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Abstract

The raw material sources and reducing processing costs for silica production remain among the most significant challenges facing researchers nowadays. This study aims to evaluate the feasibility of utilizing two widely available natural resources in El Oued Province, Algeria—sand rose and aquifer sand—as local sources for preparing amorphous silica using a sol-gel method. To achieve this, the raw materials and extracted silica were characterized using a various of techniques, including X-ray fluorescence (XRF), conductivity measurement, surface properties (BET), X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), and Fourier transform infrared spectroscopy (FT-IR). The XRF analysis revealed the presence of silica in both samples, with an SiO2 content of approximately 87.95% in the aquifer sand and only 1.208% in the sand rose. XRD and FT-IR results confirmed the presence of silica in the form of quartz, with the appearance of sharp, distinct peaks at $2\theta = 26.65°$ and 1082 cm^{-1}, respectively. Following extraction, the results showed that amorphous silica was obtained from the aquifer sand, characterized by a low electrical conductivity of 2.7 µS/cm and a specific surface area of 485.2 m^2/g. The XRD pattern also revealed a broad, centered band at $2\theta = 22.6°$, further confirming the amorphous nature of the extracted silica. These results indicate that sand rose is an unsuitable source for silica extraction due to its low silica content and limited extraction yield, while aquifer sand represent a promising and attractive local source of silica, both in its crystalline form as quartz and in its amorphous form after extraction, making them a potential feedstock for numerous industrial applications.

Keywords: Sand rose, amorphous silica, sol-gel method, quartz.

الملخص:

لا تزال مصادر المواد الخام وخفض تكاليف عمليات المعالجة لإنتاج السيليكا من أبرز التحديات التي تواجه الباحثين في الوقت الراهن. تهدف هذه الدراسة إلى تقييم إمكانية استغلال مصدرين طبيعيين متوفرين على نطاق واسع في ولاية الوادي، الجزائر، وهما وردة الرمال ورمال الخزان الجوفي، كمصادر محلية لتحضير السيليكا غير المتبلورة باستخدام طريقة سائل-هلام. ولتحقيق ذلك، تم توصيف المواد الخام والسيليكا المستخلصة باستخدام مجموعة من التقنيات، شملت فلورية الأشعة السينية (XRF)، الناقلية الكهربائية، قياس خصائص السطح (BET)، حيود الأشعة السينية (XRD)، المجهر الإلكتروني الماسح المزود بمطيافية الأشعة السينية المشتتة من حيث الطاقة (SEM-EDX)، ومطيافية الأشعة تحت الحمراء بتحويل فورييه(FT-IR) .

أظهرت نتائج تحليل XRF وجود السيليكا في كلتا العينتين، حيث بلغت نسبة SiO_2 نحو 87.95% في رمال الخزان الجوفي، في حين لم تتجاوز 1.208% في وردة الرمال. كما أكدت نتائج XRD وFT-IR وجود السيليكا في صورة الكوارتز، من خلال ظهور قمم حادة ومميزة عند $2\theta = 26.65°$ و1082 سم$^{-1}$، على التوالي. بعد عملية الاستخلاص، أظهرت النتائج الحصول على سيليكا غير متبلورة من رمال الخزان الجوفي، تميزت بناقلية كهربائية منخفضة بلغت 2.7 ميكرو سيمنس/سم ومساحة سطحية نوعية قدرها 485.2 م ²/غرام. كما أظهر نمط XRD ظهور حزمة عريضة متمركزة عند $2\theta = 22.6°$، مما يؤكد الطبيعة غير المتبلورة للسيليكا المستخلصة. تشير هذه النتائج إلى أن وردة الرمال تُعد مصدرًا غير ملائم لاستخلاص السيليكا نظرًا لانخفاض محتواها منها ومحدودية مردود الاستخلاص، في حين تمثل رمال الخزان الجوفي مصدرًا محليًا واعدًا وجذابًا للسيليكا، سواء في صورتها البلورية ككوارتز أو في صورتها غير المتبلورة بعد الاستخلاص، مما يجعلها مرشحة كمادة أولية محتملة للعديد من التطبيقات الصناعية.

الكلمات المفتاحية: وردة الرمل، السيليكا الغير متبلورة، طريقة سائل-هلام، الكوارتز.

Dedication

To my beloved mother and father,
who have been the light that illuminated my path, and the reason I have reached this stage,
through their prayers, patience, and continuous support.
May Allah protect them and reward them with all that is good.

To my beloved husband,
who has been my true support throughout this journey,
who never ceased to support and encourage me,
with his patience and presence by my side in every circumstance.
I dedicate this work to you as a gesture of loyalty and gratitude.

To my precious daughter,
the most beautiful gift in my life,
the one who has given me strength to continue on this path.
May Allah grant her lasting health and happiness.

To my esteemed professors,
who enriched my path with knowledge and understanding,
and whose guidance was invaluable to me.
May Allah reward them with the best of rewards.

To everyone who contributed, whether near or far,
to the completion of this work,
I dedicate the fruit of my efforts with all my love and gratitude.

Amal Abid

Dedication

In the name of Allah, my Creator, the One who
makes my affairs easy and protects me.
To Him belongs all praise and gratitude.

I dedicate this achievement first to myself, and then to
everyone who supported me and helped me complete this journey.
May you always remain my source of strength and support.

To the one whose name I carry with pride and honor, to the one
who removed the thorns from my path and paved the way for me
toward knowledge, after the grace of Almighty Allah —
everything I have achieved is owed to my dear father, the man
who spent his entire life striving so that I could become better
than him. May Allah grant him a long life.

To the one beneath whose feet Allah has placed Paradise,
whose prayers were the secret behind my success, whose
tenderness healed my wounds, my role model,
my first teacher, and my companion through life —
my beloved mother.

To those who have always been, and continue to be,
my support and the light of my life —
my dear sisters and my beloved brother.

To my companion on this journey and my life partner,
my beloved husband; to the one who generously gave me his
time and honored me with his kindness, who has always been
and continues to be supportive of me in every step,
and who has been my strength and source of help
throughout my life.

To those who supported me and surrounded me with
love and tenderness, and who treated me as their daughter —
my second family, my husband's family.

And to Allah belongs all thanks for granting me
the strength to reach this moment.

"All praise is due to Allah, Lord of the worlds."

Amira Rekabi

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LIST OF Abbreviations

BET	Brunauer, Emmet and Teller
BJH	Barrett-Joyner- Halenda method
EDX	Energy- dispersive x-ray spectroscopy
$FTIR$	Fourier Transform Infrared
SEM	Scanning electron microscope
XRD	X-Ray Diffraction
XRF	X-ray Fluorescence
gr	Gram
k	Kelvin
κ	Conductivity
m	Meter
nm	Nanometer
μm	Micrometer
$\mu s/cm$	Microsiemens per centimeter
$wt, \%$	Weight percent (or mass percent)

CHAPTER 1

GENERAL INTRODUCTION

1.1 Background of the study

Algeria is abounded by vast desert regions, with the Sahara Desert comprising approximately 80% of its total area[1]. The El Oued region, located in southeastern Algeria, is renowned for its sandy environment, characterized by expansive dunes and unique geological formations.Sand roses and aquifer sand are among the natural resources found in this region under study. Sand roses are typically associated with gypsum-rich formations and may contain other minerals, including quartz, calcite, and other elements, depending on their location and geological origin[2]. Despite their abundance and distinctive properties, locally available sand roses have not received sufficient attention as a potential raw material for the production of value-added silica materials. The use of locally available materials as sources of silica production not only reduces production costs but also promotes the sustainable use of natural resources.

Silica, or silicon dioxide (SiO_2), is an inorganic compound composed primarily of the elements silicon and oxygen. It has garnered increasing attention from researchers and industrialists due to its unique physical and chemical properties, making it an important material in numerous technological and industrial applications[3]. Silica is widely found in many natural sources and biological materials, such as rice husks, fly ash, clay, sand, and other silicon-rich materials. It exists in both crystalline and amorphous forms. Crystalline silica can occur in various forms, including quartz, cristobalite, tridymite, stishovite, and cosite[4]. Crystalline silica is relatively more toxic due to its ordered crystal structure and strong fibrinolytic activity, while amorphous silica generally exhibits less biological reactivity and a lower potential for carcinogenesis[5].

Several techniques have been reported for preparing silica from natural materials. These include co-precipitation[6], hydrothermal synthesis[7], and the sol-gel process[8]. The latter is among the techniques that have garnered significant attention due to its simplicity, efficiency, and adaptability to various raw materials, despite employing so-called acid-base treatments. Acid treatment can be used to remove mineral impurities and dissolved mineral phases, while alkaline treatment facilitates the conversion of silica into soluble silicate species. Subsequent acidization of the silicate solution can lead to the formation of silica gel, which can then be washed, dried, and heat-treated to obtain silica[9]. The efficiency of this process depends heavily on several factors, including the concentration and type of chemicals, the solid-to-liquid ratio, the reaction time, the temperature, the washing conditions, and the drying procedures. Therefore, optimizing the extraction process is essential to obtaining silica with specific properties.

Although the aforementioned sources, namely sand rose and aquifer sand, are available in the El Oued region of Algeria, interest in exploiting these materials as potential raw materials for silica production remains relatively limited. Therefore, studying these materials could contribute new scientific information regarding their mineral composition, their suitability for silica extraction, and the properties of the resulting product. Furthermore, developing a suitable preparation method would reduce reliance on traditional silica sources and encourage the use of locally available materials.

Accordingly, this study focuses on the preparation and characterization of silicon dioxide from sand rose and aquifer sand samples taken from the ElOued region of Algeria. The study aims to investigate the feasibility of extracting silica from the selected natural material using a liquid-gel method, and to characterize the physical and chemical properties of the raw materials as well as the extracted silica. This will be achieved using integrated analytical techniques to determine its composition, structure, form, and functional properties. The results are expected to contribute to understanding the potential of sand rose and aquifer sand as a local and economically attractive source of silica, and to promote the optimal exploitation of the natural mineral resources available in the ElOued region.

1.2 Problem statement

Silica (SiO_2) is an inorganic material that has attracted the attention of many researchers due to its unique chemical and physical properties, enabling its use in numerous medical, industrial, and environmental applications[10]. However, producing high-purity silica through extraction processes often requires suitable raw materials and processing that can consume significant quantities of chemicals and energy, potentially increasing production costs and limiting the widespread application of these processes.

In this context, local sandy materials, particularly rose sand and aquifer sands, represent readily available natural resources that can contain substantial amounts of silica in the form of quartz. However, the nature of these raw materials and their composition, often associated with other compounds such as gypsum and calcite, can make silica extraction and purification a challenge requiring the adoption of an appropriate and efficient protocol. Furthermore, the varying nature and composition of each source can affect the extraction yield and the purity of the resulting silica.

Therefore, there is a need to study the feasibility of valorizing these local sand resources by developing a suitable silica extraction process, aiming to minimize chemical and energy consumption, and monitoring the various stages of extraction and purification using appropriate analytical techniques, ultimately obtaining silica with properties and purity suitable for potential applications.

Accordingly, the research problem revolves around the following main question:

To what extent is silica extractable from sand rose and raw aquifer sand?

1.3 Objectives

Silicon dioxide (SiO_2, commonly known as silica) is a material of considerable importance owing to its distinctive physical and chemical properties, including its unique surface characteristics and relative chemical stability. These properties make silica highly versatile and suitable for a wide range of applications . It is widely used in the food and medical industries because of its safety and low toxicity[11]. In addition, silica is employed in coatings owing to its hydrophobic properties[12]. It is also extensively used as a reinforcing material for polymers and in the development of advanced composite materials. Therefore, the main objectives of the current study are:

- ✍ **a.** To prepare silica from sand rose via sol-gel process .
- ✍ **b.** To prepare silica from aquifer sand via sol-gel technique.
- ✍ **c.** To reduce the quatity of chemical used (HCl, NaOH, as well as heating energy).
- ✍ **d.** To Utilize of available analytical techniques to evaluate and characterize the extracted silica.

1.4 Significance of study

The following are the significances of this study:

i. The use locally sourced materials such as sand rose and aquifer sand as new sources that can contribute to reducing the cost of silica production.

ii. Adopting a modified extraction protocol can significantly reduce the consumption of chemicals and energy requirements . In particular, the concentration of hydrochloric acid was reduced from 6 N to 3 N, the sodium hydroxide-to-sand ratio was decreased from 7:1 to 3:1, and the heating temperature was lowered from 900 °C to 500 °C.

iii. The conductivity measurement technology was adopted to monitor and track the various stages of the silica extraction process, allowing for the evaluation of the extraction progress and the efficiency of the washing and purification stages.

iv. Exploiting and Valorizing these local natural materials and converting them from low-value raw materials into a value-added materials, with the possibility of utilizing them in many industrial applications.

1.5 Thesis Layout

Chapter 1 provides a general introduction to the study, including the research background, problem statement, study objectives, and its significance.

Chapter 2 offers a comprehensive review of the literature on silica, focusing on its physical and chemical properties, natural sources, methods of preparation and extraction, and its most important applications in various fields.

Chapter 3 presents the methodology and experimental procedures used to extract silica from sand rose and aquifer sand. This methodology is divided into three main parts: first, a description of the chemicals used in the experiments; second, a presentation of the methods for preparing and extracting silica from both sand rose and aquifer sand; and third, a presentation of the main analytical techniques and equipment used to evaluate the success of the extraction process and to verify the purity and properties of the extracted silica.

Chapter 4 discusses and analyzes the study's findings, beginning with the characterization of the raw materials, particularly their physical, mechanical, chemical, and spectral properties, and culminating in the characterization of the extracted silica and the evaluation of its properties and purity. These results will be presented and discussed in detail in subsequent sections.

CHAPTER 2

LITERATURE REVIEW

2.1 lntroduction

Pure silica (SiO_2) possesses a range of properties that make it a material of high industrial value. It has a high melting point of approximately 1710 °C, excellent chemical resistance to most acids except hydrofluoric acid (HF), low thermal resistance, low electrical resistance, and excellent optical transparency, especially in crystalline forms.

Silica occurs naturally in various forms, including crystalline forms such as quartz, tridymite, and cristobalite, and amorphous forms such as opal. The purity of silica varies depending on its source, with advanced industrial applications requiring silica with a purity exceeding 99.9%.

Algeria is among the countries with high silica deposits, with silica content exceeding 95% in some areas, one of the highest recorded globally. This high purity is attributed to the natural sorting processes that the sand undergoes due to wind, which separate the more resistant quartz grains from other, less stable minerals. Algerian sands are also characterized by lower mineral impurity levels compared to sands from other regions. For example, the proportions of iron and aluminum oxides in many of these sands are very low , making them particularly suitable for advanced industrial applications that require high levels of purity.

Physically, most Algerian desert sands have a medium to fine grain size and a shape that tends toward roundness, making them suitable for many industrial applications. They also exhibit a high degree of grain sorting, reflecting a relative homogeneity in size, a characteristic important for many industrial uses.

Silicon or Silicon Dioxide: Properties and Technological Significance Silicon photonics has attracted significant attention due to its combination of technological advantages and cost-effectiveness. Silicon, being robust and well-understood, allows high-density integrated optics at relatively low cost compared to exotic materials like IIIV compounds or lithium niobate. The processing techniques developed for silicon microelectronics provide sufficient precision for most optical applications, with current minimum feature sizes around 12 microns. While alternative silica-based optics may be lower in cost, they are mostly passive and cannot support active devices such as modulators, detectors, or sources, highlighting the intimate link between technological capability and cost in silicon photonics[13]. The physics and chemistry of SiO_2 and the Si-SiO_2 interface form the foundation for the majority of semiconductor device technologies. Understanding these interfaces has been a focus of multidisciplinary research across fields including ceramics, materials science, metallurgy, and geology. Conferences and symposia, such as the one organized under the Electrochemical Society, have helped consolidate knowledge and foster collaboration among diverse research groups studying the SiO_2 system.

Over the decades, progress in understanding the Si-SiO_2 interface has been critical for both theoretical insights and practical applications in semiconductor devices.[14]. The structure of SiO_2, or silica, is highly versatile and undergoes complex phase transformations depending on temperature, pressure, and chemical purity. Its framework consists of [SiO4] tetrahedra connected into a giant polymeric network, allowing flexibility in SiOSi angles to accommodate changing conditions. Recent investigations into silica polymorphs, including high-pressure and amorphous states, have improved understanding of its structural behavior and phase transformations. The structure of vitreous silica, in particular, remains technically important and the subject of ongoing study due to its relevance in ceramics and optical applications.[15]. Silicon Dioxide: Properties and Applications in Electronics and Solar Cells Silicon dioxide (SiO_2) is one of the most widely used insulating materials in integrated circuit (IC) technology and silicon-based device fabrication due to its excellent chemical stability and well-understood properties. Its success over other materials, such as germanium oxide, is attributed to its thermal stability and resistance to water solubility. Silicon dioxide can be produced through thermal oxidation of silicon or deposited using chemical vapor deposition (CVD) techniques, resulting in materials with varying densities, porosities, and electrical properties. In microelectronics, it plays essential roles including diffusion masking, surface passivation, ion implantation screening, and electrical insulation. It also functions as a dielectric material in capacitors and multilayer metallization systems. In MEMS applications, thicker oxide layers are often used, where properties such as stress and conformal growth become critical factors influencing material selection and performance[16]. Silicon dioxide also plays a significant role in solar cell technology, particularly in improving the efficiency and performance of photovoltaic systems. It is widely used as an interfacial layer and antireflective coating in silicon-based solar cells, enhancing light absorption and electrical performance. Advances in technology have enabled the use of lower-cost silicon materials, making solar energy more accessible and sustainable. Silicon dioxide is commonly integrated into crystalline silicon solar cells, which dominate over 90% of the global photovoltaic market. Additionally, it is utilized in both traditional and advanced solar technologies, including thin-film and nanostructured devices. These innovations contribute to higher efficiency, reduced production costs, and support the transition toward renewable energy systems and a sustainable global economy[17].

