

Sudan University of Science and Technology



College of Graduate Studies

PREPARING AND STUDYING OPTICAL PROPERTIES OF SILICA NANOPOWDER

تحضير و دراسة الخصائص الضوئية لبدرة السيليكا الناتوية

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الآيـــة

قال تعالى:

بسم الله الرحمن الرحيم

(اللَّهُ نُورُ السَّمَاوَاتِ وَالْأَرْضِ مَثَلُ نُورِهِ كَمِشْكَاةٍ فِيهَا مِصْبَاحُ الْمِصْبَاحُ فِي زُجَاجَةٍ الزُّجَاجَةُ كَأَنَّهَا كَوْكَبٌ دُرِّيٌّ يُوقَدُ مِن شَجَرَةٍ مُّبَارَكَةٍ زَيْتُونِةٍ لَّا شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ نُورٌ عَلَى ذُرِيِّ يُوفَدُ مِن شَجَرَةٍ مُّبَارَكَةٍ زَيْتُونِةٍ لَا شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ نُورٌ عَلَى نُورٍ يَهْدِي اللَّهُ لِنُورِهِ مَن يَشَاءُ وَيَضْرِبُ اللَّهُ الْأَمْتَالَ لِلنَّاسِ وَاللَّهُ بِكُلِّ شَيْءٍ عَلِيمٌ)(35).

صدق الله العظيم

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At this beautiful moment where my master study is about to finish after three years of hard studying and hardworking, I would like to specialize my thank you's to my mother and father for their unstoppable support for twenty eight years, I also want to extend my thank you's to my family for their continuous support during my study of Bachelor's degree and Master's degree and my supervisor Dr. Rawia Abdelgani who paid her time and effort to supervise this work, special thank you's to Dr. Abdelsakhi from Alneelain University who played significant role in guiding me until this work was achieved experimentally, there are awesome friends and colleagues we kept to be together during this long journey I send them my sincere thank you's and gratitude.

Abstract

Silica nanoparticles is one of the most important materials utilized in many applications and industries, it is mainly used in glass industry and in electronic devices such as Nanotransistors and Nanosensors. In the present study, preparation of silicon dioxide nanoparticles SiO₂ from natural sands was conducted by Alkali fusion method, samples are taken from different areas (Shandi, Dongla, western Bara, southern Bara), in Sudan, and characterize the optical properties of these samples. The alkali media method was performed on each sample, the sand was immersed in hydrochloric acid HCl using magnetic stirrer-hotplate, then certain amount of sodium hydroxide NaOH was mixed with crude silica sand very well and heated until the mixture became liquid and stiffened again, then dissolved with water, and filtered, after that the sulfuric acid H₂SO₄ was added to the resulting mixture to form SiO₂. It turned out that the extraction and synthesis of SiO_2 nanoparticles by means of alkali-fusion method proved that it is successful method in forming silica nanoparticles; the obtained SiO₂ was characterized by Fourier Transform Infrared spectrometry (FT-IR), and through Ultraviolet-visible spectrometry (UV). FT-IR spectral data confirmed the chemical functional groups which are present in every silica nanoparticles and different functional groups were reported for each sample, UV spectra proved that different impurities that exist in each silica sands of different locations have influence on optical properties, the result was that the energy band gap of SiO₂ increases as we go north and decreases as we go south in geographical location.

