gel technique, which allows precise control of particle size and porous structure. Tests were conducted to characterize the distinctive nature of nano silica. FTIR analysis revealed the presence of effective functional groups such as Si-O-Si. The chemical composition of the silica sample was examined. The results showed that the silica extracted from Najaf sand has high purity (up to 96%), enabling it to achieve high energy efficiency based on the strength of its bonds under hostile conditions, as measured by XRD. This outstanding performance is attributed to the nanoparticles' ability to diffuse effectively, as measured by surface morphology (SEM). The research confirmed the synergistic role of nanotechnology in enhancing the effectiveness of silica, as the ultrafine particles contributed to improving selective interactions with corrosive agents. The study concluded that nano-silica prepared from Najaf sand is a highly efficient and low-cost corrosion inhibitor, supported by the abundance of local raw materials. The study recommends the use of silicate inhibitors in the oil and infrastructure industries, with further research being conducted to exploit their effectiveness under various operating conditions, which will contribute to reducing economic losses and establishing principles of environmental sustainability.

Keywords: Nano-silica, High-silica Iraqi sand, Nano-silica inhibitors, Silica steel protection.

1- INTRODUCTION

Carbon steel with varying carbon levels (0.05%-0.25% low, 0.25%-0.6% medium, and 0.6%-1.7% high) remains the most widely used material in the construction and

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development of industrial

machinery, buildings, and pipelines. Compared to other metals, carbon steel is less expensive, has an efficient and easy design, and possesses special properties including hardness, ductility, and flexibility [1]. Therefore, corrosion-related equipment failure is a major issue, causing billions of dollars in losses across many industries, most notably the oil and gas industry. In the United States alone, annual costs associated with corrosion are expected to exceed \$500 million [2]. The cost of corrosion worldwide was estimated in a 2013 study by NACE International at approximately \$2.5 trillion, or 3.4% of global GDP. Pipeline corrosion in the oil and gas sector increases the risk of spills and environmental damage. Corrosion is estimated to cost the oil and gas production sector \$1.372 billion annually [3, 4]. The reasons are: - [internal corrosion (53.2%), external corrosion (11.7%), damage caused by other factors (15.2%), welding (3.2%), installation defects (3.3%), overpressure (0.5%), other factors (10%)].

Since carbon steel is known to be in constant contact with a variety of corrosive agents throughout the industry, this has been reduced by using a variety of corrosion inhibitors, such as organic and inorganic compounds, metal oxides and natural plants to increase the effectiveness of corrosion inhibition in a safe and effective manner without releasing any harmful residues. Crude oil contains a wide range of strong corrosives, such as chloride salts such as (NaCl, KCl, CaCl2 and MgCl), and harmful (corrosive) gases such as sulfuric acid and hydrogen sulfide (H2S, HCL). These agents are known to be present in crude oil from wells to refinery [7,8,9]. These causes are more effective and dangerous when the appropriate conditions for corrosion are available. The complex reactions caused by temperature increase the concentration of dissolved oxygen ions in the medium, making the metal susceptible to chemical attack that eventually leads to deformation of the metal and loss of its weight, thus causing catastrophic environmental effects on the nature of the process over time. Whether the causative element produces basic or acidic reactions. As a result, these media and causes will attack the target metal [10].

Corrosion begins in the anodic regions of the surface in the form of migrating ions. After leaving the anodic surface, electrons migrate through the metal structure to adjacent cathodic sites, where they combine with water and oxygen to form hydroxyl ions. These hydroxyl ions combine with iron ions to generate ferrous hydroxide, which oxidizes to form hydrated ferric oxide or rust [11-14] at the anodic sites or even at the cathodic site, depending on the nature of the corrosion process. Conversely, corrosion continues if additional anodic sites form in the adjacent regions. The electrical circuit that produces corrosion depends on the release of electrons at the anodic sites and their receipt at the cathodic sites [15-18]. Figure 1 reveals the reactions leading to corrosion.