2.2 Definition

2.2.1 Silicon

Silicon or Silicium (Si) is one of the constituent elements of silica and has been used since ancient times in the form of silicon dioxide as a key component in the manufacture of glass and cement, and in foundry work. Today, pure silicon is used for its semiconductor properties in the electronics industry (electronic chips) and in the manufacture of photovoltaic solar panels.

Silicon is extracted by reducing and purifying silica found in silica sand or quartz, using various processes, resulting in pure silicon (99.999% Si) in the form of alloys from which silicon wafers are cut [18].

Based on its purity, silicon is divided into three categories: metallic silicon (99% purity), solar silicon (99.9999% purity), and electronic silicon (99.9999999%). Over the past decade, the photovoltaic solar energy industry has experienced tremendous growth, leading to a surge in demand for silicon that has far exceeded the limited global supply. Its price has risen sharply; the price of a kilogram of silicon, which traded for just a few dollars at the beginning of the 21^{st} century.

2.2.2 Oxygen

Oxygen (O_2) is defined as a diatomic gaseous molecule that is abundant in the atmosphere and plays a crucial role in regulating metabolism in both humans and plants. In its ground state, it is a stable molecule, but at higher energy states or in reduced forms like superoxide , it can act as an active source of free radicals that disrupt cellular redox balance.

Oxygen is a non-metallic element belonging to Group 16 of the periodic table. Its electronic configuration contains six valence electrons, which enables it to form bonds with metals or more electronegative elements through ionic or covalent bonding. By sharing electrons, oxygen tends to achieve a stable octet configuration.

Oxygen can react directly with various substances, resulting in processes such as cell oxidation, rusting, and combustion. It readily combines with many other elements, except for some noble gases and a few relatively unreactive metals. It has numerous important applications, including:

- ☛ It is essential for respiration and is one of the major elements required for the existence of life.
- ☛ Liquid oxygen can be used as an oxidizing agent and propellant in space rockets.

☛ Oxygen is used in the manufacture of important chemical products, including nitric acid, ethanoic acid, trichloronitric acid, and ethanoic acid.

☛ Oxygen combined with ethyne (acetylene) is used to produce high temperatures for cutting and welding metals.

☛ Oxygen is used in the steel industry to remove carbon, sulfur, and phosphorus from molten iron.Table: 2.1 illustrates the position of silicon and oxygen in the periodic table.

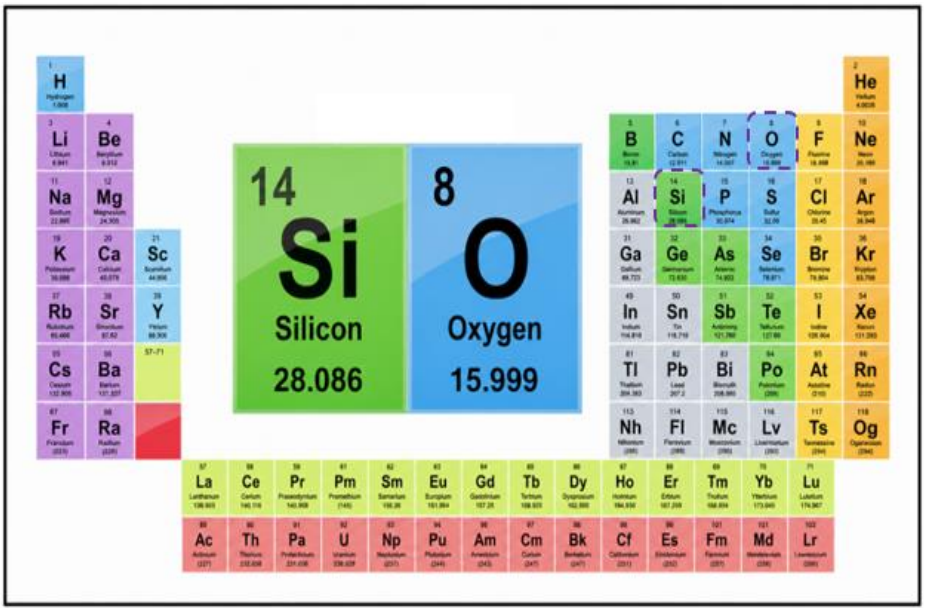


Figure 2.1: Position of silicon and oxygen in the periodic table

2.2.3 Silica

Silica is a product of the combination of silicon and oxygen. It is a naturally abundant compound found in a variety of crystalline and non-crystalline forms, including quartz, opal, diatomaceous earth, and synthetic products like precipitated silica, fumed silica, and colloidal silica. Its structure is primarily based on tetrahedra linked by shared oxygen atoms, forming complex frameworks that can adjust to environmental conditions. Quartz represents the most well-known crystalline polymorph, while amorphous silica appears as submicroscopic aggregates or engineered industrial products. Silicas versatility in particle size, surface chemistry, and porosity allows its application in glass, ceramics, polymers, electronics, and high-precision optical devices. Single-crystal quartz is grown for resonators and communication technology, while industrial silicas are tailored for catalysis, adsorption, fillers, and nanomaterials. Modern production techniques include chemical precipitation, flame hydrolysis, plasma, and sol-gel

processes, which control morphology, purity, and surface functionality to meet specific industrial or biomedical requirements[19].

The chemical and environmental behavior of silica is strongly influenced by its aqueous forms and interactions. Dissolved silica, generated by chemical and biochemical weathering of silicate rocks, exists as various species including silicic acid ($Si(OH)_4$) and soluble silicates. Its solubility is affected by temperature, pH, ionic strength, and sorption-desorption mechanisms involving soils and sediments. Silica is biologically essential, forming diatom skeletons, contributing to plant structural strength, and playing a role in animal tissue and bone development. Historically, silica has enabled technological advances, from flint tools and Roman cement to modern catalysts, fiber optics, quartz electronics, and advanced ceramics. The study of silicas chemistry, phase behavior, and interaction with biological systems continues to guide applications in environmental management, biomaterials, and sustainable resource utilization[20] Recent developments in nanotechnology have produced mesoporous silica nanoparticles (MSNs) with controlled particle size, pore structure, and surface properties, enabling applications in catalysis, adsorption, polymer composites, optical devices, and biomedicine, particularly targeted drug delivery and theranostics. Synthesis techniques include sol-gel processes, templating, Stöber modifications, and self-assembly, allowing functionalization with organic and inorganic groups. Characterization through XRD, FTIR, SEM, and XRF ensures precise control over particle morphology and chemical composition. In addition to engineered nanostructures, natural silica sources like rice husk ash, agricultural residues, clay, and coastal sands provide sustainable, low-cost precursors for high-purity silica extraction. The recovery and processing of silica from these sources reduce environmental waste and support industrial and biomedical applications, reinforcing silicas status as a critical material in both natural and engineered systems[21]

2.3 Chemical and physical properties

2.3.1 Chemical properties

It exhibits unique chemical properties attributed to its high surface area, amorphous structure, and abundance of silanol (Si-OH) groups on the particle surface and siloxane (Si-O-Si) groups within the particles[22]. These Si-OH groups impart hydrophilicity, enabling interaction with polar solvents and facilitating the addition of functional groups to the surface through covalent bonds or hydrogen interactions. At the nanoscale, SiO_2 exhibits distinct chemical properties compared to its larger counterpart, primarily due to its significantly higher surface area-to-volume ratio, resulting in a higher density of surface Si-OH groups and reactive oxygen

atoms per unit volume. These high surface functionalities enhance the reactivity and chemical modifiability of silicon nanoparticles. The functional groups present in silicon nanoparticles vary depending on the starting material and the synthesis method used. Some chemical reactions are listed in Table: 2.1.

Table 2.1: Chemical reactions of silica with different reagents.

Reacting material	**Chemical equation**
Strong bases (KOH, NaOH)	$SiO_2 + 2KOH \rightarrow K_2SiO_3 + H_2O$
	$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$
Calcium carbonate ($CaCO_3$)	$SiO_2 + CaCO_3 \rightarrow CaSiO_3 + CO_2$
Hydrofluoric acid (HF)	$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O$
Potassium carbonate (K_2CO_3)	$SiO_2 + K_2CO_3 \rightarrow K_2SiO_3 + CO_2$
Sodium carbonate (Na_2CO_3)	$SiO_2 + Na_2CO_3 \rightarrow Na_2SiO_3 + CO_2$

2.3.2 Physical properties

Silicon dioxide (SiO_2) has a very high melting point, approximately 1700 °C, due to its covalent lattice structure and the strong bonds between silicon and oxygen atoms, which require a large amount of heat to break. SiO_2 is also characterized by high hardness and toughness, making it suitable for many applications requiring high resistance to heat and corrosion. Since it lacks free electrons capable of movement within its structure, it is a poor conductor of electricity and generally behaves as an insulator. Furthermore, SiO_2 is insoluble in water and most organic solvents, while it can react or dissolve in some basic media. It also reacts with hydrofluoric acid (HF) to form fluorinated silicon compounds.Table: 2.2 lists some of the most common physical properties of silicon dioxide[23].

Silica is a widely studied material with diverse chemical, physical, and biological properties. Studies on silica in the oat plant show that silica extracted by wet-way ashing represents the solid silica of the plant, composed entirely of opal with a refractive index ranging from 1.427 to 1.440 and a surface area of 14.4 m^2/g. Dry-way ashing, however, caused changes in the silica, including partial crystallization of opal into cristobalite, increased refractive index, and significant reduction in surface area due to the incorporation of larger amounts of Na, K, Ca, Mg,

Table 2.2: Physical properties of silica.

Property	**Description**
Elements	Mainly composed of silicon and oxygen.
Hardness	Silica has a hardness of approximately 7 on the Mohs scale.
Density	Relatively high, ranging from approximately 2.65 to 2.70 g/cm^3.
Refractive index	Approximately 1.46, and it is associated with the production of glass and optical fibers.
Melting point	Approximately 1710 °C, making it suitable for a wide range of applications involving high temperatures.
Color	Generally ranges from white to gray, depending on impurities. Silica containing titanium and iron may exhibit pale red or yellowish-red coloration.

Mn, and Fe in insoluble forms, highlighting the impact of extraction methods on both chemical composition and physical structure[24] . Silica dust is also recognized for its high fibrogenic activity in humans and experimental animals, which is much greater than most other materials. This toxicity is associated with its ability to damage phagocytic cells. Once silica particles are engulfed by macrophages, they escape from phagosomes into the cytoplasm, rapidly causing cell damage, whereas non-toxic particles such as diamond dust remain within phagosomes without harm. The toxic effect of silica is attributed to its interaction with and permeabilization of the lipoprotein membranes surrounding phagosomes. Colloidal silica has been shown to be haemolytic, confirming its ability to alter membrane permeability and explaining the development of silicosis and other cytotoxic effects[25] Biogenous silica, found in diatoms and siliceous sponges, exhibits a wide range of solubility and structural forms. Experimental comparisons of silica gel and opal reveal that the soluble fraction of diatomaceous silica varies with species and organic matter content. X-ray diffraction patterns suggest that biogenous silica commonly exists as an amorphous silica gel, while infra-red spectra also resemble silica gel. Differences are observed between recent and ancient diatom frustules, with recent samples resembling silica gel and ancient samples resembling opaline silica. Siliceous sponge spicules display opaline characteristics in both dissolution behavior and infra-red spectra. These findings indicate a spectrum of biogenous silica forms, from easily soluble silica gel to pseudo-opaline structures,

with implications for marine silica cycling and material properties[26]

2.4 Some sources of SiO_2

The figure: 2.2 shows a schematic representation of some silica sources, divided into two categories: biological and natural. Amorphous silica is also found in soil, mainly in the form of sand and quartz. It can be produced industrially or prepared as nanoparticles using modern techniques. Its source and preparation method affect its properties and uses, especially in agriculture[14]. Agricultural waste is considered a sustainable source of silicon dioxide, in both crystalline and amorphous forms. Silicon-rich waste can be converted into nanosilica with modifiable properties and improved performance compared to solid materials. Besides material recovery, this value-added silica shows promising potential, particularly in agriculture, where it enhances stress resistance and reduces heavy metal absorption[27].

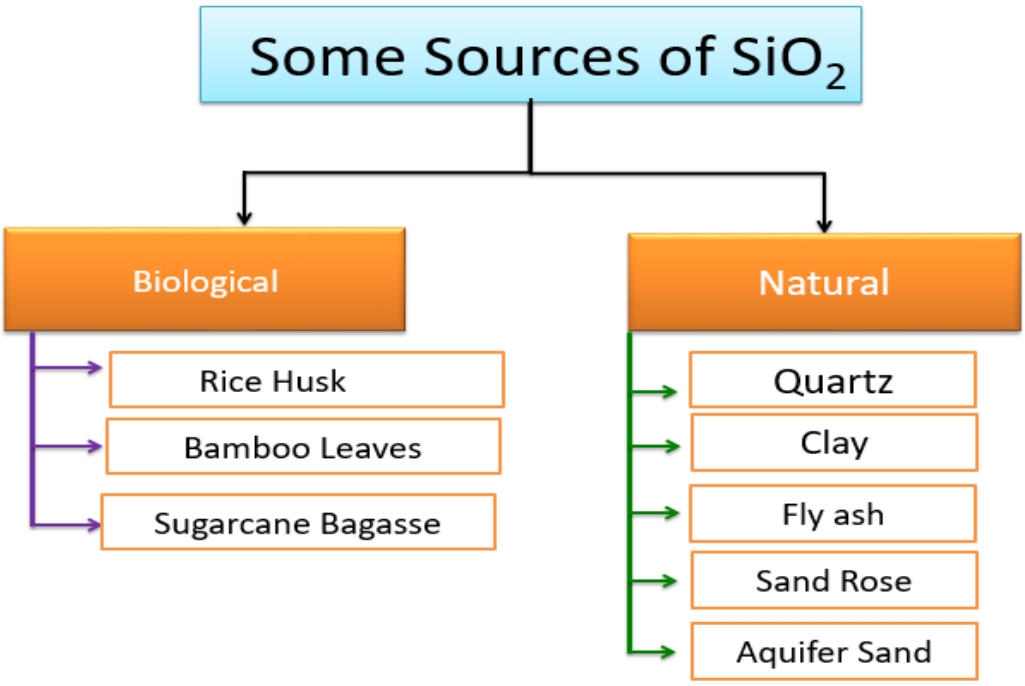


Figure 2.2: Some of Sources SiO_2

2.4.1 Biological

(SiO_2) has attracted significant attention worldwide due to its excellent biological, mechanical, and physicochemical properties. Various synthetic approaches, including sol-gel, sillca gel,

ultrasonication, chemical precipitation, and hydrothermal methods, have been developed to produce silica nanoparticles (SiO_2 NPs). In addition to chemical synthesis, biological routes have gained interest, with plant-derived materials such as bamboo leaves, rice husk, and sugarcane bagasse serving as renewable sources for SiO_2 NPs production. These biologically synthesized nanoparticles can be characterized using techniques like X-ray diffraction (XRD), transmission electron microscopy (TEM), dynamic light scattering (DLS), and Fourier-transform infrared spectroscopy (FTIR). Furthermore, SiO_2 NPs can be conjugated with metallic oxides or polymers to expand their applications, which include agriculture, electronics, material science, mechanical engineering, and especially biomedical fields, such as antibacterial applications and drug delivery. The biocompatibility and notable biological activity of these nanoparticles make them promising candidates for a variety of advanced applications[28]. Silica extraction from rice husk ash offers a sustainable solution for waste management while producing a valuable material for industrial use. The resulting silica, including silica gel, has a high surface area suitable for water adsorption as a desiccant and also exhibits natural antifungal properties. This study focuses on extracting and characterizing amorphous silica from rice husk ash to explore its potential applications in the food industry while mitigating environmental impacts[29].

2.4.1.1 Rice husk

Rice husk (RH) and its ash (RHA) are abundant by-products rich in amorphous silica (85-95%). RHA has been widely used in ceramics, catalysts, cement, and other industrial materials, making it a sustainable alternative to conventional silica sources[30]. ice husk (RH) is one of the most abundant agricultural by-products generated worldwide from the rice milling industry, accounting for about 20 wt % of the processed rice. Traditionally, rice husks have been mainly used as a low-value energy source through direct incineration. However, in recent years, they have gained increasing attention as a renewable and sustainable biological source of silica (SiO_2). Owing to their high silica content, rice husks can be efficiently converted into silica particles with high surface area, good adsorption capacity, and ease of functionalization. These properties make rice husk-derived silica particularly attractive for various high-value applications, including water purification, catalysis, drug delivery, and the fabrication of mesoporous silica structures and composites. Moreover, the integration of silica extraction with thermochemical processes such as pyrolysis enables the simultaneous production of valuable by-products, including bio-oil and syngas, ensuring comprehensive utilization of the biomass. Consequently, rice husks represent a promising precursor for producing value-added SiO_2 materials, contributing

to sustainable development and the circular economy[31].Rice husk ash (RHA) is a by-product obtained from the combustion of rice husk for energy production. It mainly contains silica along with carbon and small amounts of other components. Several methods, such as acid or alkaline leaching followed by thermal treatment, are commonly used to improve the purity of the extracted silica. Previous studies have shown that silica obtained from RHA can reach a purity higher than 98%, and even around 99% when appropriate treatment methods are applied[32].