المستخلص

تعتبر مادة السيليكا النانوية من اهم المواد، تستخدم في عدة تطبيقات منها الصناعات الزجاجية و تصميم الاجهزة الالكترونية مثل الترانسستورات النانوية و الحساسات النانوية، هذه الدراسة صممت لتحضير جسيمات السيليكا النانوية من الرمل الطبيعي حيث تم اخذ هذه العينات من مناطق (شندي، دنقلا، غرب براء جنوب بارا) في السودان، و دراسة الخصائص الضوئية لهذه العينات، تم استخدام طريقة -Alkali nagnetic stirrer لكل العينات. غمس الرمل في حامض الهيدروكلوريك HClباستخدام -Philamidal العنام و تم خلطهم hotplate لكل العينات كمية محددة من حمض هيدروكسيد الصوديوم NaOH و الرمل الخام و تم خلطهم جيدا، ثم سخن الخليط حتى اصبح سائلا ثم تصلب مرة اخرى، تم عمل محلول من الخليط ثم تمت فلترته، بعد ذلك تمت اضاف حمض السلفيوريك H_2SO4 الي المحلول الناتج لتكوين ثاني اوكسيد السيليكون، لقد Alkali-fusion و تحضير جسيمات ثاني اوكسيد السيليكون النانوية بواسطة طريقة واسطة مطياف يعطي نتائج ناجحة، تمت دراسة الخصائص الضوئية لجسيمات SiO_2 النانوية الناتجة بواسطة مطياف الاشعة الفوق البنفسجية SiO_2 النانوية، و اوضحت مجموعات الكيميائية الموجودة في اي عينة لجسيمات SiO_2 النانوية، و اوضحت مجموعات الموجودة في عينات السيليكا لها تاثير على الخصائص الضوئية لجسيمات SiO_2 النانوية حيث ان فجوة الموجودة في عينات السيليكا لها تاثير على الخصائص الضوئية لجسيمات SiO_2 النانوية حيث ان فجوة الموجودة في عينات السيليكا لها تاثير على الخصائص الضوئية الجسيمات SiO_2 النانوية حيث ان فجوة الموجودة في عينات السيليكا لها تاثير على الخصائص الضوئية الجسيمات SiO_2 النانوية حيث ان فجوة الموجودة في عينات السيليكا لها تاثير على الخصائص الضوئية الجسيمات SiO_2 النانوية حيث ان فجوة الطبقة تزداد كل ما اتجهنا الى الشمال الجغرافي و تقل كلما اتجهنا الى الشمال الجغرافي و تقل كلما اتجهنا الى المورودة و

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CHAPTER ONE INTRODUCTION

1.1 Introduction

Silica sand in general is considered to be one of the most freely available natural materials and it has different forms in the world. Research of silica nanoparticles has increased because of their easy preparation and extensive use in the production of novel systems and applications such as Nanoresonators, Nanosensors, Bone Application, Biomedical applications and Nanowire based devices etc [1]. Silica (SiO₂) is present naturally in three major phases, i.e. quartz, tridymite, and cristobalite, It is utilized as a raw material in many industries, such as glasses, mirrors, mosaic ceramics, Ferro silicon [2]. But the best-known form of silica is amorphous silica, therefore, amorphous silica continues as the focus of much fundamental research to understand its electronic structure, bonding characterization, defects, and optical properties [3]. Chemical processes, like extraction, purification, and synthesis of silica nanoparticles from natural materials have extensively conducted, for instance, amorphous SiO₂ nanopowders with high purity over 95% have been synthesized by chemical method (sol gel and hydrothermal) using organic materials like baggase ash [4]. Meanwhile inorganic material such as natural silica sand has also been utilized to synthesize SiO₂ nanoparticles via high energy milling [5], and by means of extraction process using alkali compounds KOH, NaOH with heating temperature of mixture over 360°C to form sodium silicate (Na₂SiO₃) [6]. However, naturally sand is an oxide that exists mixing with other minerals, it is important to separate these minerals from silica sand to obtain high purity silica. Besides, it is also challenging to fabricate amorphous silica with nanometer size from natural sand.

1.2 Silica SiO₂ Structure

Silicon dioxide is a three-dimensional siloxane bridged bond structure Si-O-Si, usually utilized as an amorphous or nanocrystalline form. Structural silica has tetrahedral units (one silicon atom is bonded to four oxygen atoms, most oxygen atoms will be bonded to two silicon atoms) connected to the corners by bridging oxygen atom O. The ideal structure of two-dimensional silica has linear Si-O-Si bonds, as illustrated in figure 1, where the torsional energy required for one bond rotation depends on the bond angles that vary between 145 and 150 [7].

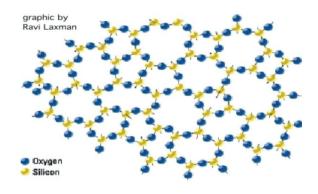


Figure 1: Bonding structure of tetrahedron amorphous silica from Ref [7]

1.2 Thesis Problem

The problem of this study is that there are some impurities with specific chemical composition and different functional groups present in silica sands depending on locations of sands, to what extent that SiO₂ nanoparticles resulting from treating these different silica sands samples get affected in terms of optical properties.

1.3 Importance of the Research

New era of technology demands a cheaper and more commercial materials and methods to optimally synthesize nanoparticles, silica sand is an example of these materials. Research of silica nanoparticles has increased because this material can easily be synthesized and widely used in the production of novel systems and applications in industry. In Sudan, as a developing country with large areas containing natural sands, it is convenient to do a research on a various samples from different areas.