The scientific community, recognizing the complexity of the problem, has begun to play an important role in limiting the causes of corrosion by halting the actions of the

destructive elements. Since corrosion is the true expression of metal deterioration in the environment, despite our best efforts to stop it, researchers have shown that the rate of corrosion can be reduced to low levels, but it cannot be completely prevented. This can be achieved by improving the corrosion resistance of metal surfaces using active inhibitors. Furthermore, traditional corrosion control measures often rely on the use of toxic and hazardous chemicals, which pose significant risks to the health of workers and the environment. Therefore, there is a need to develop sustainable corrosion inhibitors that are effective, low-cost, and environmentally benign [19]. Recently, these inhibitors have emerged as a relatively new and rapidly growing field of research [20], aiming to provide safer alternative methods for corrosion control. They are derived from natural and renewable resources and designed to have minimal adverse effects. This paper aims to provide a comprehensive overview of the most important nanotechnology-based studies on the use of sustainable corrosion inhibitors for the period 1968–2025 (see Table 1). In the current study, Najaf sand (Iraq) was used as a substrate to prepare a nano-silica-based corrosion inhibitor. The study described the role of the prepared silica through conducting some tests such as (FTIR, SEM, AFM, XRD), in addition to examining the silicate and mineral components of the sample, which was tested in a 1 M hydrochloric acid environment. It is worth noting that the Najaf environment, especially the western plateau region, contains quantities reaching billions of cubic meters of sand containing a high concentration of silica. Hence, the importance of conducting research and focusing on the role of silica using nanotechnology in creating a safe corrosion inhibitor in acidic environments.



Fig.1. reveals the corrosion phenomenon on the surface of carbon steel.

Table1. History and description of developments in the uses of silica nanoparticles (1968

- 2025).

Year	Discretion
1968-1983	The introduction of monodisperse silica nanoparticles and the first description of the Strober method occurred.
1983-1998	It was realized and demonstrated that surface-modified porous silica might be used for drug delivery. Structured porous silica, which includes periodic porous organic silica (PMO), amorphous silica (SBA), and structured matter (MCM), is a novel family of porous materials that first appeared in the early 1990s.
1998-1999	The capacity of microporous silica (SBA-15) to adsorb and release proteins from the porous structure was originally demonstrated.
2001-2003	identified SNP's controlled drug release property and presented MCM-41 as a possible drug carrier for the first time.
2003-2005	SNPs with microspores, such as IBN (called after the Institute of Bioengineering and Nanotechnology (IBN)), were made possible by porosity modification thanks to further advancements in synthesis procedures. SNPs were later shown to have further used in imaging
2006 -2018	Further advancements in materials science led to the invention of dendritic silica and ultra-small silica cages.
2024-2025	Technological development has been directed towards the use of nanotechnology from natural sources and in sustainable ways, especially in corrosion issues, as corrosion is the main cause of the destruction of all uses of steel and other industrially used metals that are susceptible to corrosion.

2- Materials and Method

2.1- Materials

2.1.1- Chemicals Materials

To prepare silica using the sol-gel method, the following chemicals were used in the present work (Table 2).

Materials	The manufacturing company and Country of origin	Purity
NaOH	Chopping and Williams/United Kingdom	40 %
H ₂ SO ₄	Sigma Aldrich	98 %

Table.2 Materials used for nano-silica synthesis.

HCl	India/ CDH	36%
Deionized water	Chemical Engineering Lab. / University of Babylon	High purity
Natural sand	Soil of Najaf land, Western Plateau / Iraq	Natural contains impurities

2.1.1-Basic material (Iraqi sand)

The researchers provided local sand as a base material for nano silica in cooperation with Kar Al-Najaf Cement Company. This sand was available in quantities estimated at billions of cubic meters in the area (Western Plateau of Najaf Governorate / Iraq). A raw sample of sand was taken for the purpose of examining the chemical components. Table 3 shows the chemical analysis of the sand using XRF analysis.

Table 3. Chemical composition of the raw sand by using XRF analysis.

Component	SiO2	Al2O3	Fe2O3	Na2O3	LOI	Other
Wt%	95.7	0.4	1.41	0.05	1.25	1.19

2.2. Method

The sand was subjected to washing with normal and distilled water several times with continuous stirring to remove dust. After that, the sample was dried at 120 °C for 5 h. Then the sample was washed with dilute hydrochloric acid solution with a concentration of 0.1 M for 20 min with continuous stirring using a magnetic stirrer bath to remove suspended impurities. This step was followed by washing with deionized water and drying for 5 h at a temperature of 110 °C.