Figure 2.3: Rice Husk

2.4.1.2 Bamboo leaves

Bamboo leaves are considered an important agricultural source of silica, containing high levels of silicon dioxide (SiO_2) ranging from 75.90 to 82.86%. Analyses have shown that bamboo leaves are rich in ash, reaching up to 24.5%, with silica constituting the major part, while the fixed carbon content is low, making them suitable for silica extraction. High-purity silica can be obtained from bamboo leaf ash using simple methods such as alkaline extraction, where controlling the concentration of sodium hydroxide and applying acid treatment helps increase silica yield and improve its quality by removing metallic impurities. Several studies have indicated that silica extracted from bamboo leaves possesses favorable properties, enabling its use in various fields such as the food and pharmaceutical industries, as well as in advanced industrial applications and as a nanoscale catalyst[33].Bamboo leaves are considered an important agricultural source of silica due to their high silica content in the resulting ash. Several studies have reported the synthesis of nanosilica from bamboo leaf ash using precipitation techniques. In one study, nanosilica was successfully synthesized from bamboo leaf ash (BLA) using the precipitation method, and the effect of ash temperature on the synthesis process was investigated. Characterization results obtained from XRD, FTIR, and EDX analyses confirmed the

formation and purity of amorphous silica. SEM and TEM observations revealed spherical silica nanoparticles with an average size of 25-30 nm. The obtained silica exhibited a high specific surface area of about 456 m^2/g, indicating the formation of mesoporous silica with superior properties compared to commercially available crystalline silica. Furthermore, the synthesized nanoparticles demonstrated good photocatalytic performance, achieving approximately 88% degradation of methylene blue within 150 minutes. These results highlight that bamboo leaf ash is a promising, eco-friendly, and low-cost source for the production of silica nanoparticles [34].



Figure 2.4: Bamboo leaves

2.4.1.3 Sugarcane bagasse

Sugar and ethanol production from sugarcane generates significant amounts of agricultural residues, particularly straw and bagasse. When these residues are burned, ash is produced that is often disposed of in landfills, which may cause negative environmental impacts. However, studies have shown that this ash contains a high percentage of silica, making it an important biogenic source for the extraction of silicon dioxide (SiO_2). Research has indicated that sugarcane bagasse ash contains between 72% and 81% silica, while sugarcane leaf ash contains about 80%. Therefore, this silica-rich ash can be valorized in several industrial applications, such as construction materials and rubber reinforcement, contributing to the transformation of agricultural waste into a value-added raw material [35].



Figure 2.5: Sugarcane Bagasse

2.4.2 Natural

Silica (SiO_2), especially quartz, is one of the most abundant minerals in nature and has been used since ancient times in tools, jewelry, and construction. Today, it remains essential in both high-tech industries, such as electronics and solar energy, and traditional applications like glass and foundry production. Natural sources include quartz, sand, clay, rice husk ash, and diatomaceous earth, each with specific properties that determine their suitability for different uses. Understanding and classifying these materials is crucial both for geological and for environmental purposes[36].Among these sources, agricultural waste has recently attracted significant attention as a sustainable alternative. Rice husk (RH) is considered one of the most promising agro-residues due to its low cost, high availability, and high silica content. It can be used to produce both amorphous and crystalline silica, which are essential in various industrial applications such as cement, electronics, polymers, and adsorbent materials. Thermal and chemical treatments are commonly applied to extract silica from rice husk, while techniques such as FTIR, XRD, and SEM are used to characterize its structural and morphological properties[37]

2.4.2.1 Quartz

Quartz sand is a premium raw material derived from quartz stone, distinguished by its excellent physical and chemical characteristics[38]. Although chemical purification methods are widely used for metallic minerals, their application to non-metallic minerals such as quartz remains relatively limited. Traditional techniques, including bioleaching and high-temperature leaching, often fail to meet the high-purity requirements of modern industries. Advanced methods, such as high-temperature pressure leaching and catalyzed leaching, have been developed

to improve purification efficiency, reduce acid consumption, and avoid the use of fluoride-based reagents, offering promising solutions for sustainable industrial applications[39].Impurities in high-purity quartz, such as aluminum, iron, and sodium, significantly affect its performance in advanced applications like electronics, photovoltaics, and optics. Several purification techniques, including acid washing, chlorination, and calcination, have been applied to address these impurities. However, these methods still face limitations in efficiency, cost, and environmental impact. Therefore, developing more efficient and environmentally friendly purification techniques is essential to produce high-purity quartz suitable for modern technological industries[40].



Figure 2.6: Quartz

2.4.2.2 Clay

Natural clay minerals composed of phases such as nontronite, muscovite, quartz, and hematite can be transformed into a silica-rich material through calcination and acid treatment. Studies have shown that calcination at high temperatures (around 700 °C) leads to the formation of an amorphous phase, while subsequent treatment with hydrochloric acid effectively removes aluminum and iron ions from the clay structure. This process increases the specific surface area and porosity of the material, resulting in a silica-enriched amorphous structure. Such chemical and thermal treatments demonstrate an effective approach for obtaining high-surface-area amorphous sillica from natural clay minerals[41].Mesoporous silica was produced from anorthite clay using mechanical activation by grinding followed by selective acid leaching. The effects of grinding time and acid treatment on the pore characteristics were evaluated using techniques such as X-ray diffraction (XRD), BET surface area analysis, thermogravimetric analysis (TG), and particle size measurements. The findings showed that grinding and heat treatment altered the crystal structure of the clay. However, acid treatment had the most

significant effect on the pore structure by increasing the specific surface area of the samples. In addition, a positive relationship was observed between the solubility of anorthite clay and the surface area of the resulting mesoporous silica[42]

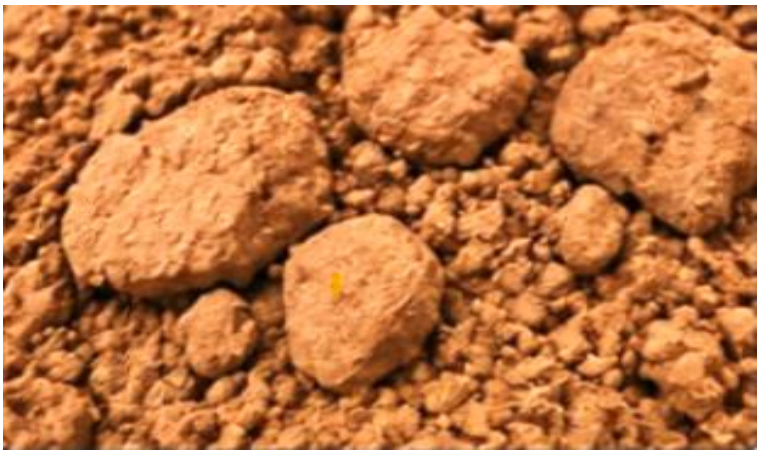


Figure 2.7: clay

2.4.2.3 Fly ash

Fly ash (FyA) is a by-product generated during coal combustion in thermal power plants. Due to its high silica and alumina content, it exhibits important pozzolanic properties, which make it widely used in the construction industry, particularly in cement and concrete production. In addition to its role in improving the mechanical properties of concrete, fly ash has also attracted attention as a low-cost material for pollutant removal and for the synthesis of advanced materials such as geopolymers, zeolites, and silica-based nanomaterials[30] Mineral construction materials are extensively utilized in waste management, especially for the solidification and stabilization of hazardous wastes. Among these materials, fly ash produced as a by-product of hard coal combustion in power plants has gained significant attention, is commonly used as a mineral additive in cement-based systems due to its high silica content. Its incorporation helps form stable and durable matrices capable of immobilizing heavy metals. Therefore, fly ash is considered a valuable material for waste stabilization processes[43] Fly ash is produced in large quantities from coal combustion in thermal power plants, particularly in countries with high energy demands like India. A considerable portion of this fly ash remains unutilized, posing significant environmental and waste management challenges. With rapid urbanization, industrial growth, and population increases, both energy production and fly ash generation continue to rise annually. This situation highlights the urgent need for researchers to develop effective strategies for fly ash utilization. Current studies have examined its origin,

chemical composition, physicochemical characteristics, and potential applications, especially in the construction industry and in other innovative fields[44]



Figure 2.8: Fly ash

2.4.2.4 Sand Rose

Sand Roses and Sand Interactions: Composition, Applications, and Environmental Influence Sustainable construction approaches have explored the use of sand-gypsum mixtures inspired by natural sand roses found in desert regions. Cold sintering of sand with uncalcined gypsum offers an energy-efficient method to produce durable construction bricks. The mechanical strength of these materials is significantly influenced by factors such as gypsum particle size, applied pressure, and cyclic loading conditions. Mixtures containing more than 30% fine gypsum and subjected to high can achieve compressive strength superior to conventional concrete bricks. Additionally, protective coatings like calcium carbonate improve resistance to weathering, while recycling does not reduce performance. This method promotes sustainability by reducing energy consumption, costs, and environmental impact while supporting circular economy practices[45] Mineralogical studies of sand dunes and sand roses in southern Algeria reveal that quartz and gypsum are the dominant mineral phases. Advanced characterization techniques such as XRD, FTIR, SEM, and EDS confirm that quartz is the primary component in sand dunes, while gypsum is predominant in sand roses. Variations in mineral composition were observed between regions, with higher quartz concentration in Ouargla samples and higher gypsum concentration in Adrar samples. Grain size analysis also showed differences between the two areas. These findings highlight the potential of these natural materials as important industrial resources for applications requiring silica and gypsum[2] Sand also plays a significant role in ecological systems, particularly in plant growth and adaptation. Studies on Rosa rugosa demonstrate

that sand coverage depth strongly influences plant survival and vigor. Plants buried deeper (up to 30 cm) showed higher survival rates, faster leaf development, and greater overall growth compared to those planted at normal depth. Deeper sand coverage promoted the formation of new roots near the surface, enhancing plant stability and nutrient uptake. These results indicate that sand can positively influence plant adaptation in coastal and sandy environments, supporting its role in ecological restoration and landscape stabilization [46]



Figure 2.9: Sand Rose

The sand rose is not a flower like the ones we know, but a desert rock naturally formed from sand, also known as the "desert flower" or "sand rose."

It is a type of desert rock called the "desert rose." This rose grows through successive stages, like the stages of rock fossilization that usually form in the upper level of groundwater through a series of reactions between minerals present in the water, such as calcium, and the calcareous grains present in the sand. When the water evaporates, calcium sulfate (CaSO4, 2H2O) remains, which forms what we call gypsum. It slowly decomposes due to oxidation factors, becoming a powder when it reacts with water. Then, what is called hydrated calcium sulfate is obtained, which in turn crystallizes, forming shapes like flower petals joined together at different angles, giving it a rose-like appearance. It comes in different sizes, from small to large, reaching up to a meter in length, forming a wonderful and beautiful crystalline shape that catches the eye. It is the color of sand or brown, which is the most common color. The sand rose is found in hot, dry desert regions and is used in homes by placing it in the form of small ornaments for decoration.

2.4.2.5 Aquifer Sand

Around the world, clay binders, and additives in specific proportions to produce green sand castings. Beyond its role in mold and core making, silica sand is widely used in industries such as glass production and construction. The heavy consumption of silica sand can put a strain on these resources, leading to potential shortages. Identifying alternative sources can help reduce dependence on a single supply and lower costs. In addition to traditional binders and additives, various organic materials are available that can improve the properties of sand molds, depending on the type of metal being cast, including cast iron, steel, aluminum, and brass. Exploring alternative sand sources can therefore result in high-quality castings while reducing production expenses[47].Silica sand is an important industrial material mainly composed of quartz and widely used in glass, foundry, and abrasive industries. Studies, such as those conducted in the Lemi region of Ethiopia, show that high-purity silica sand with suitable grain size and low impurities can meet industrial standards. With large estimated reserves, such deposits represent valuable natural resources with strong economic and industrial potential[48]



Figure 2.10: Aquifer Sand

2.5 Industrial synthetic sources of SiO_2

The utilization of industrial waste, particularly metallurgical residues, represents an important source for producing high-purity silicon dioxide through modern and resource-efficient methods. Among these methods is ammonium fluoride technology, which relies on sublimation and desublimation processes, enabling the separation of amorphous silica and the recovery of valuable components such as iron oxide. This technique falls within the broader approaches for synthesizing industrial silica, alongside the solgel method, which is based on chemical hydrolysis

Table 2.3: Silica content at different sources

Name	**Content (%)**	**References**
Rice husk	**94**	[49]
Bamboo leaves	**70.5**	[50]
Sugarcane bagasse	**64.38**	[51]
Clay	**58.10**	[52]
Fly ash	**98.65**	[53]
Sand dunes	**97.63**	[54]
Sand Rose	**1.208**	This work
Aquifer Sand	**87.95**	This work

and condensation reactions to form high-purity silica. One of its main products is silica gel, known for its high porosity and wide application in adsorption and drying processes, as well as colloidal silica, which consists of fine nanoscale particles dispersed in a liquid medium and is widely used in advanced coatings and electronic industries[55].

2.5.1 Sol gel method

In the Sol-gel process, colloidal dispersions of small molecules or polymers (sols) are transformed into liquid-filled porous materials (gels), which can then be dried to produce aerogels through evaporation or aerogels via supercritical fluid extraction. This method allows the preparation of nanoporous amorphous materials in both thin-film and bulk forms, with excellent control over surface area, pore volume, particle size, and size distribution. Additionally, the pore surfaces can be modified using liquid or vapor-phase techniques to adjust pore size, surface chemistry, and catalytic activity for specific applications. Strategies have also been developed to control the microstructure of the dry gel and to characterize access to the resulting pore structure, making the SolGel method a flexible and advanced approach for producing silica and nanoscale ceramic materials[56] This study highlights the role of pH in the solgel process for silica coating of magnetite nanoparticles, demonstrating that reaction conditions significantly influence the surface properties of the resulting materials[57].

2.5.1.1 Silica gel

Silica gel is particularly useful for controlling humidity because of its ability to adsorb water vapor. In museum collections, it can be used inside exhibition cases to maintain stable relative humidity and reduce humidity-induced damage to objects. Its efficiency depends on several factors, including its buffering capacity, the amount of silica gel used, leakage rate, and external relative humidity conditions. The hysteresis-corrected buffering capacity (MH) is considered an important variable for evaluating the efficiency of silica gel. Selecting the appropriate quantity of silica gel is essential for achieving effective humidity control while avoiding unnecessary costs. When some parameters, such as leakage rate or external humidity conditions, are unknown, general recommendations based on average conditions can be applied for both temporary exhibitions and permanent displays. Proper use and regular maintenance of silica gel make passive humidity control a simple, practical, and cost-effective method for protecting museum collections from humidity-related deterioration[58]. The increasing interest in silica gel has encouraged researchers to investigate inexpensive and renewable sources for its production. Rice husk is considered one of the most promising sources because it is an abundant agricultural waste product in rice-producing countries. Its use provides an economical source of silica while also contributing to the valorization of agricultural waste. Silica gel has a rigid three-dimensional network of colloidal silica and can exist in several forms, including aqua-gel, alco-gel, xerogel, and aerogel. Among these materials, silica aerogels are characterized by high specific surface area, high porosity, low bulk density, excellent thermal insulation properties, and a low dielectric constant and refractive index. These properties allow them to be used in thermal insulation, acoustic barriers, supercapacitors, catalytic supports, and other advanced technological applications. The production of silica from rice husk involves several important parameters, including the pH of the synthesis process, the temperature used to burn the rice husk, and acid leaching before the formation of rice husk ash. These conditions can significantly influence the properties and quality of the final silica product. Consequently, developing economically viable processes capable of producing rice-husk-derived silica with specific and controlled properties is of considerable importance.

2.5.1.2 Colloidal sillca

Nanosilica is one of the most widely used nanomaterials due to its unique physicochemical properties compared with bulk silica. These properties make it suitable for many industrial applications such as reinforcing materials, pigments, pharmaceuticals, and thermal and electrical

insulators. However, silica nanoparticles in powder form tend to agglomerate strongly during the drying process, which limits their performance. To overcome this problem, nanosilica can be prepared in the form of colloidal silica, where the nanoparticles are dispersed in a liquid medium, usually water, resulting in better stability and dispersion. Colloidal silica can be produced through several methods, including neutralization of soluble silicates, ion exchange, hydrolysis of silicon compounds, and dispersion of pyrogenic silica. Nevertheless, some of these techniques require expensive raw materials or involve high energy consumption. Recently, the use of low-cost materials such as silica fume, combined with ultrasonication techniques, has attracted attention as an alternative approach. Ultrasonic treatment enhances particle dispersion, reduces agglomeration, and can contribute to the formation of stable colloidal nanosilica suspensions[59]. Silica has many important applications in fields such as hydrometallurgy, catalysis, cement production, and paper coating. One of the effective methods used to synthesize silica particles is ultrasonic spray pyrolysis (USP), which operates at high temperatures around 900 řC. This technique allows the formation of spherical submicron silica particles from an aerosol, and the particle size can be controlled by adjusting the concentration of the precursor solution. The obtained particles are typically characterized using techniques such as SEM, EDS, and XRD, which showed that the produced silica has an amorphous structure. Compared with other synthesis methods, the USP technique offers advantages such as a simple setup, continuous one-step synthesis, and the ability to produce nanoparticles from different precursors[60].

2.6 Extraction of SiO_2

Methods of Extracting Silicon Dioxide (SiO_2) from Industrial Solid Wastes Silicon dioxide (SiO_2) can be efficiently extracted from industrial solid wastes such as fly ash and coal gangue using different physicochemical treatment methods depending on the desired purity, surface area, and application. One effective method involves a combined organic acidinorganic alkali-ultrasonication process for extracting SiO_2 from fly ash . Initially, fly ash is leached with acetic acid (HAc) at 25 °C for 1 hour using a FA/HAc ratio of 1:3 to remove impurities . The treated material is then dissolved in 15% NaOH solution at 100 řC for 2 hours with a FA/NaOH ratio of 1:4.5 . After cooling and adding water (5.42 g/100 mL), ultrasonication is applied for 20 minutes to enhance silica recovery . Under optimized conditions, the SiO_2 yield reaches 51% with a high purity of 98.65% . The extracted silica also demonstrates excellent adsorption performance toward cationic dyes such as methylene blue and malachite green[53].

Another advanced approach is based on activating coal gangue using supercritical water combined with alkaline treatment before silica extraction . In this method, coal gangue is treated in a reactor with supercritical water and alkali activators such as KOH, transforming kaolinite into more reactive mineral phases . After activation, silica is extracted using an acidalkali combination process . The extraction rate of Si can reach 69.2% when KOH is used as an activator . The obtained SiO_2 has a purity of 96.2% and exhibits very high specific surface areas ranging from 710 to 872.8 m^2/g depending on reactor scale . Furthermore, modified SiO_2 shows excellent selective adsorption capacity for xylene, indicating strong potential for environmental and industrial applications[61] A simpler and more conventional method for silica recovery from coal gangue is acidic leaching . Since coal gangue mainly consists of SiO_2 and other mineral components, controlled acid treatment allows silica extraction under optimized conditions . The efficiency of extraction depends on parameters such as acid-to-material ratio, leaching temperature, and leaching time . By carefully adjusting these factors, SiO_2 meeting national product standards can be successfully obtained . This method offers a relatively straightforward and practical solution for large-scale silica recovery from abundant coal gangue resources[62].