1.4 Research Objectives

The main objectives of this research is to

- 1. Synthesize silica nanopowder from natural silica sands of different samples as a raw material via alkali-fusion method (dry method).
- **2.** Studying the optical properties of each sample.

1.5 Research Layout

The research contains four chapters: chapter one, represents the introduction of the research, the thesis problem, the importance of the research, and the overall objectives. The second one speaks about literature reviews which show the previous studies carried out in the same topic but via various methods. Chapter three is about the methodology of the research, the extraction of the sample and explaining how the alkali-fusion method has been employed and overall steps done to obtain silica nanoparticles. Chapter four represents the results and the discussion of the research with the conclusion.

CHAPTER TWO

THEORTICAL BACKGROUND AND LITERATURE REVIEW

2.1 Nanomaterials

Nanomaterials are considered the cornerstones and basic building blocks of the study of Nanoscience and Nanotechnology, they are defined as materials where at least one dimension is less than 100 nm, nanoscale materials have been interesting since their discovery because of their unique electrical, optical, magnetic, chemical and mechanical properties emerge at this small scale, these new properties actually have the potential to impact greatly in electronics, optoelectonics, medicine and other fields. Generally they are classified into three categories based on dimensions; zero dimension nanomaterials include spheres and clusters, etc. one dimension nanomaterials such as nanofilms, nanoplates, and nanoparticles or nanopowders represent three dimension nanomaterials [8].

2.2 Synthesis Techniques of Nanomaterials:

There are two approaches that nanoscale materials can be synthesized by, top-down approach, and bottom-approach, each approach includes several synthesis methods [9].

2.2.1 Top-down Approach

In this approach bulk materials are broken down into smaller sizes, when the process continues the material ends up having nanostructure form, the main advantage of this, it leads to crystalline structure of samples with few defects such as single crystal or polycrystalline material, the synthesis methods obey this

approach are for example, mechanical milling, laser ablation, and photolithography techniques [9].

2.2.2 Bottom-up Approach

In this approach nanostructure particles are synthesized by gas phase or liquid phase chemical reactions collecting atom by atom, it results in the formation of small particles, synthesis methods such as Hydrothermal method, Sol-Gel method, Alkali media (dry method), and Chemical Vapor Deposition method follow bottom-up approach [9].

2.3 SiO₂ Nanoparticles Synthesis Techniques

Silica nanoparticles actually can be synthesized by high energy ball milling method which is top-down approach and hydrothermal, sol-gel, and alkali fusion methods which belong to bottom-up approach.

2.3.1 High Energy Ball Milling

Suitable powder charge of material is placed in high energy mill, along with a suitable milling medium, different types of all milling can be used for synthesis of nanoparticles in which balls impact upon the powder charge, this method is used for producing large quantities of fine nanoparticles. The kinetics of mechanical milling or alloying depends on the energy of transferred to the powder from the balls during milling, energy transfer is governed by many parameters such as the type of mill, amount of powder, milling speed, size of balls, dry or wet milling, temperature of milling, and the time of milling [10].

2.3.2 Hydrothermal Method

Generally hydrothermal synthesis is defined as crystal growth and synthesis under high temperature and high pressure water conditions from substances that are not soluble in water under normal temperature and pressure (T< $100 \, \text{C}$, P< $1 \, \text{atm}$), and only water is used as solvent [11]. Most hydrothermal reactions are

performed in sealed reactor called autoclave, a pressure vessel or high pressure bomb. Usually hydrothermal reactors are metal autoclaves. The working conditions of autoclaves vary for different materials including glass, quartz, and strength alloys. Temperature, pressure, and corrosive resistance of reactor materials are the most important parameters for selecting the reactor; the pressure generated in a sealed vessel is always controlled and estimated below the strength of autoclave materials for safety concerns [12].

2.3.3 Sol-Gel Method

It is liquid phase synthesis method; the temperature in this process is low for nanoscale production of nanomaterials, so it can be performed at room temperature [13]. This method uses compounds that contain high chemically active components as precursor, it uniformly mixes these raw materials in the liquid phase and performs hydrolysis and condensation chemical reactions to form a stable transparent sol system in solution, the sol slowly polymerizes between the aged colloidal particles to form gel with a three dimensional structure. The gel is dried, sintered and solidified to prepare molecular or nanostructure materials [14].

