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CHEMISTRY DEPARTMENT

SYNTHESIS OF SILICA NANOPARTICLES FROM CHRYSOTILE TO STRENGTHEN CONCERETE

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APPROVAL FORM

The signatories named below have proved that they have taken part in the supervision, and reading of the dissertation and would like to praise the dissertation for its approval by the Bindura University of Science Education. The research project is titled:

PREPARATION OF NANO SILICA FROM ASBESTOS

Submitted by TINEVIMBO C MAZIRIRI

Signature of student: **Date: 21/06/22**

Signature of Supervisor: **Date:02/10/23**

Signature of Chairperson: **Date: 02/10/23**

DECLARATION FORM

I MAZIRIRI TINEVIMBO would hereby declare to the Bindura University of Science Education that the information in this research project is my unique work and all the materials and sources of data used were acknowledged as expected. This information has not been published or submitted for any educational purposes to fill any institutional requirements.

Signature of Student: **Date: 21/06/22**

ACKNOWLEDGEMENT

First, I would like to give all my glory to the Almighty Lord who has been with me from day one up until the last day I submitted this dissertation. I am deeply grateful to my supervisor, Prof M Mupa, for his unending support by guiding me and making corrections on the way. I pray that the Lord God will continue to give him the spirit of not giving up on his students as he did for me and may he bless him. In addition, I would like to honour the work of the entire chemistry department and colleagues who helped with the ideas and information until the goals of the dissertation were achieved.

DEDICATION

This work is dedicated to my father, my mother, and my supervisor Professor Mupa.

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ABSTRACT

Nano fibrous silica with a high surface area was produced from chrysotile by the acid leaching method using Hydrochloric acid, Sulphuric acid and Phosphoric acid. Natural mineral chrysotile asbestos from Zvishavane was used as the starting material. The fibers were modified by chemical treatment with 2M HCl and the mineral dissolution was monitored using X ray powder diffraction and a FTIR to highlight the effects of the leaching process. The results showed that the applied concentration of the acid solution of 2M and processing time of 4hours were sufficient to effectively remove the magnesium hydroxide later and transform chrysotile to porous silicon dioxide. The main purpose of the production of silica nanoparticles was to strengthen concrete. Silica Nps are used in concrete due to its finer particles and pozzanic behaviour are valuable in high performance of concretes. Nano silica improves the bonding between the cement mixtures and easily aggregates, therefore a concrete with high flexural strength and compressive strength can be produced using nano-silica. Silica fume is used to produce a highly enhanced concrete with high early strength and compressive strength can be produced using nano silica.

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ABBREVIATIONS AND ACRONYMS

FTIR- Fourier Transform Infrared

XRD- X-ray Diffraction

EDS-Energy Dispersive X-ray Spectroscopy

Nps- Nano particles

HCl- hydrochloric acid

H_2PO_4- Phosphoric acid

SEM- Scanning electron microscope

TEM- Transmission electron microscope

XRF- X-ray Fluorescent

CHAPTER 1

1.0INTRODUCTION

1.1BACKGROUND

Fine and coarse aggregate are combined to form concrete, a composite material, which is then joined by a fluid cement (cement paste) that eventually solidifies (cure).Concrete is extensively used in the construction of various structures such as Building, Bridges, Dams and roads .Poor concrete affects the long term durability, strength and lifespan of buildings. Low Strength concrete also leads to development of cracks on civil engineering structures. Polycarboxylate superplasticizers (PCE) have been used in the past to improve the cracking resistance of concrete. Temperature rise inhibitors (Cellulose and Starch) have also been used as water retention agents, thus modifying cement hydration.Nanotechnology is also making inroads in the strengthening of concrete especially at earlier stages. Nano concrete has a number of advantages such as reduced costs, higher strength, good workability, saving of cement and also enhancement of the hydration process.

Because it can be easily woven, is resistant to temperature and corrosive chemicals, has a high tensile strength, and is robust, chrysotile asbestos is a naturally occurring material that is easy to mine. In Zimbabwe Chrysotile is largely mined in Zvishavane. The idealized chemical formula of chrysotile is $Mg_3(Si_2O_5)(OH)_4$, although some of the magnesium ions may be replaced by iron or other cations. Chrysotile is very rich in Silicon dioxide and this study is going to show the synthesis of silica nanoparticles using the acid leaching method from a low cost and easy to mine mineral.

It has been discovered that silica nanoparticles considerably increase the strength of concrete when added. By promoting the hydration reaction and filling the micropores in the structure of the cement paste, silica nanoparticles increase the strength and durability of concrete. As a result, the concrete's porosity is reduced, enhancing the cement mortar's strength and mechanical qualities. According to the calorimetric results, the inclusion of SNPs hastened the hydration process, and the inactive time was cut by 4 h when 2% SNPs were added compared to the control. The impact of adding SNPs in the right amounts on concrete's mechanical and durability qualities was examined at a 0.25 w/b ratio. Comparing the findings of the mixtures with SNPs added to them to the control indicated an improvement in

compressive strength of 61% after 3 days and 25% after 28 days. The durability experiments at 28 days revealed that the porosity, sorptivity, and water absorption decreased by up to 25–40% and the density of the interfacial transition zone with the integration of SNPs.