2.6.1 Silica extraction from waste

Silicon dioxide (SiO_2) can be recovered from various industrial and agricultural wastes using several physicochemical methods, enabling the valorization of waste and the production of valuable materials.

One industrial approach involves recovering silica from photonic industrial waste powder (PWP) using an alkali fusion process. In this method, a silicate-rich supernatant is separated from the solid sediment, with the supernatant serving as a precursor for synthesizing mesoporous silica materials such as MCM-41. The prepared MCM-41 exhibits a very high specific surface area (1082 mš/g), narrow pore size distribution (2.95 nm), and large pore volume (0.99 cmş/g). After modification with tetraethylenepentamine (TEPA), the material demonstrates strong CO adsorption capacity compared to other similar adsorbents, highlighting the potential of converting industrial waste into high-performance mesoporous adsorbents.[63]

Agricultural wastes are also important renewable sources of silica. In one method, agricultural residues such as groundnut shells, walnut shells, and wood carpentry waste are burned to obtain ash, followed by chemical extraction using a bottom-up synthesis approach to produce nanoscale silica particles. XRD, SEM-EDX, and FT-IR analyses confirm the successful forma-

tion of SiO_2, with particle sizes ranging from 9 to 30 nm, indicating the nanoscale structure of the extracted material. This approach supports the concept of waste-to-wealth by converting biomass residues into high-value silica materials[64]..

Another simple extraction method uses rice straw as a silica source. Rice straw is heated and extracted into a basic solution, followed by precipitation in an acidic medium to form silica particles. Aggregated particles of approximately 200 nm were obtained, and XRD and FTIR analyses confirmed that the final product is silica free from graphite impurities. However, small amounts of KCl remain due to the use of KOH as the extraction agent, indicating the need for further purification to obtain higher-purity silica.[65].

2.6.2 Extraction of silicon dixide from clay

Silica nanoparticles were synthesized from bentonite clay through a series of thermal and acid treatments aimed at reducing alumina content and enriching silica concentration. The silica-rich clay was treated with sodium hydroxide solutions (10 wt% and 40 wt%) to produce sodium silicate solutions (SSS). These solutions were hydrolyzed using nitric acid at varying concentrations (5 M, 10 M, and 15 M) in the presence of ethanol as a co-solvent. By adjusting the concentrations of silica-rich clay, HNO, and ethanol volume (1030 ml), silica particles ranging from nanometer to micrometer scale were obtained. Higher concentrations of clay and nitric acid directly influenced particle size, while increasing ethanol volume produced bimodal particle distributions. Characterization was performed using ICP-OES, atomic absorption spectroscopy, XRF, SEM, and XRD[66]. Sodium silicate was prepared from SiO_2 concentrate obtained after aluminum extraction from clay sourced from Sancheong-Hadong, Korea. The concentrate was leached with NaOH at temperatures ranging from 25-100 °C under atmospheric pressure. Approximately 80% of silicon was extracted at 25 °C within 30 minutes. The molar ratio of SiO_2/NaO in the resulting sodium silicate solution ranged from 2.5 to 3.1. Impurities included 0.11 wt.% aluminum and 67 ppm iron. Out of 150 g of concentrate, about 100 g dissolved, while the remainder was undissolved. Analysis showed negligible formation of insoluble sodium compounds during NaOH leaching, and filtration performance was evaluated under varying pressure and temperature conditions.[67]. Pure amorphous silicon oxide was synthesized from treated kaolinitic clay (Kankara clay, Nigeria) using chemical extraction. The clay was first beneficiated, then thermally activated and acid-leached. Raw, thermally treated, and leached clay samples were reacted with 3 M NaOH at 200°C under continuous stirring with reflux for 3 hours to form sodium silicate solution. The solution was filtered and precipitated with 3 M

HCl to produce a gel-like white substance. The gel was aged for 18 hours, washed multiple times with deionized water, and dried at 80°C for 12 hours to obtain pure white silica particles. XRD analysis confirmed the amorphous structure of the synthesized silicon oxide, while microstructural evaluation showed particle aggregation typical of solgel synthesized powders[68].

2.6.3 Silica extraction from sand

Methods of Extracting Silicon Dioxide (SiO_2) The extraction of silica from white sand in Sulawesi Island began with sampling at several coastal points and different depths. The sand was first cleaned to remove impurities and iron sand, followed by preparation and identification stages. Silica was then extracted using the alkali fusion method with a high-concentration base. Elemental composition was analyzed using XRF, crystal structure using XRD, and functional groups using FTIR. XRF results showed that the beach sand was mainly composed of CaO (>80%). XRD confirmed the presence of silica (SiO_2) in quartz form, while FTIR identified the characteristic SiO functional groups..[69]. In another study, silica was extracted from Bengkulu beach sand using a solidliquid extraction (leaching) method in three main steps. The sand reacted with NaOH at 95°C (alkaline fusion) to form sodium silicate (NaSiO), followed by the addition of HCl to produce silicic acid Si(OH). Drying of the silica gel then yielded SiO_2. Initial analysis indicated that Bengkulu sand contained 69.87% silica, and after extraction the purity increased to 97.3% according to XRF results. XRD characterization showed that the formed silica was in the amorphous phase, and FTIR confirmed the presence of SiOSi and SiO bonds characteristic of silicon dioxide. Silica was also extracted from Bajul Mati beach sand (Malang) using a low-cost and more environmentally friendly method combining leaching and sol-gel processes. The procedure involved leaching with 7 M HCl to remove impurities, forming a clear sodium silicate solution, then performing a sol-gel process by titrating the solution with oxalic acid until pH 6 to form Si(OH). The product was characterized using XRF, XRD, FTIR, and SEM, along with an economic analysis. The results showed that the extracted silica had a cristobalite structure, with a purity of 93.9% based on XRF analysis. FTIR identified silanol and siloxane groups, and SEM images revealed agglomerated particles of approximately 1 m. The economic study indicated strong potential for future industrial application of this method[70].

2.6.4 Extracting silica from rice husks

Rice husk is one of the most abundant agricultural residues generated from rice production worldwide. It constitutes nearly one-third of the total grain mass and contains a high ash content (18-20%), of which silica represents approximately 85-95%. Due to its low nutritional value, rice husk is not suitable for animal feed, although it is used as boiler fuel and for power generation. The high silica content makes it economically viable to extract silica from rice husk ash (RHA). A simple process was developed to manufacture precipitated silica from rice husk waste, addressing both waste disposal problems and value-added material production. Experimental results demonstrated good performance and feasibility for industrial implementation.[71]. A simple method for producing pure silica from rice hull ash was developed based on alkaline extraction followed by acid precipitation. The produced silica gels were heated at 80°C for 12 hours to obtain xerogels. Characterization using energy dispersive X-ray (EDX) and inductively coupled plasma (ICP) spectroscopy showed that the xerogels contained 93% silica with 2.6% moisture, and an extraction yield of 91%. Major impurities included Na, K, and Ca. Acid washing before extraction reduced calcium concentration (<200 ppm), while final water washing was more effective in lowering overall mineral content (Na <200 ppm and K <400 ppm). XRD analysis revealed the amorphous structure of the silica xerogel, and FTIR confirmed the presence of siloxane and silanol groups[72]. A comprehensive review on silica extraction from rice husk highlighted the sustainability of using low-cost and renewable waste materials to produce engineered silica particles. Rice husk serves as a renewable raw material not only for silica production but also for biofuel generation. The review described both traditional and modern synthesis methods for tailoring silica particles from rice husks. It provided background information on rice husk composition and discussed various extraction techniques, emphasizing their applications in multiple industrial fields. The study underlined the importance of developing efficient and sustainable processes for large-scale silica production from agricultural waste.[73].

2.6.5 Silica extraction from wastewater

Methods of Extracting and Utilizing Silicon Dioxide (SiO_2) A novel study investigated the recovery of silicon from carbonized raw sewage sludge (RS) to produce high-purity nano-SiO_2. The key step involved early rupture of SiOSi bonds by OH in the SiO tetrahedron network, leading to the formation of RS-derived sodium silicate ($Na_2O(SiO_2)_x \cdot (H_2O)_y$). Silica gel was formed through the cohesion of colloidal silica particles, influenced in part by the bridging

role of hydrated Na. O1s peaks of SiO_2 were decomposed into SiO bridging oxygen atoms and hydroxyl groups, while FTIR analysis showed residual SiOH bands, decreasing as sol pH increased. The high-purity RS-SiO_2 exhibits potential for various environmental and biotechnological applications, providing an environmentally beneficial method for reutilizing sewage sludge.[74]. Silica nanoparticles (SiNPs) extracted from rice husk ash were studied for nitrate ion removal from aqueous solutions. The SiNPs, with a purity of 98%, particle size around 50 nm, surface area of 30 m^2/g, and average pore width of 2.72 nm, demonstrated high nitrate adsorption capability of 14.22 mg/g under optimal conditions (pH 6, contact time 50 min, SiNP dosage 0.15 g/vol). The method offers advantages such as reduced sample pretreatment steps, shorter processing time, and reliable nitrate removal from polluted water.[75]. The use of mesoporous silica materials (MPSs) for removing dyes from textile wastewater has been extensively reviewed. MPSs are promising adsorbents due to their high surface area, tunable and uniform pore structure, high pore volume, ordered pore arrangement, thermal and mechanical stability, and wide functionalization possibilities to enhance adsorption. Various operational conditions such as contact time, pH, temperature, and agitation speed affect dye adsorption efficiency. SBA-MPSs showed higher adsorption capacity than MCM-MPSs, and functionalized MPSs performed better than non-functionalized forms. Although mesoporous silica-based nano-sorbents have high potential for industrial dye removal, further research is required to develop cost-effective and more efficient commercial applications.[76].

2.6.5.1 Comparison between extraction methods

Chemical and Automated Extraction Techniques Chemical extraction techniques are widely employed in supramolecular chemistry to study the molecular recognition, binding, and transport of various chemical species. These methods are relevant in multiple areas, including biochemical processes, analytical chemistry, environmental protection, recycling, catalysis, and medicine. Over the years, numerous selective receptors have been developed for cations, anions, salts, and zwitterions using different molecular architectures and binding modes. Experimental approaches often involve studying the distribution of species between two immiscible phases, typically aqueous and organic, to evaluate the receptors complexation behavior and its suitability for separation, monitoring, or concentration of specific species. Such techniques have been applied historically to quantify cation binding strengths and remain essential tools in research and industrial applications.[77]. In soil science, determining the potassium content is crucial for crop production, nutrient management, and fertilizer recommendations. Various chemical ex-

traction methods, including ammonium lactate (KAL), ammonium acetate (KAA), ammonium acetate with EDTA (KAAEDTA), Bray (KBRAY), and barium chloride, have been compared to evaluate their efficiency in extracting potassium from different soil types. Studies indicate that while extraction results vary among methods, strong correlations exist between the techniques, and KAL is often considered the most precise. The comparative analysis demonstrates that any of these extraction methods can be used to accurately predict soil potassium concentrations and can form the basis for predictive models in soil nutrient management.[78]. Automated nucleic acid extraction methods offer significant advantages in clinical laboratories, improving workflow and reducing variability compared to manual extraction. Evaluations of automated systems such as Qiagen EZ1 and bioMérieux easyMAG compared to manual extraction demonstrate that automated methods achieve similar amplification efficiency for most targets, with minor exceptions. The easyMAG system exhibited the lowest operational complexity and was most effective at low target concentrations, although it was the most expensive. These findings highlight that automated extraction systems provide reproducible and efficient alternatives for nucleic acid isolation in diagnostic and research settings.[79].

2.6.5.2 production of mesoporous silica nanoparticles

Mesoporous Silica Nanoparticles in Biomedical Applications Mesoporous silica nanoparticles (MSNs) have attracted significant attention due to their controllable morphology, particle size, and porous structure, which make them suitable for various applications such as catalysis, adsorption, optical devices, and biomedical fields. Several synthesis methods have been developed to produce well-dispersed MSNs and hollow silica nanoparticles with tunable dimensions ranging from a few nanometers to hundreds of nanometers. These methods include fast self-assembly, soft and hard templating techniques, the modified Stöber method, dissolvingreconstruction processes, and modified aerogel approaches. The resulting materials demonstrate promising potential in applications such as high-performance catalysis, antireflection coatings, polymer nanocomposites, and controlled drug-release systems.[80]. Mesoporous silica nanoparticles are widely investigated as drug carriers because one of the major challenges in medicine is delivering drugs efficiently to diseased or tumor cells while minimizing toxic side effects. Their unique structural properties, including pore size, pore volume, surface area, particle size, and drug-loading capacity, strongly influence their performance in drug delivery systems. Since the introduction of materials such as MCM-41, SBA-15, and MCM-48 as controlled drug carriers, research on MSNs has increased significantly. Surface functionalization using organic

or inorganic groups further enhances their ability to deliver drugs to targeted sites. These nanoparticles are also being explored in medical imaging, diagnosis, cellular uptake studies, targeted drug delivery, cell tracing, and biosensing applications.[81]. Recent advances in drug delivery technologies have further expanded the importance of mesoporous silica nanoparticles due to their high thermal, chemical, and mechanical stability. These materials can be synthesized through relatively simple and cost-effective methods, while synthesis parameters can be adjusted to control particle morphology, pore size, and pore volume. MSNs have been widely used to load both small molecules and large biomolecules such as proteins and siRNA, making them versatile carriers for therapeutic applications. Recent research has also focused on modifying their structure to enhance their use in drug-resistant chemotherapy and antimicrobial therapies. Although their biomedical potential is significant, further studies are required to better understand their interactions with biological systems before full clinical application can be achieved[82].

2.7 Methods for the Preparation of Silica (SiO_2)

Shape-controllable silicon nanostructures are of great interest due to their diverse applications, particularly in the biological field, thanks to their unique properties such as biocompatibility and thermal stability. This study focuses on the factors that control the formation of these structures and their various shapes, as well as their formation mechanisms and methods for optimizing their performance. It also reviews their most important applications, such as stabilization, biocatalysts, and drug delivery [83].This study investigates the preparation of various nanostructures from amorphous SiO_2, such as nanofibers, tubes, springs, and ribbons, using chemically catalyzed vapor deposition (CCVD). This method utilizes starter materials like Si, SiO_2, and graphene capsules, with the presence of small amounts of SiC guiding the growth of the structures towards different shapes. The growth process follows a vapor-liquid-solid (VLS) mechanism, allowing for the creation of diverse and shaped nanostructures [84].

Silica (SiO_2) is a major component of the Earths crust and has gained significant importance in nanotechnology applications. Recent studies highlight environmentally friendly and cost-effective methods for producing silica nanoparticles from agricultural wastes such as rice husk, rice straw, maize cobs, and bagasse. These methods focus on converting waste materials into valuable silica products, promoting sustainability and reducing environmental pollution. The processes involved include controlled burning to obtain ash followed by chemical extraction techniques, making this approach both economically viable and eco-friendly [85]. Nano-silica can be effectively prepared from rice husk ash using different techniques such as precipitation and sol-gel methods. In the precipitation method, silica is extracted using sodium hydroxide solutions, while the sol-gel method involves acid treatment and calcination processes. Characterization techniques such as infrared spectroscopy confirm the presence of silanol and siloxane groups, while UV-visible analysis indicates optical properties with a wide bandgap. Particle size analysis shows that nano-silica particles can be produced in the range of approximately 13-24 nm, demonstrating the effectiveness of these methods for producing high-quality nanomaterials [86] Colloidal silica can be synthesized through various preparation methods, including ion exchange of silicates, hydrolysis and condensation of silicon compounds, direct oxidation of silicon, and mechanical dispersion of silica powders. Each method produces silica particles with different properties in terms of size, shape, purity, and stability. For instance, silica prepared from tetraethoxysilane offers high purity and uniform particle size but at a higher cost, while methods based on liquid silicates provide easier control but may introduce impurities. Direct oxidation methods produce spherical, monodispersed particles with good purity and lower en-

vironmental impact. The choice of preparation method depends on the intended application and required material properties[87]

2.7.1 Hydrothermal

The hydrothermal method is commonly used to synthesize silica (SiO_2) nanoparticles because it allows good control over particle size and ensures high purity. In this method, silica forms in aqueous solutions under controlled temperature and pressure. Due to its advantages in controlling morphology and stability, hydrothermally produced silica nanoparticles are widely used in industrial and environmental applications, often combined with techniques like membrane processes to improve their properties[88].The hydrothermal method was compared with the solgel method for synthesizing SiO_2 particles using TEOS as a precursor and NHOH as a catalyst. The results showed that silica prepared at room temperature had an amorphous structure, while hydrothermal treatment improved the material by making it more crystalline. This confirms that the hydrothermal method enhances the structural properties of SiO_2[89]. In addition, recent studies have demonstrated the successful synthesis of amorphous silica nanowires (SiO_2 NWs) from rice husk ash using an FeO-assisted hydrothermal method. The obtained nanowires exhibited controlled morphology with nanoscale diameters and high aspect ratios. Characterization confirmed their amorphous structure, while optical analysis revealed enhanced band gap and strong blue emission. These results highlight the effectiveness of the hydrothermal method as a simple and cost-efficient approach for producing silica nanostructures with promising optoelectronic applications[90].