2.3.4 Alkali fusion Method

It is one of the bottom up methods that conducted by fusing the particles with alkali media such as sodium hydroxide at certain temperature range from 400 – 1100 C and then recrystallizing the molecules into nanosize particles, the obtained nanoparticles will have gel form (nano gel), the gel will be dried in a furnace with suitable temperature to form fine nanopowder [15].

2.4 Previous Studies on Silica Nanoparticles

2.4.1 Preparation and Characterization of Silica Nanoparticles by Wet Mechanical Attrition of White and Yellow Sand

This study was carried out by Magda A Akl, Hesham F Aly and et al, in 2013, preparation of silica nanoparticles SiO₂ by wet mechanical attrition of white and yellow sands using a lab scaled ball mill. The prepared of SiO₂ nanoparticles characterized by scanning electron microscope (SEM), diffractometry (XRD), energy dispersive spectroscopy (EDS), Fourier Transform Infrared (FTIR). Different experimental factors affecting the mill process were thoroughly studied such as milling duration time, water volume used and the initial size of sand particles. XRD figures showed the general silica characteristic for white and yellow sands with an intense sharp peak centered at $2\theta = 26$ which indicate that the sample are crystalline with crystal size 26.776 nm and 25.455 nm for milled white and yellow sands respectively. SEM data revealed that resulting SiO₂ nanoparticles have particle size range of 23-38 nm for white sand and yellow sand using water as wetting agent for 8hr and 400 rpm speed. FTIR spectra of silica obtained show that SiO₂ nanoparticles have common chemical bonds silanol (Si-O), siloxane (Si-O-Si) and water molecules which are usually present in silica nanoparticles, the bands of IR corresponding are assigned to various vibrations in the solid ranging from $400 cm^{-1} - 4000 cm^{-1}$ [16].

2.4.2 Synthesis of SiO₂ Nanopowders Containing Quartz and Cristobalite Phases from Silica Sands

In 2015, a study done by Munasir, Triwikantoro and et al, on synthesis and characterization of SiO₂ nanoparticles from silica sands, it was performed by two different methods, dry method and hydrothermal process. The SiO₂ nanoparticles were characterized by XRD to investigate the phase formation and crystal structure, X-ray fluorescence (XRF) to evaluate the elemental composition, FTIR

to study the silica functional groups, SEM and TEM to demonstrate the morphology and microstructural properties. form XRD patterns, it was observed for method 1 and method 2 samples of SiO_2 nanoparticles consist of amorphous phase and small amount of crystalline phases, it turned out that there exists both quartz and cristobalite phases for both samples, the amount of crystalline phases were 19.84% and 17.99% respectively, meanwhile estimated crystalline size was 81 nm for dry method sample and 67 nm for hydrothermal method sample. FTIR data of SiO_2 nanoparticles revealed that from both methods bands that are typical and common ranging from $400 \ cm^{-1} - 3500 \ cm^{-1}$. XRF data analysis revealed that resulting SiO_2 nanoparticles have purity as high as 98.9% for method 1 sample and 98.30% for method 2sample. SEM figures reported that some agglomerated SiO_2 particles with sphere-like morphology, and TEM show that the microstructure of both samples of SiO_2 nanoparticles having particle size around 72 nm [17].

2.4.3 Synthesis and Characterization of SiO_2 Nanoparticles by Sol-Gel Process and Its Degradation of Methylene Blue

This work was performed by Ruchi Nandanwar and et al, the study deals with the sol-gel synthesis of SiO_2 material and also provides a basic understanding of the effect of calcination temperature on the growth of SiO_2 by hydrolysis of TEOS with ethanol, deionized water and catalyst mixture. The properties of resulting materials were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), and optical properties through UV-visible spectroscopy and Photo Luminescence (PL). The XRD study of pure SiO_2 with calcination temperature at 300° C shows that material formed SiO_2 nanoparticles have hexagonal crystal structure referred to quartz phase in amorphous SiO_2 nanoparticles, and the primitive lattice with lattice parameter a = b = 4.913 A and c = 5.405 A, also at calcination temperature the crystallite size is 50.55 nm. SEM results revealed that obtained silica nanoparticles were having spherical

morphology in regular shape. UV-visible spectra of silica sample have wide band gap showing absorbance in the ultraviolet region, the absorption edges at around 351 nm at 300 C calcination temperature, the corresponding band gap to this wavelength is 3.53419eV [18].