The nano-silica was prepared in the present work according to the practical scheme (Figure 2), using the top-down approach to prepare nanoparticles. 60 grams of high purity sand (Najaf, Iraq) resulting from the previous step were mixed with 80 grams of caustic soda (Kuwait / Kuwait Industrial Projects / Najaf Refinery) well and then the product was heated at 400 degrees Celsius for approximately 35 minutes until water bubbles appeared from the product, indicating the completion of the reaction to produce sodium silicate hydrate eq1.

$$Sand(natural) + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$
(6)

This was followed by cooling the product and then the resulting sodium silicate was dissolved in 600 ml of deionized water with continuous stirring for 20 minutes to achieve complete dissolution. The product was filtered and collected in a 1000 ml container, followed by the gradual addition (drops) of concentrated sulfuric acid ((analytical grade, Fluka Company, Germany)) until white silica gel eq2 was formed.

 $Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2(gel) + Na_2SO_4 + H_2O$ (7)

The final product was washed several times with deionized water for several times to extract pure silica, finally the final product was left to dry at 110°C for 24 hours in the laboratory (College of Chemical Engineering / University of Babylon / Iraq). The final product is pure silica extract powder prepared by sol-gel method (see Figure 3). The nano size was obtained, where 15 grams of the resulting pure silica powder were taken to be ground for 12 hours, to obtain a nano size of 95.9 by a nano ball mill (NQM-0.4 MODELPLANETARY BALL MILL) at the Nanotechnology and Advanced Materials Research Center (Iraq / Baghdad / University of Technology).



Fig.2. Schematic diagram of the preparation process of nano-silica by the sol-gel method.



Fig.3. Graphic summary of the preparation stages of nano-SiO₂ by sol-gel.
(a): natural sand, (b): mixing of solution sodium silicate (Na₂SiO₃), (c): silica gel, (d): filtration stage, (e): product pure silica (SiO₂).

2.2.1- Characterization of nanosilica

The tests were carried out to characterize nanosilica in this work, as follows. The silica content and the number of mineral impurities in the samples were measured by XRF Philips spectrometer (model PW 2404, Philips, Amsterdam, Netherlands/Cement Car Factory Company Laboratory, Najaf), see Table 2. The Fourier transform infrared spectroscopy device (Shimadzu, IR Prestige-21), (German origin/Laboratory of Nanotechnology and Advanced Materials Research Center/Technological University) was used to determine the functional groups of nanosilica from 4000 to 400 cm-1 in the wavenumber range using Original graphical software. The X-ray diffraction (XRD) pattern of the sample was recorded on an X-ray diffractometer (40 kV, 30 mA) (Chinese origin/Laboratory of Nanotechnology and Advanced Materials Research Center/Technological University). AFM was used to examine the prepared silica nanoparticles to measure the average particle size and particle size distribution. Scanning Probe Microscope (SPM AA3000-Scanning Probe Microscope, USA/Laboratory of Nanotechnology and Advanced Materials Research Center/Technological University) was used. SEM (origin from China/Laboratory of Nanotechnology and Advanced Materials Research Center/Technological University) was performed on the resulting silica sample to determine the surface morphology of the solgel-blocked silica nanoparticles.

3- Results and Discussion

X-ray Diffraction (XRD) Analysis

Upon examination, X-rays were observed to spread in multiple directions, forming a large, hill-like protrusion with a peak in the range $2\Theta = 15^{\circ}-30^{\circ}$, indicating the presence of an amorphous structure and a highly disordered form of silica. The amorphous phase would predominate. This interpretation is consistent with reference [20].

Figure (4) shows the X-ray diffraction pattern of nanosilica prepared by the sol-gel method. The absorption pattern confirms the amorphous phase of nanosilica, with the absence of sharp, strong peaks and a broad, high-intensity diffraction peak at $22.52\Theta = 22^{\circ}$; this result is almost identical to the findings of references [14-21].



Fig.4. XRD test analytical of the nano-silica sample.