2.7.2 co precipitation

The co-precipitation method is widely used for the synthesis of silica nanoparticles can be efficiently produced from geothermal sludge using a method. The process led to high yield and purity under optimal conditions, and the results confirmed good material quality. Overall, it offers a simple and environmentally friendly approach for silica production[91]Silica powders with spherical shape and low agglomeration can be prepared by the precipitation method using sodium metasilicate and hydrochloric acid in an emulsion system. Typically, cyclohexane is used as the organic phase, while non-ionic surfactants help stabilize the emulsion. The preparation method strongly affects particle size and uniformity, with homogenization giving better results than simple stirring or ultrasound. Factors such as temperature, reagent addition, and phase composition are also important. The best results are obtained at 25 °C, producing silica

particles smaller than 1 m with a high surface area (340-390 m^2/g[92] This work presents a simple and efficient method for producing nanoparticles in aqueous solution under mild conditions. The process involves the formation of very small metal particles, which are quickly coated with a silica layer that limits their growth and prevents agglomeration. This coating forms naturally due to local changes in pH during the reaction. The obtained nanoparticles can also be transformed into metal oxides while preserving their nanoscale size. Overall, this method offers a low-cost and environmentally friendly approach for synthesizing stable nanoparticles[93].

2.7.3 So-gel

Sol-Gel Method for Synthesis of Silica Nanostructures and Advanced Materials The sol-gel method is one of the simplest and most effective techniques for synthesizing silica nanostructures, allowing precise control over particle size and morphology by adjusting reaction parameters. In this method, tetraethyl orthosilicate (TEOS) is commonly used as a precursor, while acetic acid and water act as catalysts and hydrolyzing agents. Parameters such as aging time and calcination temperature significantly influence the final properties of the silica nanoparticles. Results show that uniform silica nanospheres can be successfully synthesized, with optimal conditions producing particles in the nanoscale range of approximately 80-87 nm[94] The sol-gel process has a long historical background and has led to the development of advanced silica-based materials with a wide range of applications. Since its early discovery, the technique has evolved into a versatile method used in synthesis, coatings, adsorption, chromatography, environmental remediation, and biotechnology. Silica materials produced through sol-gel are highly transparent and suitable for optical applications. Over time, innovations such as silica gel production and aerogel synthesis have expanded its industrial and scientific importance, making it a key technology in modern materials science[9] The use of additives such as formamide in the sol-gel process plays an important role in improving the structural properties of silica gels. Formamide helps in producing crack-free silica gels with enhanced thermal stability and a more interconnected network structure. It promotes stronger SiOSi bonds and leads to materials with larger pore volumes and mesoporous structures, while samples prepared without additives tend to be microporous. These improvements make formamide-assisted sol-gel methods suitable for producing high-quality silica materials with controlled porosity and stability[95]

The sol-gel method is widely used for the synthesis of silica (SiO_2) nanoparticles due to its simplicity and effectiveness in controlling particle size and morphology. In this method, silica is typically formed through the hydrolysis of tetraethyl orthosilicate (TEOS) in the presence of

a solvent such as ethanol, often with additives like surfactants to improve dispersion and stability[96]. Silica nanoparticles have attracted significant attention due to their wide industrial applications. In this context, several studies have reported the successful synthesis of SiO_2 nanoparticles using the solgel method. The results confirmed the formation of amorphous silica with spherical morphology and an optical band gap of about 5 eV. The synthesized nanoparticles showed good photocatalytic activity, achieving nearly 69% degradation of methylene blue within 240 minutes. In addition, the material exhibited promising gas-sensing performance, particularly toward HS gas, with fast response and recovery times[97]. In another study, silica (SiO_2) nanoparticles were prepared via the solgel method using tetraethyl orthosilicate (TEOS) and PVP as a surfactant. Characterization techniques such as XRD, FTIR, and TEM confirmed the formation of amorphous silica with an average particle size of approximately 25 nm. Furthermore, EDX analysis verified the presence of silicon and oxygen in the expected composition.[98].

2.7.4 Thermal method

Thermal analysis methods are widely used to study the physical properties, chemical reactions, and thermal behavior of materials. These techniques provide valuable insights by observing how materials respond to temperature changes. Applications range from academic research to industrial processes, where instruments such as differential scanning calorimetry (DSC), thermogravimetric analysis (TG), and hot-stage microscopy allow scientists to monitor transformations like melting, decomposition, and phase changes. These methods are essential tools for understanding material behavior and improving analytical investigations[99] Thermal methods are also extensively applied in environmental and atmospheric studies, particularly for analyzing carbonaceous aerosols. Techniques such as thermal/optical carbon analysis and thermal desorptiongas chromatography/mass spectrometry (TD-GC/MS) enable the identification and quantification of organic and elemental carbon components in particulate matter. These methods provide detailed chemical composition data and can detect numerous organic compounds with high sensitivity. They are especially useful for real-time monitoring and understanding the sources and behavior of atmospheric pollutants, although challenges remain in improving accuracy and expanding their applications[100] Temperature plays a crucial role in altering material properties, from minor effects like thermal expansion to major transformations such as phase transitions. Various thermoanalytical techniques are used to detect and measure these changes, including TGA, DTA, and DSC. Additional methods are employed to

determine heat capacity, thermal conductivity, and thermal expansion. These techniques are widely available through commercial instruments and are essential for practical applications in material science, engineering, and quality control, enabling precise characterization of thermal behavior in different materials[101]

2.8 Applications of silica

Silica plays an essential role in the glass industry, as it is the primary material used in glass production. It is mainly found in the form of quartz sand, which is used to manufacture different types of glass such as container glass, flat glass, and glass fibers. Thanks to its high thermal stability and resistance to chemical reactions, silica helps in producing glass that is strong, durable, and of high quality[102]. The glass industry plays an important role in everyday life and covers several sectors, including containers, windows, fibers, household products, and technical applications. Different types of glass are produced using specific methods based on their intended use. In recent years, the industry has seen significant progress, with faster production and noticeable improvements in quality. Modern techniques are also used to enhance the properties of glass. Although there are many glass compositions, they can be grouped into a few main categories, and the choice mainly depends on the application and cost. Glass is widely manufactured and traded around the world, mainly because of its durability and long lifespan[103].

2.8.1 Glass industry

Silicate glasses are essential materials in modern life and have played a major role in the development of human civilization. Their evolution began with natural glasses and progressed to advanced materials designed for specific technological uses. Today, they are widely used in various fields, making them indispensable in both industry and daily life[104]. Functionalized silica nanoparticles (SiO_2 NPs) have recently received considerable attention because of their distinctive physicochemical properties, which make them suitable for various applications. These applications include catalysis, drug delivery, biomedical uses, and environmental remediation. The wide range of uses of these materials is mainly due to the possibility of modifying their surface, allowing their properties to be adapted to specific requirements. However, despite their importance, some challenges still limit their practical applications, which highlights the need for further research in this area[105]Silica-based chemically modified electrodes are reviewed in this article, highlighting the role of pure and functionalized silica as well as solgel derived materials.

These electrodes have shown good performance in the selective detection and preconcentration of various analytes, especially when silica is modified with organic or inorganic groups. In addition, solgel techniques enable enzyme immobilization for biosensor development, making silica-based materials promising for future electrochemical applications[106].

2.8.2 Catalysis

Mesoporous silica is a type of silica distinguished by its large surface area and well-structured pores, which makes it highly suitable for use in heterogeneous catalysis. It can be easily modified with different functional groups to improve its catalytic efficiency. In addition, it helps in achieving a better dispersion of active species and enhances their interaction with the support. Despite these advantages, some challenges remain, such as stability issues and control over the distribution of active sites. Overall, mesoporous silica is still considered a promising material for the development of efficient catalytic systems[107].Silica aerogels are special materials known for their high surface area and porous structure, which make them very effective as supports in heterogeneous catalysis. They have been widely used in different gas-phase reactions, such as oxidation, hydrogenation, and FischerTropsch processes. These characteristics help in better dispersion of the active species and improve the overall catalytic performance. For this reason, silica aerogels are considered promising materials for catalytic applications[108].

2.8.3 Adsorbents

Magnetic mesoporous silica materials were prepared by coating magnetite nanoparticles with a silica layer. Increasing the magnetite content changed the particle shape and reduced the thickness of the silica shell. The silica layer helps protect magnetite, especially in acidic conditions. These materials showed good ability to adsorb both humic acid and malachite green dye, where the acid enters the pores while the dye attaches to the surface. Calcium ions also affect the adsorption efficiency. Thanks to their magnetic properties, these materials can be easily separated from water, making them suitable for water treatment applications[109],Public health faces increasing challenges due to water pollution, particularly in rural areas lacking advanced treatment facilities. While point-of-use water treatment systems are a practical and cost-effective solution, they remain limited in their ability to efficiently remove modern contaminants. Therefore, there is a need for flexible systems capable of simultaneously detecting and treating contaminants. Silica nanoparticles are a promising option due to their effectiveness and selectivity, especially when combined with enzymes, which enhances their performance and

helps conserve water[110]

2.8.4 Nanomaterials

The rapid growth of the global population has increased the need for higher food production, while crop yields are negatively affected by pests and climate change. Silica nanoparticles have emerged as an effective approach to address these challenges due to their unique physicochemical properties, including small size and high surface reactivity. These nanoparticles are widely used in agriculture to promote plant growth under both normal and stress conditions. They can function as nano-fertilizers and nano-pesticides, improving crop performance. Additionally, silica nanoparticles enhance plant tolerance to biotic and abiotic stresses by stimulating antioxidant enzymes, protective compounds, and plant hormones. As a result, they play a significant role in improving plant resistance to environmental stresses such as drought and toxic metals, contributing to sustainable agriculture[111].Silica nanoparticles (SiO_2 NPs) have gained considerable interest because of their remarkable and adaptable physicochemical properties. Thanks to these features, they are widely applied in several fields, including catalysis, drug delivery, biomedical uses, and environmental protection. Their efficiency can be further improved through surface functionalization, which enhances their reactivity and selectivity. Despite these advantages, some limitations still exist, especially in terms of stability and large-scale application[105].

2.9 Conclusion

In this chapter, we presented the main aspects of silica (SiO), including its physical and chemical properties, forms, natural sources, and preparation methods. We also discussed its important applications in glassmaking, catalysis.

Overall, silica is an important material with diverse applications. This chapter provides a basis for our study of silica extraction from sand rose, focusing on a simple, cost-effective, and environmentally friendly process for the valorization of this local natural resource.

CHAPTER 3

RESEARCH METHODOLOGY

3.1 Introduction

This chapter comprises three main sections. The first section presents the chemical materials used in this study, outlining their basic properties. The second section details the methodology employed for preparing silica from both rose sand and aquifer sand, explaining the experimental stages involved in the extraction process. The third section focuses on the techniques used to characterize the extracted silica and evaluate its physical and chemical properties.

The characterization techniques employed in this study included sieve analysis, X-ray fluorescence (XRF) analysis, electrical conductivity measurement, Brunauer-Emmett-Teller (BET) surface area and porosity analysis, X-ray diffraction (XRD) analysis, scanning electron microscopy combined with energy-dispersive X-ray spectroscopy (SEM-EDX), and Fourier transform infrared spectroscopy (FTIR).

To illustrate the overall sequence of the research methodology and the study's implementation phases, a flowchart depicting the contents of this chapter is presented in figure: 3.1.



Figure 3.1: An illustrative map showing the content of Chapter III.

3.2 Chemicals Used

Table: 3.1 illustrates the properties of the chemicals used in this study, including hydrochloric acid, sodium hydroxide, silver nitrate, and distilled water. It should be noted that all the above-mentioned chemicals used were obtained from the Laboratory of Applied Chemistry and the Environment, Faculty of Exact Sciences, University of El Oued.

Table 3.1: Properties of the Chemicals Used

No	Name	Chemical formula	Properties	Company
1	*Hydrochloric acid*	HCl	37 wt.% ($\approx$ 12 M)	VWR Chemicals (France)
2	*Sodium Hydroxide*	NaOH	pellets	RM Chemicals
3	*Silver nitrate*	$AgNO_3$	MW = 169.8 g/mol	Fluka (Germany)
4	*Distilled water*	H_2O	1.2 $\mu s/cm$	Setif

3.3 Methods

3.3.1 collect of samples

The sand rose, also known as the desert rose, is widespread in many areas of El Oued Province. These natural crystalline formations result from the interaction of groundwater and sand with evaporation and drying processes that occur over long periods. They have been identified as a potential natural source of silica for scientific and industrial applications.

In this study, two types of samples were collected namely : Sand Rose (SR), and Aquifer Sand (AS) From a farm west of Al-Reguiba city, with the coordinates 33.548127° N, 6.674853° E, as shown in the figure: 3.2. It should be noted that the aquifer sand was obtained from a 40 m-deep artesian well located on the same farm.

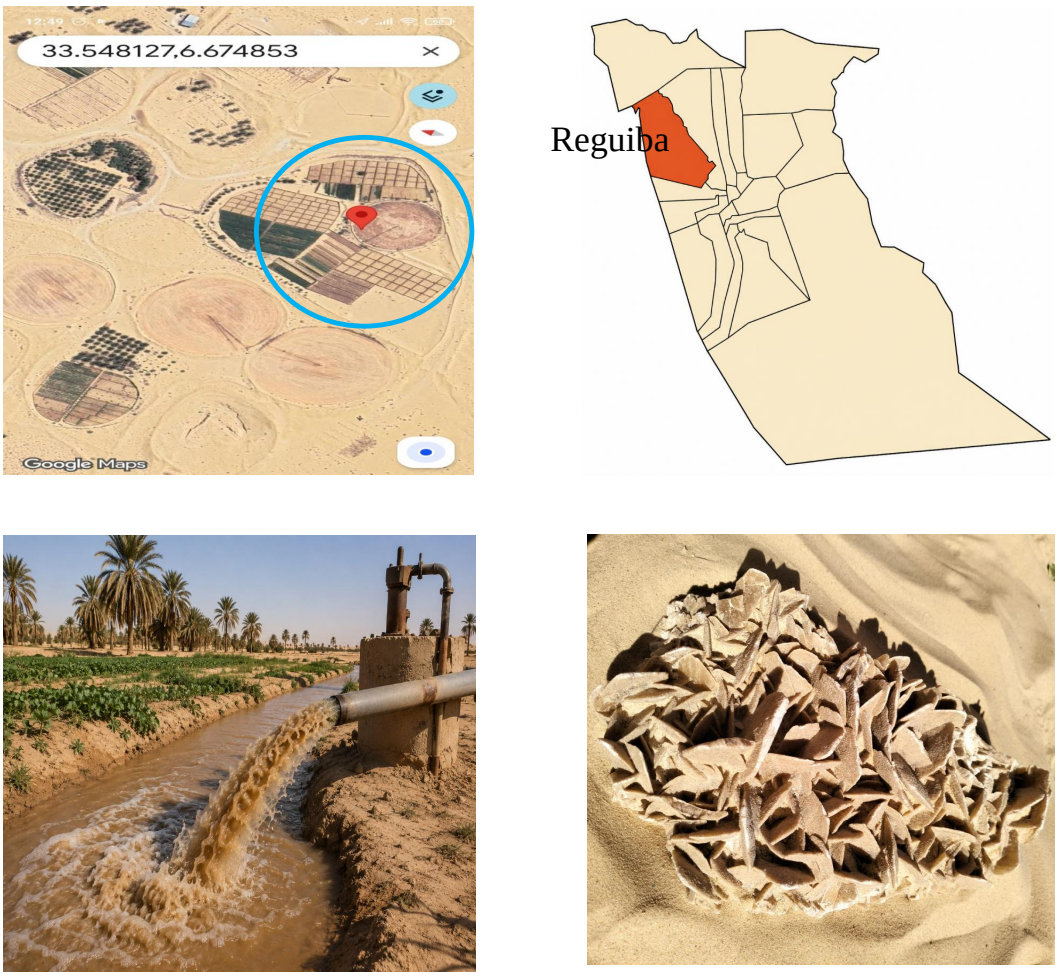


Figure 3.2: An illustration showing a map and the geographical location of the study samples.

3.3.2 Sieving and microscopic protocol

A 100 gr sample of aquifer sand was weighed and placed on the upper sieve of a mechanical vibrating shaker (Retsch, Germany). The sieve shaker was operated for 10 minutes, allowing the sand particles to pass through the sieves arranged according to their size. Larger particles were collected from a specific sieve opening, while smaller particles passed through subsequent sieves. After the sieving process was complete, the mass obtained on each sieve was measured and recorded. The results of the particle size distribution are shown in Table: 4.2, chapter IV.

According to microscopic test,Less than 1 gr of Aquifer sand was placed on a glass slide of a microscope (Stereo Blue) connected to a computer screen, and an image was screened of the diameters of the sand grains, as displayed in figure: 3.3

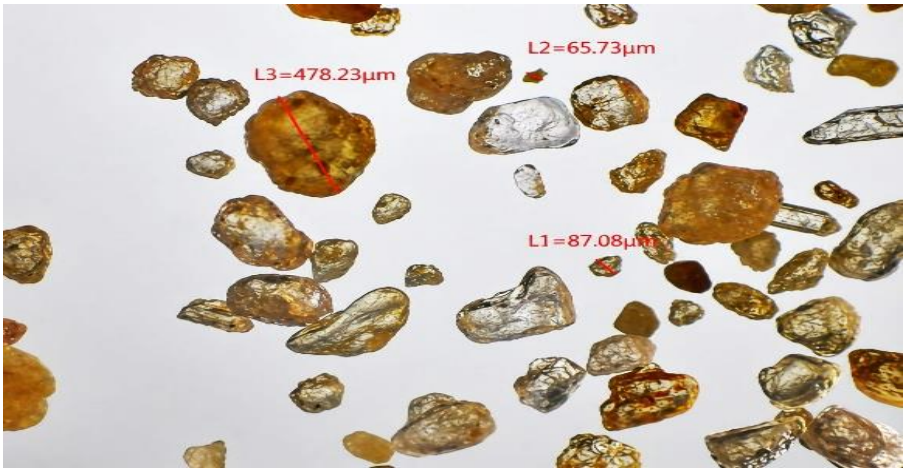


Figure 3.3: A microscopic image showing some diameters of a sample of Aquifer sand.