2.4.4 Synthesis of Silica Nano powder produced from Indonesian Natural Sand via Alkali-fusion Route

The present study was carried out in 2013 by Munasir, Sulton A, and et al on extraction of silica Nano powder, it is conducted by means of alkali fusion route using sodium hydroxide, it was designed to obtain amorphous silica, resulting SiO₂ nanoparticles was characterized by XRF to study the elemental composition, XRD to investigate the phase formation and crystallinity, TEM for evaluating particle size and microstructure, from XRF table it was found that the silica sand inherently is dominated by Si, Ca, Fe, or in terms of oxides, SiO₂, CaO and Fe2O3 with concentration 65.90% of SiO₂. XRD pattern of produced SiO₂ nanoparticles exhibits a wide peak and low intensity indicating that the silica powder is an amorphous phase with particle size less than 100 nm and a crystalline phase (quartz phase) also formed in the sample. TEM figure revealed that the morphological profile of silica nanopowder has spherical agglomerated grains having particle size of nanometer < 200 nm [19].

2.4.5 Radhip N R, Optical Properties Study for Silica Nano Particle from Indian Sand

Optical properties of silica nanoparticles were studied by Radeep N.R, et al, in 2015 using different characterization techniques, Particle size analyzer, Raman spectroscopy, Fourier Transform Infrared spectroscopy. The silica samples were synthesized by ball milling for different durations, 50hrs, 75hrs, 100hrs, 125hrs, 150 hrs. From particle size analyzer data, the particle size ranges between 94 - 468 nm for 150hrs milled powder but for 50hrs it is between 371-468 nm, it turned out that there is linear reduction of particle size range from 50hrs to

150hrs, the particle size range peak becomes narrower with increasing milling hours. Raman spectral data reveal that for different milling time, powder shows the relatively same Raman spectra, from 50hrs to higher milling time, the intensity of Raman spectra was decreased, so as the particle size decreases the intensity of the peak decreases. The spectra of all samples obtained by FTIR show the absorption band at from $461cm^{-1}$ to $467cm^{-1}$ which corresponds to the Si-O rocking vibration, the band appeared at $798cm^{-1}$, $799cm^{-1}$ is due to the Si-O bending vibration, the band around $1089cm^{-1}$ correspond to asymmetric stretching vibration of Si-O-Si band, the peak appeared at about $3435cm^{-1}$ is related to the O-H stretching vibration of H_2O in the sample. FTIR analysis also showed that all the bands were slightly shifted towards lower number as the particle size decreased [20].

CHAPTER THREE

EXPERIMENTAL METHOD

3.1 Materials and Equipment

In this study, the following materials have been used to perform the experimental method, silica sands from different areas, 35 % HCL, 98% NaOH, 95-97% H₂SO₄, DI water. The equipment were beaker glass with sizes of 100 ml, 400 ml; measuring glass, balance, crucible, boiling flask, filter paper, magnetic stirrer-hotplate, vessel glass, oven.

3.2 Procedures

3.2.1 Extraction of SiO₂

Procedure: purification of SiO₂ was performed via alkali-fusion Method, samples of sands were taken from (Shandi sample 1, Dongla sample 2, western Bara sample 3, southern Bara sample 4), Sudan, this method was used for each sample. Crude sand was Weighted about 150 grams, and then 100 ml of HCL with concentration (7.1 M) was added to a beaker, The acid was transferred to a magnetic stirrer-hotplate to dissolve the sand's impurities. After heating, the sand was added slowly as CO₂ produced according to the equation.

$$CaCO_3 + 2HCL \rightarrow CO_2 + H_2O + CaCL_2$$
 (3.1)

To condense the acid vapor; boiling flask with ice water was placed on the top of the beaker. The heating would continue until no more bubbles were produced, after that, the spent acid was decanted and the acidic sand was washed with water until it became gray in color. The pure sand was transferred to a paper plate for drying; the sand was weighted again and reported an average around 140g.

3.2.2 Purification of SiO₂

Procedure: about 80 grams of NaOH weighed and put in a stainless steel crucible, 60 grams of the crude SiO_2 sand was added to NaOH and mixed well, and then the mixture was heated up until became liquid and then stiffened again.

$$2NaOH(s) + SiO_2(s) \rightarrow Na_2SiO_3(s) + H_2O(g)$$
 (3.2)

After that, quickly the mixture was removed from the crucible and put in a beaker, then added 400 ml of $\rm H_2O$ to dissolve the mixture, the solution was filtered and transferred into glass vessel, To it, added about 25 ml of 95-97% sulfuric acid drop by drop.

$$2Na_2(SiO_3) + H_2SO_4 \rightarrow 2(SiO_2) + 2Na_2SO_4 + H_2O$$
 (3.3)

The mixture filtered and washed with water, after drying it in oven $(475C^0)$ for 25 minute), Excess Silicic acid decomposed and SiO_2 was formed, finally pure silicon dioxide grinded into a fine powder and weighed again.