Scanning Electron Microscope (SEM) Analysis

The surface morphology and structural measurements of the raw silica sand were examined using a scanning electron microscope (SEM). As shown in Figure 5a, b, SEM images of the unit structures, which are amorphous aluminosilicate materials, reveal irregular size and structure. On scanning electron microscopy (SEM) (halite, sodium chloride), silica nanoclusters and a cube-like structure can also be seen (Figure 5a). The process of work has been supported by several studies [13,14,15]. This halite crystal was formed as a by-product of the preparation of nano silica powder [16,17]. Halite was found in the final nano silica powder sample (Figure 5b) after washing because some sodium chloride remained, and these primary particles tended to aggregate. Comparison of the SEM and XRD pattern analysis results reveals that the silica is amorphous nano silica [22]. The particles have a spherical shape with an average size of 26.75–28.93 nm and a porous appearance. This interpretation is consistent with studies by references [22–25].



Fig. (5a, b). Scanning electron microscope of raw sand and nano silica prepared by solgel method.

Atomic Force Microscopy and Average Particle Size (AFM)

Atomic Force Microscopy (AFM) was used to detect the average diameter of SNPs. A high resolution of (444 * 452) pixels and a scanned area of (1622 * 1651) nm² were used to obtain the sharp topography of the NS. As a result, the average diameter of the prepared nano silica was 95.9 nm [DLS brook haven 90plus-USA] (see Figure 6). Figure (7-a) shows the granule aggregation distribution diagram and particle size distribution. The surface topography of the nano silica can be clearly seen in this research in the 2D and 3D images, which showed a non-spherical shape and small, agglomerated grains of the nano silica particles, as shown in Figures (7b, c), respectively. This result is consistent with references [26-27].



Fig.6. Results of particle size analysis of silica prepared by sol-gel method after grinding for 12 hours.

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Fig.7. (a) Particle size distribution of NS prepared by sol-gel method, (B,C) 3D images of the prepared NS.

FT-IR Analysis

Figure (8) shows the FT-IR spectrum of NS for each sample obtained from Iraqi silica sand using the sol-gel method, by gradually adding concentrated sulfuric acid to the filtrate of sodium silicate solution. The examination reveals the chemical composition of the nano silica product. The spectrum shows a broad, more intense band at 1047 cm⁻¹, typical of the Si-O-Si asymmetric stretching vibration, consistent with [25, 26, 27]. This band is less intense, but on the other side of the spectrum, relative to the range (438-795) cm-1, which

is due to the symmetric stretching vibrations (Si-O-Si). These are the characteristic solid lines of silica, consistent with [28, 29]. Water molecules adsorbed on the SiO₂ surface cause the -OH peak to broaden, with a peak at 2818-3391 cm⁻¹. The bending vibration of -OH bonds in water molecules adsorbed on the nano silica surface is responsible for the hydroxyl group stretching frequency and the peak at 1631 cm⁻¹, the bound water in the resulting silica. This is consistent with the results of references [28-31]. The above readings are consistent with the chemical analysis as shown in Table (4).



Fig.7. FTIR spectra of silica nanoparticles from natural sand.

Wavenumber(cm ⁻¹)	Assignment	References
438-795	(Si-O-Si) symmetric stretching (siloxane group)	[25,26,27]
1047	1047 Si-O-Si asymmetric stretching	
1631	H ₂ O	[30]
2818-3391	-OH, bending vibration	[30]

4- Discussion

With the availability of high-quality sand in the Western Plateau/Najaf Ashraf region, Iraq, with a silica content of up to 95.7%, the synthesis of silica nanoparticles from local raw sand is an environmentally friendly and cost-effective process. Silica sand is a good alternative raw material that can be used as a source of silica and chemically processed to produce silica nanoparticles. The surface area of the chemically processed silica nanoparticles was significantly higher than other nanoparticle production methods. Atomic force microscopy (AFM) examinations of the prepared samples revealed that the average

particle diameter of the silica nanoparticles was less than 100 nm, confirming the validity of the approach to preparing nano silica from the silica peak. XRD experiments revealed that the silica nanoparticles were amorphous in shape. Scanning electron microscopy (SEM) data revealed a spherical shape for the produced silica nanoparticles, with a size ranging from 26.57 to 28.93 nm. FTIR spectra also showed the SNPs at 1047 cm¹. Comparing the SNPs' spectra with the literature revealed that they were fully consistent with the SNPs under study, ensuring the authenticity of the sol-gel-prepared nanomaterials. FTIR spectra confirmed that the produced amorphous silica nanoparticles were following the standard.

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