3.3.3 Conductivity Measurement protocol

A ratio of 1 gr of either sand rose or aquifer sand to 5 ml of distilled water is added erlenmayer and then place on a stirrer for half an hour. After that, the mixture is filtered using filter paper. The filtrate is taken to a conductivity analyzer, and then the results are recorded as listed in Table: 4.6.

3.3.4 Preparation of Silica from Sand rose

Amorphous silica was prepared from Sand Rose (SR) using the sol gel technique. Initially, the Sand Rose was thoroughly washed with hot distilled water to remove soluble salts and adhering impurities, and then ground into a fine powder using a mechanical mill. The resulting powder was dried in an oven at 105 °C for 24 h.

To remove acid-soluble metal oxides, the dried powder was treated with 3 M hydrochloric acid (HCl) at a ratio of 1 g of Sand Rose per 3 mL of acid solution under continuous stirring overnight. The acid-treated suspension was subsequently filtered under vacuum using a Büchner funnel, and the solid residue was thoroughly rinsed with distilled water until the pH of the filtrate reached neutrality.

The alkali treatment was then carried out by thoroughly mixing the acid-treated sample with sodium hydroxide at a mass ratio of 1 g of sample to 3 g of NaOH. The mixture was homogenized by grinding and then heated in an electric muffle furnace at 500 °C for 2 h to convert silica into sodium silicate. The obtained product was dissolved in distilled water and filtered to remove the insoluble residue. The filtrate was then acidified with hydrochloric acid until the pH reached 2, resulting in the formation of silica gel.

Finally, the silica gel was washed several times by centrifugation at 4000 rpm for 5 min per cycle to remove the sodium chloride formed during acidification and any residual impurities. The purified silica was then air-dried overnight at room temperature prior to further characterization.The Preparation steps for the Silica from Sand rose are presented in figure: 3.4.

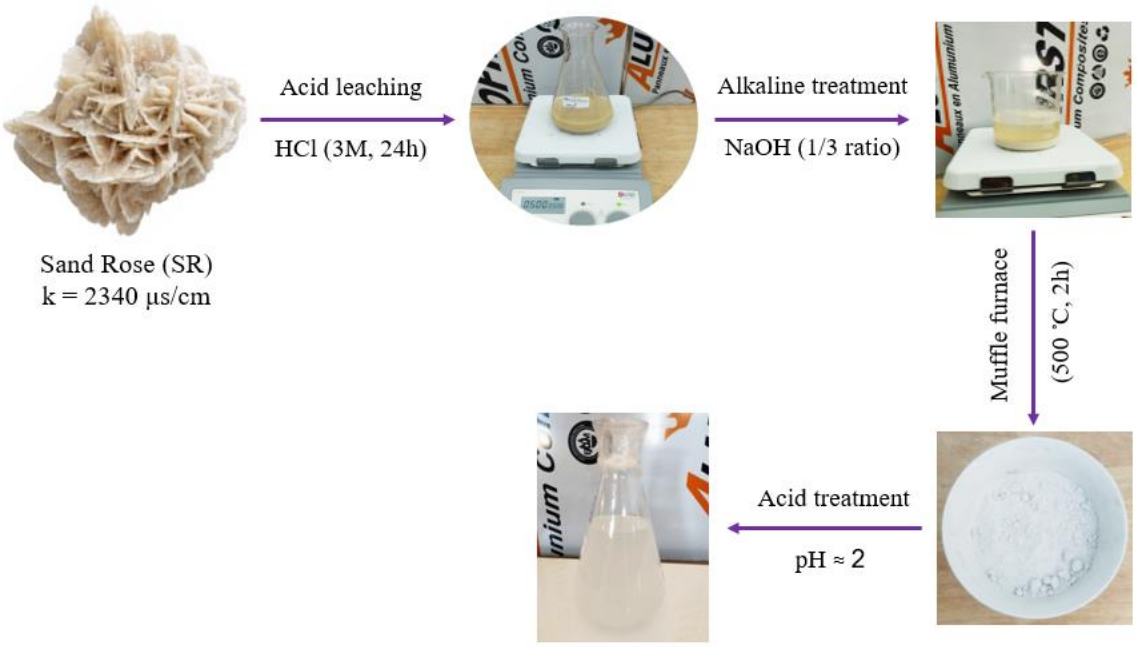


Figure 3.4: Preparation steps for the Silica from Sand rose.

3.3.5 Preparation of Silica from Aquifer sand

Amorphous silica was extracted from Aquifer Sand (AS) using the sol-gel method. Firstly, Aquifer Sand samples underwent repeated washing with hot distilled water to remove dissolved salts and surface impurities, followed by drying in an oven at 105 °C for 24 h to remove moisture.

To purify the raw material from acid-soluble metal oxides, an acid wash was performed using a 3 M hydrochloric acid (HCl) solution at a ratio of 3 mL of acid per 1 g of aquifer sand, with continuous stirring overnight to ensure efficient reaction. The solid phase was then separated from the liquid by vacuum filtration using a Buchner funnel, and the precipitate was washed several times with distilled water until the filtrate's pH was neutral.

After acid treatment, the purified precipitate was mixed with sodium hydroxide at a mass ratio of 1:3 (sample:sodium hydroxide) and the mixture was thoroughly ground to a homogeneous mixture. The mixture was then transferred to a muffle furnace at 500 °C for two

hours to convert the silicon dioxide to sodium silicate. After the heat treatment, the product was dissolved in distilled water, and the undissolved material was removed by filtration. To re-precipitate the silica, hydrochloric acid was gradually added to the filtrate until the pH decreased to 2, resulting in the formation of silica gel.

In the final stage, the silica gel was purified by repeated washing using a centrifuge at 4000 rpm for five minutes per cycle to remove the resulting sodium chloride and any remaining impurities. Finally, the silica was left to air dry overnight at room temperature before undergoing various characterization processes. The Preparation steps for the Silica from Aquifer Sand are summarized in figure: 3.5.

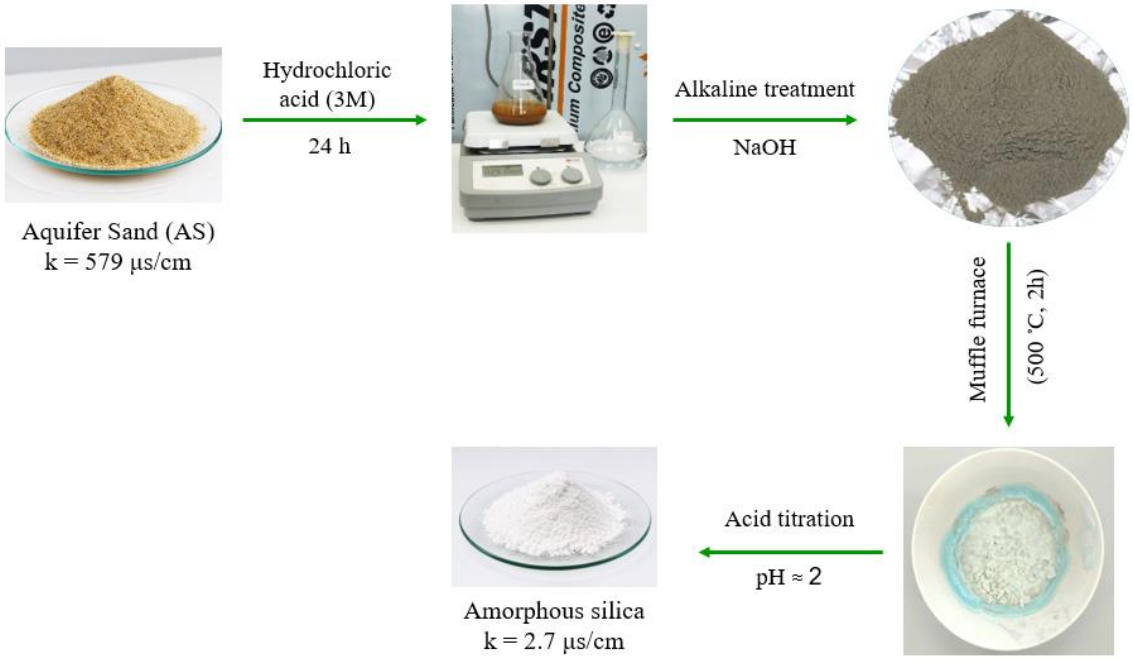


Figure 3.5: Preparation steps for the Silica from Aquifer sand.

3.4 Instruments

To evaluate the efficiency of the extraction process and characterize the silica prepared from both Sand rose and Aquifer sand, a set of appropriate characterization techniques and instruments were used in this thesis, as detailed in Table: 3.2.

Table 3.2: Characterization techniques used for the analysis of the extracted silica.

No	Technique	Purpose	Location
1	*sieving*	Particle size distribution	Faculty of Biology, University of El Oued
2	*XRF*	Chemical composition	CRAPC of Laghouat
3	*Conductivity*	Electrical conductivity	Faculty of Sciences, University of El Oued
4	*BET*	Surface area	CRAPC of Laghouat
5	*XRD*	Crystal structure and mineral phases	Faculty of Sciences, University of El Oued
6	*SEM-EDX*	Surface morphology and elemental composition	CRAPC of Biskra
7	*FTIR*	Functional groups	Faculty of Sciences, University of El Oued

3.4.1 Sieve Analysis

Sieving is one of the few principal methods, in which a relatively large quantity of material is physically subdivided in size classes. The separated fractions can be accu rately weighed and, thus, the determination of the size distribution functionbased on massmay be accurate. The major advantages of the sieving technique are that it allows relatively large samples for analysis or separation, that the principles are easy to understand and that the sieves and sieving equipment are relatively cheap. Also, size fractions are being obtained, which can be used for other purposes (e.g., further analysis or product testing). A critical point is to determine the effective cut size of the sieves in relation to both sieving conditions and particulate properties[112].

Sands are most generally measured by sieving. The basic principle of this technique is as follows. A sand sample of known weight is passed through a set of sieves of known mesh sizes. The sieves are arranged in downward decreasing mesh diameters. The sieves are mechanically vibrated for a fixed period of time. The weight of sediment retained on each sieve is measured and converted into a percentage of the total sediment sample. This method is quick and sufficiently accurate for most purposes. Essentially it measures the maximum diameter of a

sediment grain. This method is useful in analysis of terrigenous sediment[113]. Figure: 3.6 represents sieve shaker for different particle size distribution.



Figure 3.6: Sieve shaker for different particle size distribution analysis.

3.4.2 XRF Analysis

XRF is one of the best methods for elemental analysis. In XRF, the sample is bombarded with X-rays. This excites the sample to generate X-ray fluorescence. The X-rays shoot individual electrons out of the atoms of the elements, primarily out of the inner atomic shells K and L. The resulting vacancies are filled up again by electrons from higher energy shells. The excess energy of these electrons is then emitted in the form of X-ray fluorescence radiation. This radiation is characteristic for each element like a fingerprint and independent of the atoms chemical bond. The intensity of the radiation is proportional to the concentration of the element in the sample. XRF, however, can directly analyze each element without destroying the sample and no sample preparation is required. Figure: 3.7 exhibits XRF instrument.



Figure 3.7: XRF device .

3.4.3 Conductivity Measurement

Electrical conductivity measurement is the simplest and perhaps most sensitive method for expressing the ionic content of a solution. It is a device used to test a liquid's ability to conduct electricity, which is primarily determined by ion concentration, temperature, and cell shape. It is measured in units such as microsiemens per centimeter ($\mu m/\text{cm}$)[114].

The principle of measuring the conductivity of electrolyte solutions relies on passing an alternating current between two electrodes immersed in the solution, according to Ohm's law. Positive and negative ions move towards the electrodes of opposite charge, and the electrical conductivity is calculated based on the applied voltage and resulting current, while also accounting for the effect of the solution's temperature. Fundamentals of Measurement and the Cell: Electrodes: These are often made of platinum or graphite to ensure stability. Alternating Current: This is used to prevent electrolysis or electrode polarization, which would alter the solution's concentration. Distance and Area: The cell's sensitivity depends on the electrode surface area (A) and the distance between them (L). Factors Affecting the Reading: Ion Concentration: The higher the number of free ions in the solution, the higher the conductivity. Temperature: Ion movement changes with temperature; therefore, modern devices incorporate automatic temperature correction. Ion Nature: Different types of ions have varying speeds and capacities for carrying electrical charge.



Figure 3.8: Conductometer instrument .

3.4.4 BrunauerEmmett Teller (BET)

Measuring surface properties is a crucial aspect of studying a wide range of materials, making the BET device an essential tool used by researchers and engineers to analyze material surfaces and evaluate their physical and chemical properties.

This device consists of three main components :

- The main unit, which includes the furnace, vacuum pump, electrical circuit, and electronic measuring device.
- Drainage cells.
- Control system.

The device operates based on the adsorption of nitrogen gas onto the surface of the sample being measured. When activated, the gas flows into the analysis cell, and excess molecules are removed until an extremely low pressure is achieved within the cell. Due to nitrogens high affinity for solid surfaces with fine pores, it quickly adheres to the samples surface.

Subsequently, electronic signals are analyzed to determine the amount of adsorbed nitrogen, enabling the calculation of the actual surface area of the sample. Repeating this process enhances the accuracy of measurements by removing excess molecules and re-measuring adsorption, providing precise data on the materials porosity and surface area.



Figure 3.9: BET instrument

3.4.5 XRD Analysis

X-ray diffraction (XRD) is one of the most widely used techniques for characterizing crystalline materials. It relies on the interaction of X-rays with the crystalline planes of atoms within the material, resulting in diffraction as described by Bragg's Law. This technique provides precise information about the crystal structure, the phases present, and the degree of crystallinity, as well as detecting impurities and, in some cases, estimating the crystal size.

XRD analysis produces a distinctive pattern of peaks at different diffraction angles (2θ). Each crystalline phase represents a unique set of peaks that can be used to identify the material's composition and compare it to standardized cards in the ICDD-PDF database.

In this study, XRD measurements were performed using a PROTO Benchtop diffractometer (Figure: 3.10,). The instrument was equipped with :

- A Cu-target X-ray tube operating at 30 kV and 20 mA.
- radiation with a wavelength of $\lambda_{\mathrm{CuK}\alpha} = 1.54060$ Å was used.
- Diffraction patterns were recorded over a 2θ range of 10°–80°, with a scanning step of 0.1°.



Figure 3.10: X-ray diffractometer (XRD) instrument

3.4.6 Scanning electron microscopy (SEM-EDX)

Scanning electron microscopy (SEM) is one of the most important characterization techniques used to study the morphological properties of materials. It relies on the principle of a focused electron beam interacting with the sample surface. The instrument consists of several key components, including an electron source, electromagnetic lenses to focus the electron beam, a scanning system, detectors for the emitted signals, and a computer unit for data collection, analysis, and image rendering.

During the scan, the sample is scanned by an electron beam in a regular scanning pattern. This causes electrons to interact with the atoms on the sample surface and emit various signals, such as secondary electrons, reflected electrons, and characteristic X-rays. These signals are captured by specialized detectors and then processed to produce high-resolution images that reveal the surface morphology, particle shape, size, degree of agglomeration, pore structure, and surface distribution of the sample.

To determine the elemental composition of the sample, Energy Dispersive X-ray Spectroscopy (EDX) combined with scanning electron microscopy (SEM) is used. This technique relies on analyzing the characteristic X-rays emitted as a result of the interaction of the incident electron beam with the atoms of the material. Each atom possesses a unique emission energy, enabling the identification of the chemical elements present and the performance of qualitative and semi-quantitative analyses of their concentrations. The SEM-EDX system is an integrated technique that combines morphological analysis with elemental analysis, making it an effective tool for characterizing materials and evaluating their physical and chemical properties.

In this study, a Hitachi TM3030Plus instrument equipped with an EDX module was used to study the surface morphology and elemental composition of the extracted silica sample. The aim was to evaluate the effect of the extraction process on the surface structure and purity of the silica, as illustrated in the accompanying figure: 3.11[115].

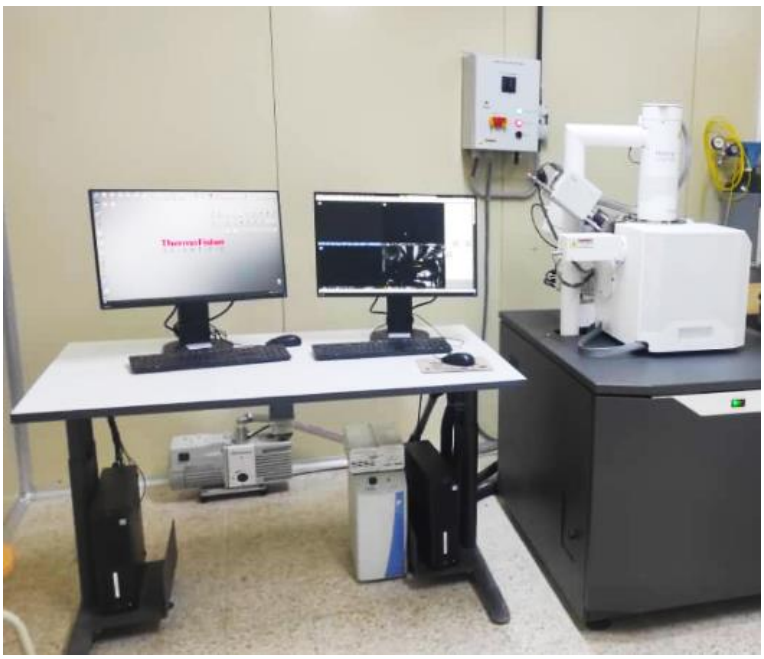


Figure 3.11: SEM-EDX instrument

3.4.7 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is an abbreviation for Fourier Transform Infrared Spectroscopy, FTIR is one of the most important analytical techniques used to characterize chemical substances. It relies on the absorption of infrared radiation by molecules at specific wavelengths, which excites molecular vibrations, whether stretching or bending. This allows for the determination of the nature of chemical bonds and functional groups present in a substance, as well as the study of its molecular structure and symmetry. Spectral data can also be used to estimate certain structural properties, such as the bond strength constant and bond length.