3.3 Fourier Transform Infrared Data

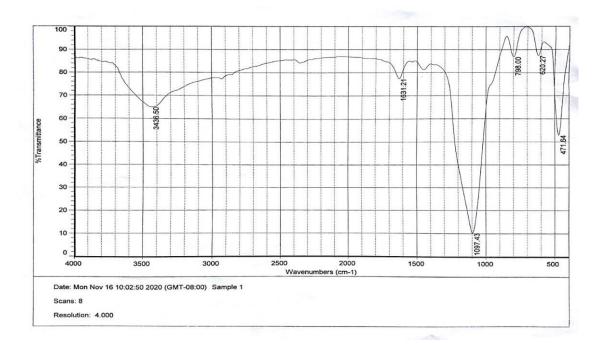


Figure (3.1) FT-IR spectrum of SiO_2 nanoparticles for Sample 1

Table (3.1) IR spectral data of SiO_2 nanoparticles for Sample 1

Wavenumber cm ⁻¹	Functional Groups	Type of Vibration
3436.50	water molecule	O-H stretch
1631.21	water molecule	O-H bend
1097.43	Siloxane	Si-O-Si asymmetric stretch
798.00	Siloxane	Si-O-Si symmetric stretch
620.27	Silanol	Si-O stretch
471.64	Siloxy	Si-O bend

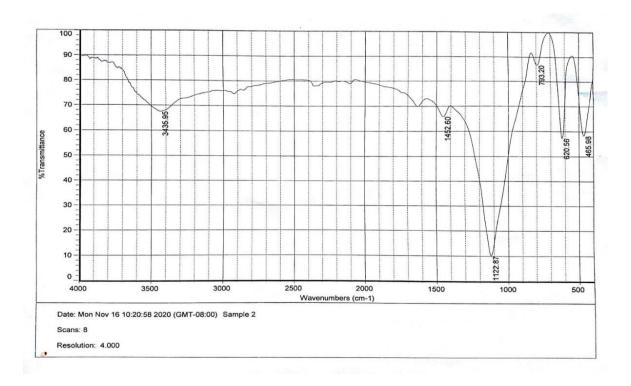


Figure (3.2) FT-IR spectrum of ${\rm SiO_2}$ nanoparticles for Sample 2

Table (3.2) IR spectral data of ${\rm SiO_2}$ nanoparticles for Sample 2

Wavenumber cm ⁻¹	Functional Groups	Type of Vibration
3435.95	water molecule	O-H stretch
1452.60	Alkene	C-H bend
1122.87	Siloxane	Si-O-Si asymmetric stretch
793.20	Siloxane	Si-O-Si symmetric stretch
620.56	Silanol	Si-O stretch
465.98	Siloxy	Si-O bend

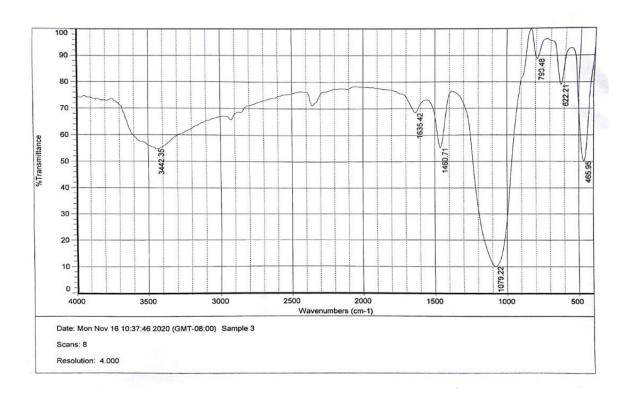


Figure (3.3) FT-IR spectrum of SiO_2 nanoparticles for Sample 3

Table (3.3) IR spectral data of SiO_2 nanoparticles for Sample 3

Wavenumber cm ⁻¹	Functional Groups	Type of Vibration
3442.35	water molecule	O-H stretch
1635.42	water molecule	O-H bend
1460.71	Alkene	C-H bend
1079.22	Siloxane	Si-O-Si asymmetric stretch
790.48	Siloxane	Si-O-Si symmetric stretch
622.21	Silanol	Si-O stretch
465.95	Siloxy	Si-O bend