The infrared spectrum consists of two main regions: the stretching region, which appears at high wavelengths and represents the major functional groups in the molecule, and the bending region, which occurs at low wavelengths and is known as the fingerprint region. Each substance has a distinctive pattern of peaks that can be used to identify and distinguish it from others, even among compounds containing similar functional groups.

In this study, infrared spectra were recorded using a Shimadzu IR Affinity-1 spectrometer (Japan), with potassium bromide (KBr) discs serving as the inert carrier for the samples. Measurements were performed within the wavenumber range of 4000- 400 cm^{-1}, with only the main peaks being displayed. This technique was used to identify functional groups and compare the chemical composition of rose sand, aquifer sand, and extracted silica samples to evaluate the structural and chemical changes resulting from the silica extraction process, as illustrated in the accompanying figure: 3.12.



Figure 3.12: FTIR instrument

In conclusion, it is worth noting that characterization and analysis techniques are fundamental pillars of scientific research, contributing to the validity and accuracy of results and providing a comprehensive understanding of the properties of the studied materials. Although several advanced analytical techniques could have enriched this study, limited resources and capabilities prevented their application. Nevertheless, the most prominent and appropriate analytical techniques were selected and implemented to achieve the study's objectives, ensuring the reliability of the findings and providing a solid scientific basis for their discussion and interpretation in the following chapter.

CHAPTER 4

RESULTS AND DISCUSSION

The concluding chapter focuses on analyzing and discussing the results of silica extraction from sand rose and aquifer sand. This is achieved through the use of various physical and chemical analytical techniques to assess the success of the extraction and purification process in obtaining high-purity silica.

4.1 Results And Discussion

4.1.1 Characterization

4.1.1.1 Sieve Analysis

Sieve analysis (or the sieve test) is a commonly used separation technique for determining particle size distribution due to its simplicity, low equipment requirements, and broad applicability[112]. Two types of samples, namely sand rose and aquifer sand, were sieved to determine their particle size distribution, the obtained results are tabulated in tables: 4.1,and 4.2 , respectively. The sieving results showed that all particles in the rose sand sample were concentrated at a size of 224 μm, with 100 wt.% of the sample retained on this sieve. This uniform particle size distribution is attributed to the pre-grinding process, which produced particles of a homogeneous size prior to the sieving analysis. Meanwhile, The sieving results demonstrate that the aquifer sand sample is predominantly medium-sized and granular, with most particles concentrated in the 80 to 315 μm range. The highest percentage of mass (51.88 wt.%) was retained on the 315 μm sieve, followed by 25.45 wt.% and 22.38 wt.% on the 224 μm and 80 μm sieves, respectively. Large (>1250 μm) and very fine (<36 μm) particles were almost nonexistent, representing only 0.014 wt.% and 0.024 wt.%, respectively, indicating a relatively homogeneous particle size distribution.

The presence of small amounts of coarse or very fine particles in a sand rose sample is a desirable property in extraction processes because it promotes homogeneous chemical treatment, including reducing the consumption of chemicals during acid leaching and alkaline dissolution, and it prevents the mass transfer of sand particles during separation, which is reflected in the quality and purity of the resulting silica.

Table 4.1: Mass distribution of 100 g of ground Sand Rose after sieve analysis.

Size.μm	**224**
mass (wt,%)	100

Table 4.2: Mass of sand fractions separated on each sieve for a 100 g Aquifer Sand sample.

Size.μm	**1250**	**315**	**224**	**80**	**36**
mass (gr,%)	0.014	51.88	25.45	22.38	0.024

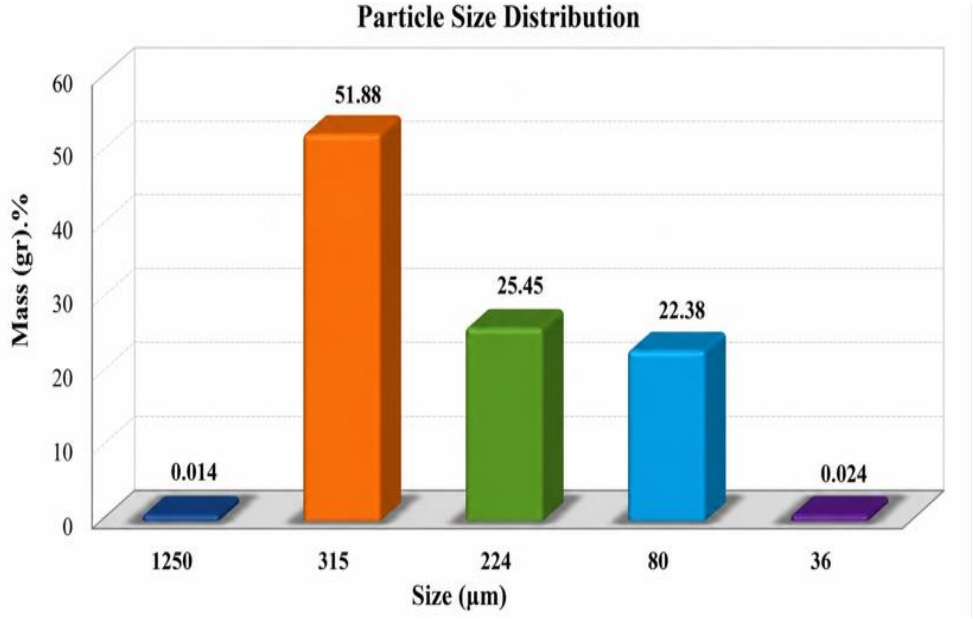


Figure 4.1: 3D column chart of the particle size distribution of the Aquifer Sand sample.

4.1.1.2 XRF Analysis

☛ **a) Sand Rose :**

X-ray fluorescence (XRF) analysis revealed that the Rose Sand sample is predominantly composed of sulfur and calcium, as evidenced by the high content of sulfur trioxide (SO_3: 50.89 wt%) and calcium oxide (CaO: 47.32 wt%).Whereas, the silicon dioxide (SiO_2) content was relatively low, at only 1.208 wt%, indicating that the sand rose sample is of a gypsum or calcareous nature, and the relative yield of silica extracted from this raw material will be very

low. The other oxides detected, detailed in Table: 4.3, were present in very small quantities, not exceeding 0.6g in total, reflecting chemical treatment facilitates their removal or even reduction.

Table 4.3: Elemental content (wt.%) of Sand Rose as determined by XRF analysis.

Element	**K_2O**	**P_2O_5**	**Fe_2O_3**	**SrO**	**Al_2O_3**	**MgO**	**SiO_2**	**CaO**	**SO_3**
wt.%	0.017	0.046	0.069	0.115	0.139	0.186	**1.208**	47.32	50.89

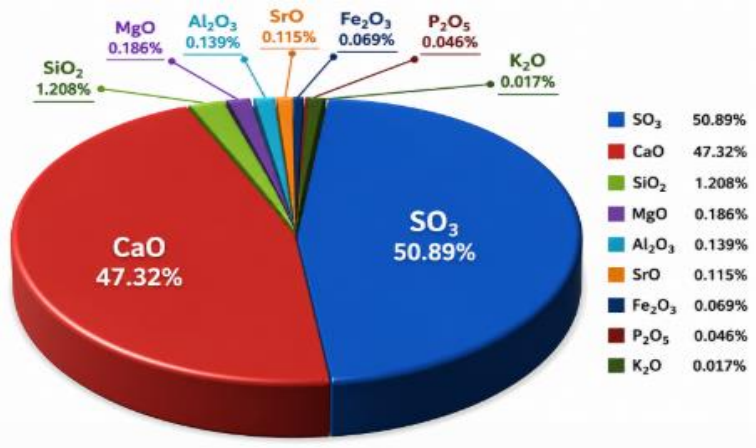


Figure 4.2: 3D pie chart of the oxide composition (wt.%) of the Sand Rose sample determined by XRF analysis.

☛ b) Aquifer Sand:

Table: 4.4 shows the results of X-ray fluorescence analysis of the aquifer sand sample. The results indicate that the aquifer sand sample has a siliceous composition due to silicon dioxide (SiO_2) being the main component at 87.95% by weight, while the remaining oxides did not exceed 13%. Therefore, the aquifer sand is considered a promising raw material for silica extraction compared to rose sand samples. Further purification steps are expected to reduce the concentrations of associated mineral impurities. X-ray fluorescence analysis of the extracted silica will be necessary to evaluate the effectiveness of the extraction and purification process.

Table 4.4: Elemental composition (mass.%) of Aquifer Sand as determined by XRF analysis.

Element	**Rb_2O**	**P_2O_5**	**Cl**	**SrO**	**Fe_2O_3**	**MgO**	**SO_3**	**CaO**	**K_2O**	**Al_2O_3**	**SiO_2**
mass.%	0.008	0.139	0.180	0.289	0.625	0.749	0.887	1.971	2.483	4.705	**87.95**

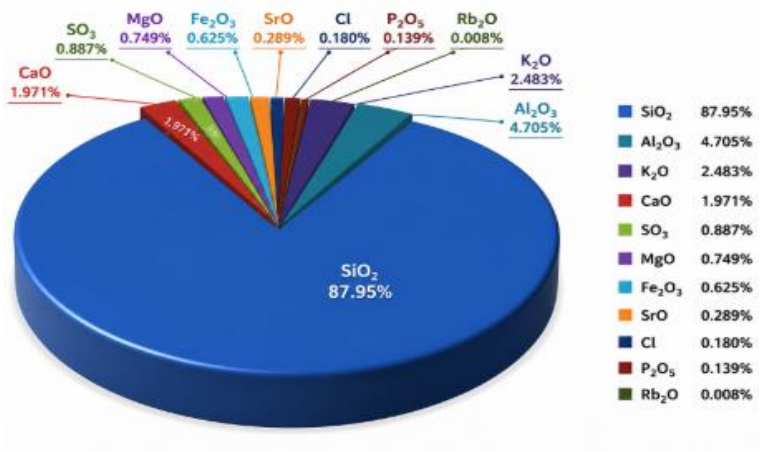


Figure 4.3: 3D pie chart of the oxide composition (wt.%) of the Aquifer Sand sample determined by XRF analysis.

☛ c) After extraction and comparison :

Table: 4.5 compares the oxide composition of the aquifer sand before and after silica extraction. It is evident from the results that the extracted silica was significantly improved by chemical treatment. In particular, the SiO_2 content increased by 9.49%, from 87.95% wt in the raw aquifer sand to 97.44% wt. after extraction. This substantial increase indicates the effectiveness of the extraction and purification process in concentrating the silica and removing a significant proportion of the mineral impurities present in the raw sand. Most of the associated oxides, such as rubium dioxide (RbO_2), strontium oxide (SrO), magnesium oxide (MgO), and potassium oxide (K_2O), were removed, while the proportions of other oxides, such as phosphorus pentoxide (P_2O_5), ferric oxide (Fe_2O_3), Sulfur trioxide (SO_3), calcium oxide (CaO), and aluminum oxide (Al_2O_3), were significantly reduced. However, the increase in chlorine content from 0.180% to 1.001% is attributed to chloride residues resulting from the use of hydrochloric acid (HCl) during the titration process. Overall, the results confirm that the extraction process effectively improved the silica purity of the aquifer sands.

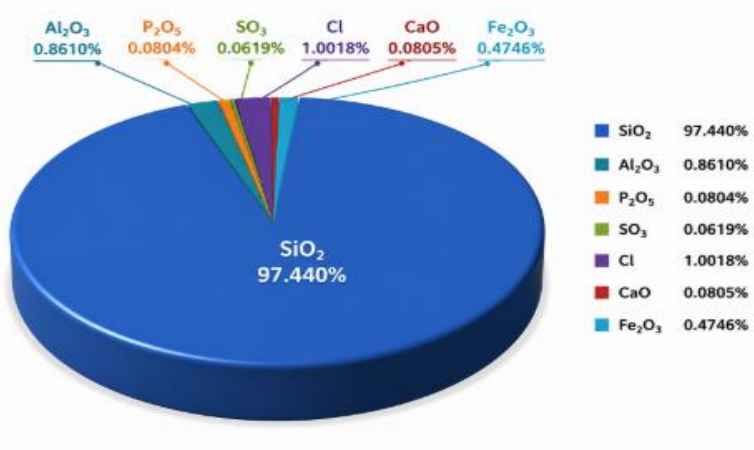


Figure 4.4: 3D pie chart of the XRF analysis of silica extracted from Aquifer sand

Table 4.5: Comparison of the oxide composition (mass %) of aquifer sand before and after extraction determined by XRF analysis.

Oxide	**Mass (%)**	
	Before Extraction	After Extraction
Rb_2O	0.008	–
P_2O_5	0.139	0.080
Cl	0.180	1.001
SrO	0.289	–
Fe_2O_3	0.625	0.474
MgO	0.749	–
SO_3	0.887	0.061
CaO	1.971	0.080
K_2O	2.483	–
Al_2O_3	4.705	0.861
SiO_2	**87.95**	**97.44**

4.1.1.3 Conductivity Measurement

Table: 4.6 Elucidates a significant difference in electrical conductivity between the raw samples (sand rose or aquifer sand) and the extracted silica. The raw sand rose exhibited a relatively high conductivity of 2340 $\mu s/cm$, while the aquifer sand showed a lower value of 576 $\mu s/cm$. This difference is attributed to the presence of dissolved salts and ionic impurities in the raw materials. After extraction, the conductivity of the silica extracted from the aquifer sand decreased significantly to 2.7 $\mu s/cm$ (as depicted in figure: 4.5), indicating substantial removal of dissolved ionic species during the extraction and washing processes. Therefore, the very low conductivity of the extracted silica provides further evidence of the effectiveness of the purification process and the purity of the extracted silica.

Table 4.6: Comparison between the conductivity of the studied raw samples and the extracted silica.

	Conductivity ($\mathcal{K}$: $\mu s/cm$, at 22.5 °C)	
Studied Samples	Raw	Extracted Silica
Sand Rose	2340	-
Aquifer Sand	576	2.7



Figure 4.5: Conductivity value of silica extracted from Aquifer Sand.

4.1.1.4 Surface properties

The surface properties of the silica extracted from aquifer sand were carried out using the nitrogen sorption analysis. The corresponding results are presented in Table: 4.7, and figure: 4.6. It can be seen from Table: 4.7 The extracted silica has a high surface area, with a specific surface area (BET) of 485.2 m^2/g, and Langmuir Surface Area of 690.3 m^2/g, reflecting a highly developed surface area that can provide a considerable number of active sites for surface interactions. In addition, the average particle size and pore width were 12.36 nm and 9.386 nm, respectively. The relatively small particle size indicates the finely divided nature of the extracted silica, whereas the average pore width falls within the mesoporous range (2-50 nm), confirming the predominantly mesoporous structure of the extracted silica. The combination of a high specific surface area and advanced medium porosity suggests that the extracted silica possesses textural properties suitable for applications requiring easy surface access, particularly adsorption and catalysis processes.

The adsorption and de-adsorption curves also show a narrow retardation loop, where the branches of the curve converge and remain nearly parallel within the relative pressure range P/P^0=0.1 to 0.9, before adsorption increases sharply at higher relative pressures. According to the IUPAC classification, this behavior is consistent with Type IV adsorption, exhibiting an H4 retardation loop[116]. This pattern is characteristic of materials with a mesoporous structure, where the latter part of the curve is associated with capillary condensation within the mesoporous structures. Therefore, the shape of the adsorption-deadsorption curve provides further evidence supporting the BJH analysis results, which showed an average pore width of 9.386 nm, within the mesoporous range (2-50 nm), thus confirming the mesoporous nature of the extracted silica.

Table 4.7: Surface and Textural Properties of Silica Extracted from Aquifer Sand.

Parameter(s)	**SiO_2**
BET Surface Area (m^2/g)	485.2
Langmuir Surface Area (m^2/g)	690.3
Average Particle Size (nm)	12.36
BJH Adsorption average pore width (nm)	9.386

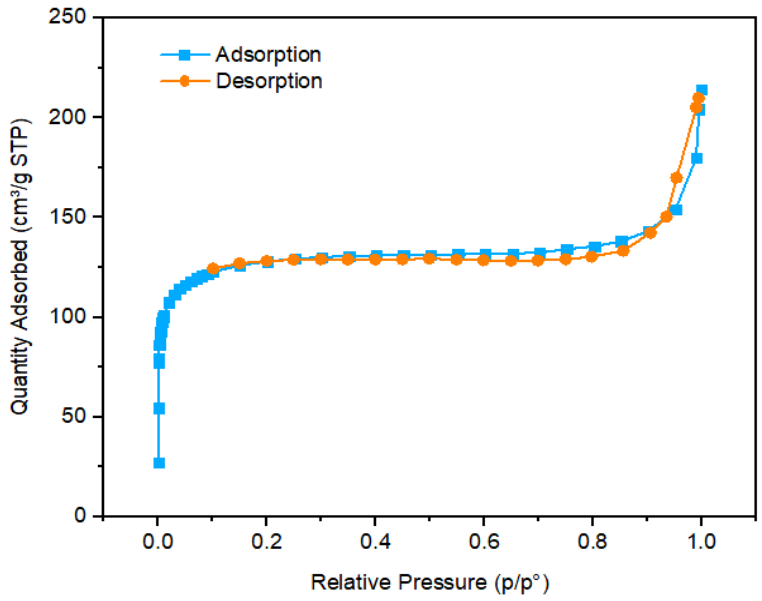


Figure 4.6: Nitrogen adsorption-desorption isotherms of Silica Extracted at 77k.

4.1.1.5 XRD Analysis

The XRD patterns of sand rose, aquifer sand, and silica extracted from aquifer sand were characterized and are plotted in figure: 4.7(a,b, and c). According to figure: 4.7(a), the sand rose sample consists mainly of three mineral phases: gypsum, calcite, and quartz. Gypsum was identified by its strong diffraction peaks at $2\theta = 11.65°$, $23.43°$, $31.37°$, and $33.38°$ [117]. Calcite was identified by the characteristic peaks at $2\theta = 27.34°$ and $29.28°$ [118], while quartz was detected at $2\theta = 20.90°$ and $26.65°$. Similarly in figure: 4.7(b), the aquifer sand sample was detected to predominantly consist of silica, as evidenced by its characteristic diffraction peaks at $2\theta = 20.90°$,$26.64°$, $36.57°$, $39.52°$, $45.65°$, and $54.88°$. In addition, minor phases were identified, with diffraction peaks at $2\theta = 27.34°$ and $40.28°$, which can be attributed to calcite and alumina, respectively. After silica extraction (refer to figure: 4.7(c)), most of the sharp peaks associated with mineral phases disappeared, while a broad peak centered at $2\theta = 22.6°$ was recorded, confirming highly efficient amorphous silica formation [119].