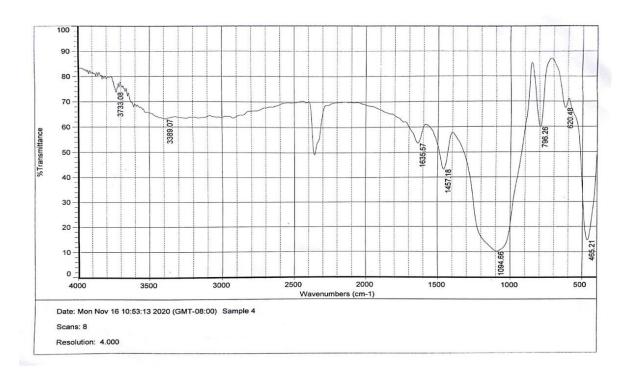


Figure (3.4) FT-IR spectrum of SiO_2 nanoparticles for Sample 4 Table (3.4) IR spectral data of SiO_2 nanoparticles for Sample 4

Wavenumber cm ^{−1}	Functional Groups	Type of Vibration
3733.08	water molecule	O-H stretch
3389.07	water molecule	O-H stretch
1635.57	water molecule	O-H bend
1457.18	Alkene	C-H bend
1094.66	Siloxane	Si-O-Si asymmetric stretch
796.26	Siloxane	Si-O-Si symmetric stretch
620.48	Silanol	Si-O stretch
465.21	Siloxy	Si-O bend