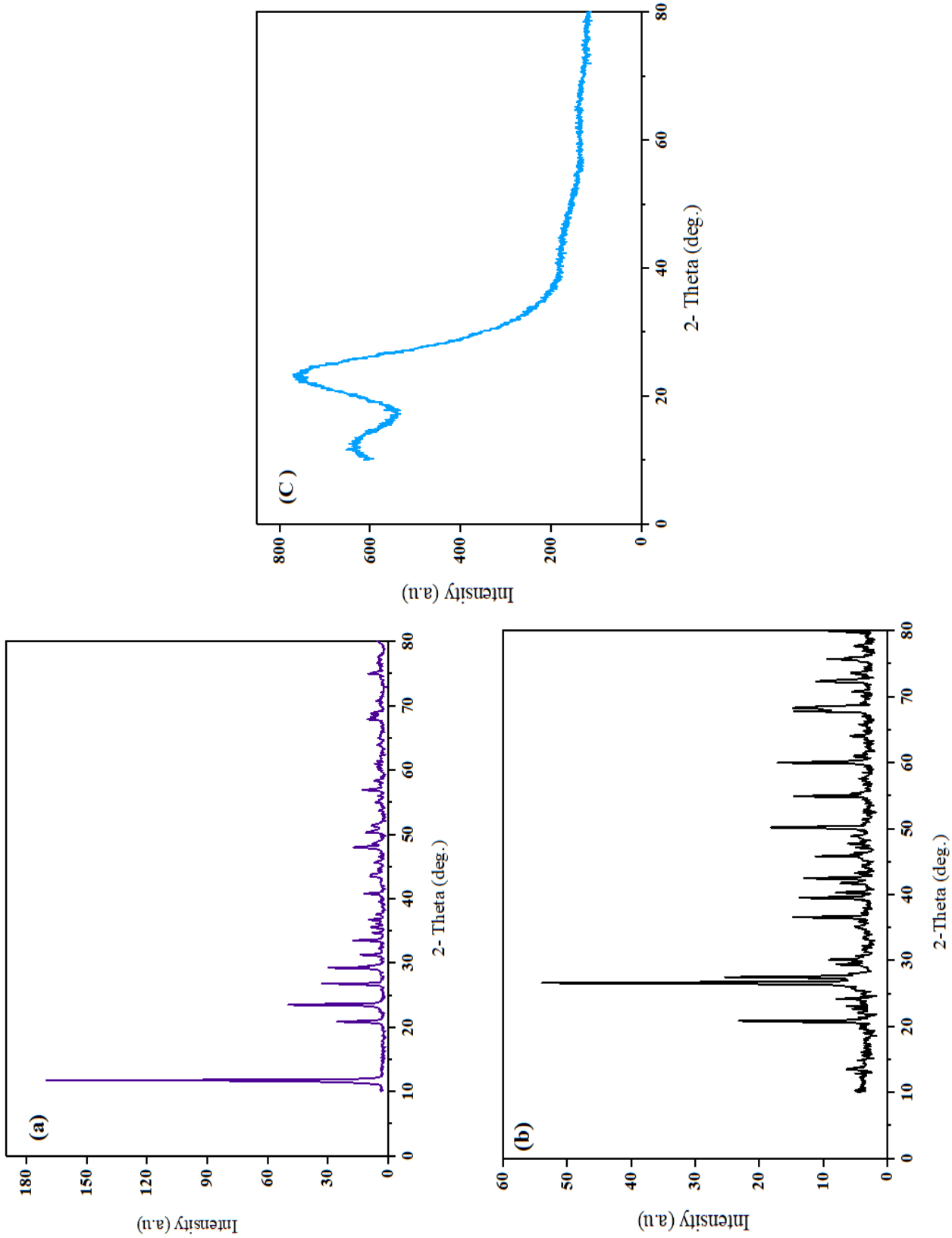
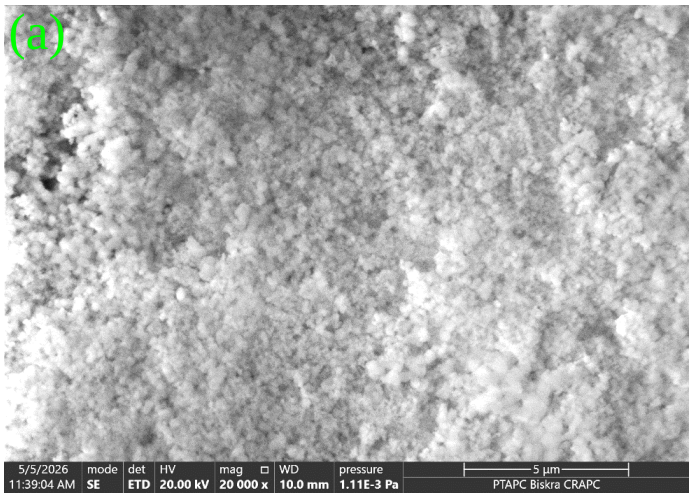


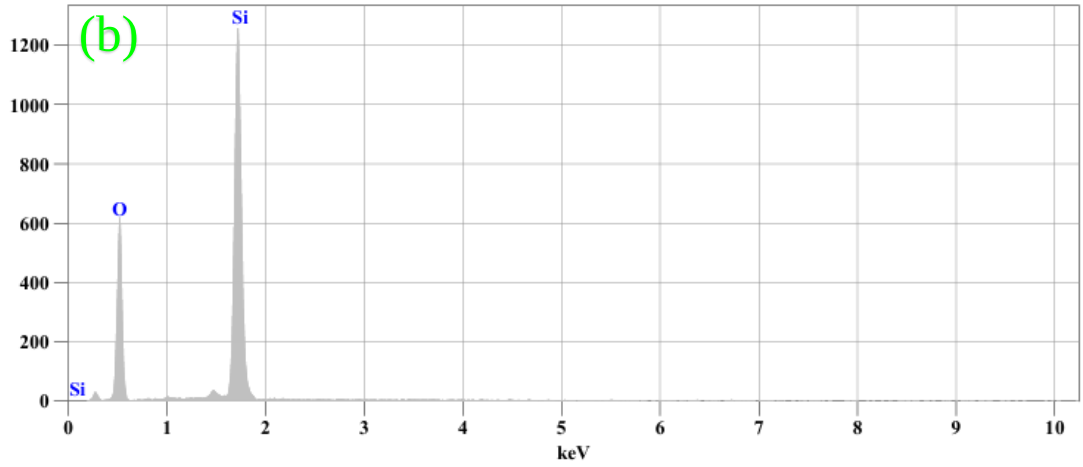
Figure 4.7: XRD patterns of (a) Sand Rose, (b) Aquifer Sand, and (c) extracted silica.

4.1.1.6 SEM-EDX Analysis

The morphology of the silica extracted from aquifer sand and corresponding elemental composition were diagnosted by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX) at a magnification of 20 000x, as illustrated in figure: 4.8.It can be observed from figure: 4.8,a that the silica consists of irregular, fine-grained particles with a relatively irregular structure and porosity. These grains is attributed to the amorphous nature of the extracted silica. This structure also indicates a large number of active surface sites, which is consistent with the previously obtained high specific surface area (BET) value.

The EDX spectrum figure: 4.8,b shows prominent peaks for Silicium (Si) and oxygen (O), the two main components of silica (SiO_2). The high intensity of the silicon peak compared to the other elements is evidence of successful extraction and the production of high-purity silica. These results concur with the XRF results, which showed a high SiO_2 content, confirming the formation of high-purity extracted silica. The absence of peaks for the other elements confirms the removal of most of the mineral impurities present in the raw aquifer sand during chemical treatment.





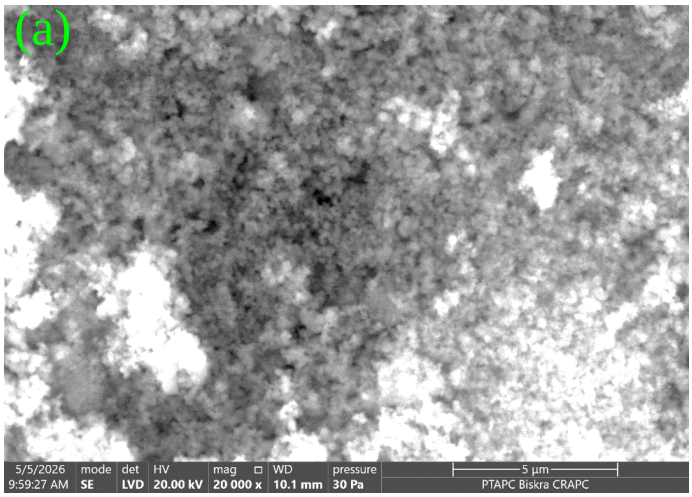


Figure 4.8: (a) SEM images of Silica extracted (SiO_2), and (b) the corresponding EDX spectrum

4.1.1.7 FT-IR spectral results

To characterize the significant structural functions present in the raw sand samples and its extracted, Fourier-transform infrared was used. The FTIR spectra of sand rose, aquifer sand, and extracted silica from aquifer sand are displayed in figure: 4.9 (a,b, and c).

According to spectra (figures: 4.9 (a), and (b)), the spectrum reveals the main chemical functions and structural properties of the studied sand rose and aquifer sand. A set of FTIR spectra between 3429 cm^{-1} to 3700 cm^{-1} are attributed to the stretching vibrations of the (OH) hydroxyl groups[120]. The weaker bands observed at 1616 cm^{-1} are associated with the bending vibration of H-O-H, indicating the presence of physically adsorbed water. Several distinct absorption peaks were observed at approximately 2350, 1427, and 876 cm^{-1}. These bands can be attributed to carbonate (CO_3^{2-}) vibrations, including the asymmetric stretching and bending modes of the carbonate group. The presence of these characteristic carbonate bands provides evidence for the presence of calcite ($CaCO_3$) in the sand rose and aquifer sand samples[121]. Moreover, It also showed distinct absorption peaks at 462 and 1100 cm^{-1}, which are attributed to the characteristic vibrations of the Si-O-Si bonds. Additionally, peaks appeared at approximately 693 and 799 cm^{-1}, which are associated with the symmetric and asymmetric stretching vibrations of the Si-O bonds, respectively. These peaks indicate the presence of quartz as a component of the two samples. Furthermore, a peak was observed at approximately 523 cm^{-1}, which can be attributed to the asymmetric bending vibration of the Si-O-Al bonds[122].

After extraction process, The disappearance of some absorption bands associated with hydroxyl groups (OH) is clearly observed in the region between 3400 and 3700 cm^{-1}, as well as in the range from 1300 to 1700 cm^{-1}. A significant decrease in the intensity of some bands associated with carbonate components, particularly the band at approximately 2350 cm^{-1}, was also noted in the spectrum of the extracted silica (figure: 4.9 (c)) compared to the spectra of the sand rose and the aquifer sand (figures: 4.9 (a), and (b)). This decrease and the disappearance of some bands associated with non-silica components indicate the removal of a significant portion of impurities during the extraction process. Therefore, the FTIR results confirm the successful extraction of silica from the subsurface sand sample, yielding a silica-rich material of high purity.

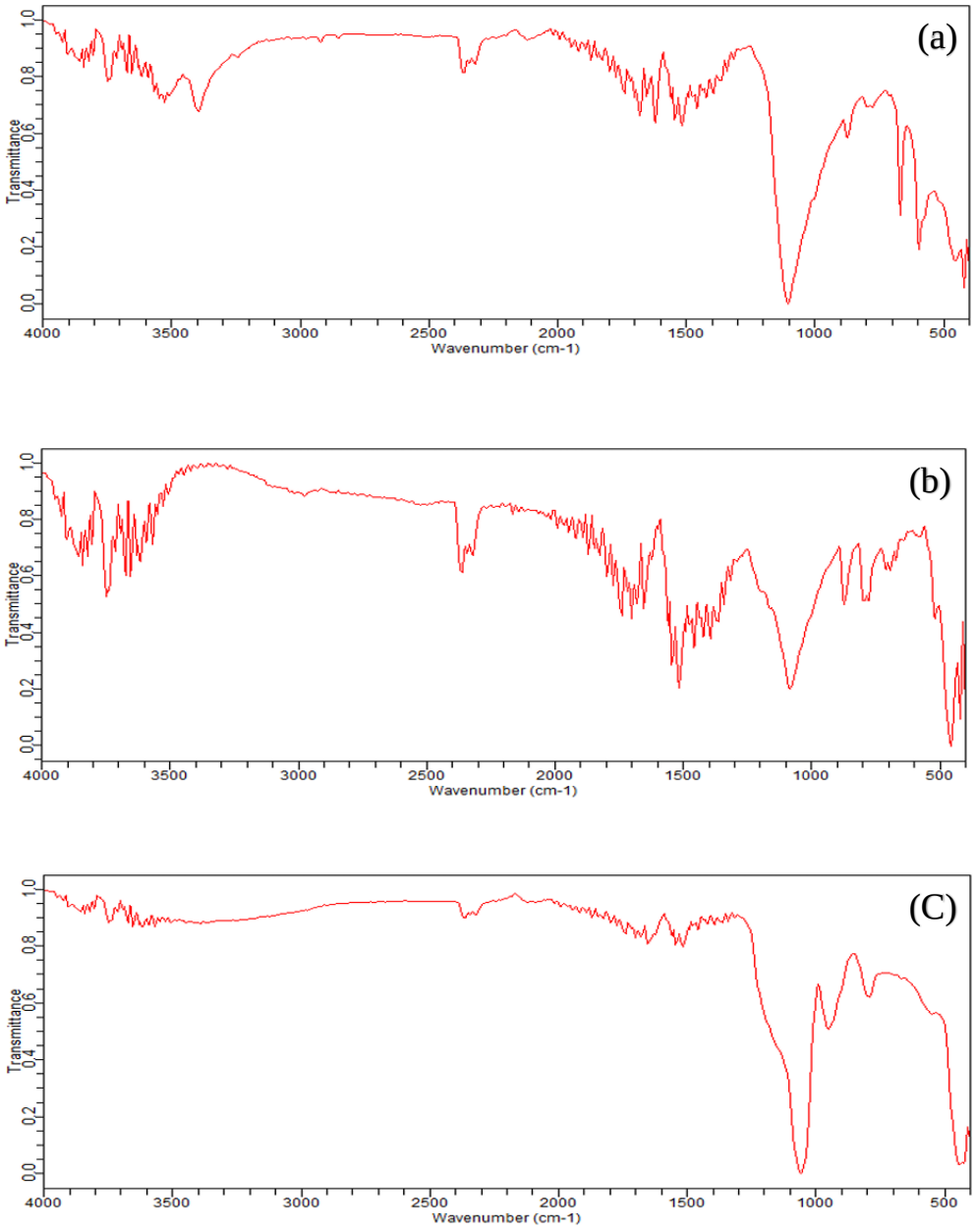


Figure 4.9: FTIR spectra of : a) Sand Rose, b) Aquifer Sand, and c) Extracted Silica.

CONCLUSION

In conclusion, this study confirmed the feasibility of successfully preparing silica from two local natural sources, namely sand rose and aquifer sand, using the sol-gel method. A protocol was adopted to minimize the consumption of chemicals and energy required for the extraction process. The results showed that aquifer sands represent a promising source for silica production, with an extracted silica yield of 97.44% and a very low electrical conductivity of 2.7 μS/cm, reflecting its high purity. The extracted silica also exhibited a high specific surface area of 485.2 m^2/g. X-ray diffraction (XRD) analysis confirmed the presence of a broad spectrum centered at $2\theta = 22.6°$, further confirming the amorphous nature of the extracted silica.

In light of the results obtained and mentioned above, this study achieved the following specific objectives:

✍ **1.** Silica was successfully prepared from rose sand , although the extraction yield was limited. This is mainly attributed to the low silica content in the raw material, which was present at a limited percentage not exceeding 1.208%.

✍ **2.** Silica was successfully prepared and characterized from aquifer sand. The silica content in the extracted product reached 97.44%, highlighting its potential as a promising local source for producing high-purity silica.

✍ **3.** Chemical and energy consumption was reduced: The adopted protocol contributed to reducing the amount of chemicals and energy used in the extraction process. The concentration of hydrochloric acid was reduced from 6 M to 3 M, the ratio of sodium hydroxide to sand was reduced from 7:1 to 3:1, and the heat treatment temperature was reduced from 900 °C to 500 °C. This reflects the potential for developing a more economical and less resource-intensive extraction process.

✍ **4.** The results of various characterization techniques confirmed that the extracted silica is amorphous and has a nanostructure, based on XRD and SEM analysis.

Therefore, this study confirms that aquifer sands, in particular, represent a promising local raw material with potential economic value for the production of high-purity amorphous silica. These results also open the window to future studies on aquifer sands and expanding the application of extracted silica in industrial and environmental fields.

RECOMMENDATION

Based on the findings of this study, the following recommendations can be proposed:

➤ Reconsider the use of sand rose as a source for silica extraction, given its low silicon dioxide content and the high proportion of associated mineral components.

➤ Prioritize the use of aquifer sands as a promising local source for silica extraction, owing to their high silicon dioxide content and potential for utilization in various industrial applications.

➤ Continue to develop and refine the silica extraction protocol for aquifer sands, aiming to increase extraction yield and enhance the product's purity as well as its physical and chemical properties.

➤ Investigate the feasibility of scaling up the silica extraction process from the laboratory level to pilot and industrial scales, while evaluating the process's economic and technical viability and its potential for large-scale implementation.

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