3.4 Ultraviolet visible Data

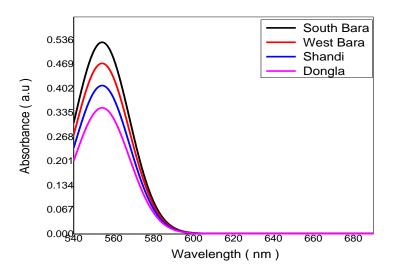


Figure (3.5) the absorbance spectra for four samples of SiO_2

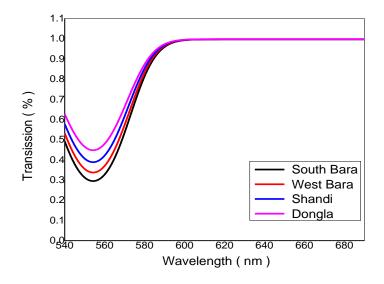


Figure (3.6): The transmittance spectra for four samples of SiO_2

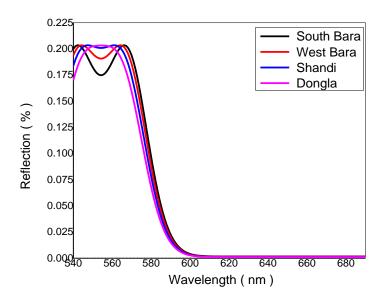


Figure (3.7): The reflectance spectra of four samples of SiO_2

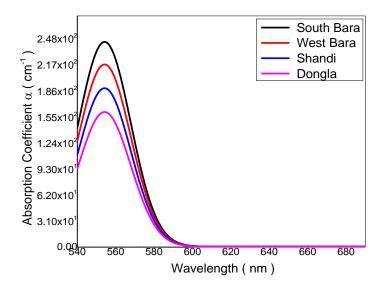


Figure (3.8): The absorption coefficient of four samples of ${\rm SiO_2}$

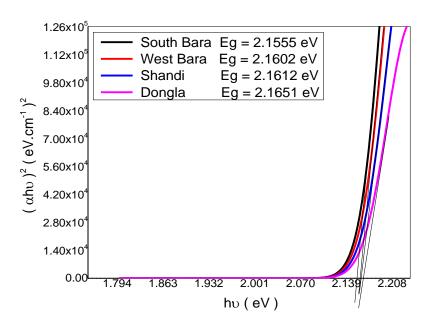


Figure (3.9): The energy band gap of each sample of SiO_2

CHAPTER FOUR

DISCUSSION AND CONCLUSION

4.1 Fourier Transform Infrared Spectroscopy Analysis

The IR data was collected and the range of functional groups peaks from 4000 cm⁻¹ - 400 cm⁻¹ with 4cm⁻¹ resolution and 8 scans at room temperature, the spectra and spectral data of obtained SiO₂ nanopowder are presented in Fig (3.1), Fig (3.2), Fig (3.3), Fig (3.4), with corresponding tables to each Figure. The IR band in range 3000 – 4000 cm⁻¹ is assigned to the O-H stretching vibrations of water molecule H₂O in the sample, the weak absorption band at 1631 cm⁻¹ and 1635 cm⁻¹ corresponding to the adsorbed H₂O molecules deformation vibrations, the IR band at 1452 cm⁻¹, 1457 cm⁻¹ and 1460 cm⁻¹ can be referred to the presence of symmetric and asymmetric bending vibrations of C-H bond of -CH2-CH2 (alkene), the very strong and high absorbance peaks at 1079.22 cm⁻¹, 1094 cm⁻¹, 1097 cm⁻¹ and 1122.87 cm⁻¹ are assigned to Si-O-Si asymmetric stretching vibrations which the bridging oxygen atom moves parallel to the Si-Si lines in the opposite direction to their Si neighbors, the band at 790 cm⁻¹- 798 cm⁻¹ is due to symmetric stretching vibrations of Si-O-Si where the oxygen atom move at right angle to the Si-Si lines in the Si-O-Si plane, whereas IR band at 620 cm⁻¹ indicates to Si-O stretching vibrations of SiO₂, the band in a wavenumber at 465 cm⁻¹ and 471 cm⁻¹ can be referred to Si-O bending vibrations where the oxygen atom moves perpendicular to the Si-O-Si plane.

4.2 Ultraviolet spectroscopy Analysis

UV-visible spectrometer was conducted to study the optical properties of each obtained SiO₂ nanoparticles in terms of absorbance, transmittance, reflectance, absorption coefficient versus wavelength, and the energy band gap. The UV spectra of absorption show that the obtained SiO₂ sample 4 synthesized from silica sands taken from south Bara has highest absorbance peak at around 0.536 a.u, whereas the lowest absorbance peak recorded for SiO₂ from Dongla sample 2 at 0.201a.u. The UV transmission spectra indicate that SiO₂ sample 2 has highest transmittance with peak at around 0.5%, and SiO₂ sample 4 has lowest peak at about 0.3%. The UV spectra of reflection reveal that reflectance peak of SiO₂ sample 2 occur at one wave length value at 0.200% approximately, whereas reflectance peaks of other samples occur at two different wavelength values. As the SiO₂ sample 4 has highest absorbance, so it is natural that the SiO₂ sample 4 will have highest absorption coefficient at 248cm⁻¹, and SiO₂ sample 2 has the lowest absorption coefficient at 155cm⁻¹. The UV spectra of energy band gap show explicitly that obtained SiO₂ sample 4 has the smallest band gap 2.1555 eV, and SiO_2 sample 2 has the largest energy band gap 2.1651 eV.

From overall optical data resulted from FT-IR and UV-visible spectrometry we can deduce that SiO₂ nanoparticles synthesized from silica sands taken from different areas the optical properties of each sample depend on the location of area that the silica sand was taken from, we found that SiO₂ sample 2 obtained from silica sand from Dongla which is located in northern Sudan reported the largest energy band gap, whereas SiO₂ sample 4 obtained from silica sand taken from southern Bara located in central Sudan reported the smallest energy band gap. We noticed that, the energy band gap of SiO₂ nanoparticles decreases as we head towards south Sudan, First Dongla, next Shandi, next western Bara and then south Bara, the band gap of each sample 2.1651 eV, 2.1612 eV, 2.1602 eV,

2.1555 eV respectively. The reason for this might be, the silica sand grains in north Sudan are cleaner and have fewer impurities with six functional groups as were reported by FT-IR, when we move from north towards south the silica sands start to contain more functional groups peaks which can affect a little bit the optical properties of synthesized SiO_2 nanoparticles.

4.3 Conclusion

The SiO_2 nanoparticles have successfully been synthesized from natural silica sands employed from four different areas across Sudan by means of alkali-fusion method (dry method). Having been used this method we were able to recognize the difference in optical properties between the four samples and this is due to presence of additional functional groups in the samples as we go towards South areas.

4.4 Recommendations

- Nanotechnology nowadays is shaping the modern world and soon will dominate every aspect of future technology; therefore we recommend that nanomaterial's synthesis and characterization should be of interest of material researchers due to excellent properties of materials at this small scale.
- ➤ People who do research on silica nanoparticles should use two, three or four synthesis methods for obtaining more accurate results of differences in optical properties of silica nanoparticles synthesized from natural sands when employed from different geographical locations.

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