1.2 PROBLEM STATEMENT

Low Strength concrete has been a major problem especially in developing countries. Concrete is known to have low tensile strength compared to other binding materials, due to this it has to be reinforced to avoid cracks .Shrinkage forces can become greater than the strength of the concrete which leads to development of cracks in buildings and bridges. This results in low durability of various buildings, dams and bridges. This study will focus on strengthening concrete using a small percentage of silica nanoparticles synthesized from a cheap and easy to mine mineral.

1.3 AIM

- To develop a synthetic procedure for silica from asbestos

1.4 OBJECTIVES

- To determine the effect of acid on the yield of silica
- To determine the effect of reaction time on the yield of silica
- To determine absorption properties of silica
- To determine the bulk density of silica

1.5 JUSTIFICATION

Because it can be easily woven, chrysotile asbestos has various industrial uses and is both naturally abundant and simple to mine. It is durable and has high tensile strength

Chrysotile is very rich in silicon dioxide and the nano fibriform silica is composed of hydrous silicon dioxide with a minor quantity of magnesium trapped inside the SiO network when the magnesium leaching degree is over 90%. A useful product is generated from a cheap and easy to mine mineral which leads to strengthening of concrete and most civil engineering structures.

In order to create high performance concretes, silica NPs can be employed in concrete due to their tiny particles and pozzoulanic activity (J. H. Brouwers, 2010). In order to create concrete with improved qualities, primarily high early strength or low penetrability concretes, silica fume is utilized. A concrete with high flexural strength and compressive strength can be

made with nano-silica because it boosts density and strengthens the link between cement and easily-mixed particles.

1.6 LIMITATIONS

The absence of machinery for the characterization for example SEM, TEM, XRF and FTIR. Moreover, the absence of certain chemicals and the contamination of some chemicals will later on give rise to wrong results.

1.7 THE STUDY VALIDALITY

This study, judging from the mentioned possible advantages, is not purely an academic study. It is research that could have a positive impact on, most importantly, civil engineering companies and Human exposure.

CHAPTER 2
LITERATURE REVIEW

2.1 INTRODUCTION

The most prevalent type of asbestos is chrysotile, sometimes known as white asbestos, which makes up around 95% of the asbestos in the United States and a comparable amount worldwide. It belongs to the serpentine subgroup of phyllosilicates and is a soft, fibrous silicate mineral, making it different from other asbestos form of minerals in the amphibole group. $Mg_3(Si_2O_5)(OH)_4$ is the idealized chemical formula for it. The substance possesses physical qualities that make it suitable for use in building materials, but when released into the air and ingested, it causes major health hazards.

Fig 2.1 Image of Chyrsotile

Chrysotile is easily crushed into fibrous strands made up of smaller bundles of fibrils despite having a hardness comparable to that of a human fingernail in its bulk form. Natural fiber bundles can range in length from a few millimeters to more than ten centimeters, however chrysotile that has undergone industrial processing often has shorter fiber bundles. Each fiber bundle contains tens or hundreds of individual fibrils, each of which is even smaller than the fiber bundles themselves, measuring 0.02-0.03 m in diameter. Chrysotile fibers may be spun into thread and sewn into fabric and have a high tensile strength. Additionally heat-resistant, they make superior thermal, electrical, and acoustic insulators. Despite having a hardness similar to that of a human fingernail in its bulk state, chrysotile is easily crushed into fibrous threads made up of smaller bundles of fibrils. The length of natural fiber bundles can range from a few millimeters to more than 10 centimeters, while industrially processed chrysotile frequently has shorter fiber bundles. Tens or hundreds of tiny fibrils, each smaller than the

fiber bundles themselves and measuring 0.02-0.03 m in diameter, make up each fiber bundle. Chrysotile fibers have a high tensile strength and can be spun into thread and sewed into cloth. They are also heat-resistant and make excellent electrical, acoustic, and thermal insulators. (From Wikipedia, the free encyclopedia)

2.2 SILICON DIOXIDE

SiO_2 is also called silica. It is natural component made of two of earth's most abundant materials which are silica and oxygen.SiO_2 is mostly found as quartz. The earth's crust is 59% silica. Silica is a non-metal with a giant molecular structure bonded with covalent bonds. It has four O_2 atoms connected to silicon atom and 2 oxygen atoms bonded to each silicon atom in a tetrahedral structure. Silica has outstanding properties which has made it useful and major component in glass which are high thermal stability, good abrasion resistance, insoluble in many acids excluding HF and a good electrical insulator. Due to the high quantities of silicon dioxide in glass, this made glass a good raw material for the synthesis of nano silica particles. The giant molecular structure of SiO_2 gives its high melting point (about 1700^0C).Due to this high melting point some substances are added to lower its melting point. The substances added to the silicon dioxide to lower its melting point are called flexes (for example Lithia, potash and soda), the melting point will decrease to a lower range of 800^0C to 900^0C. Moreover, Silica is also used in building materials, as a refractory since it can be put in furnace lining and as a precursor to the fabrication of other ceramics. (Land,2012)

2.3 SILICA NANOPARTICLES

Silica nanoparticles are regarded as mesopores of silica that have got unique physiochemical properties. These nano carriers can also be prepared in a variety of sizes and shapes including nanotubes, nano ribbons, Nano helices and nano tubes. Silica nanoparticles got a narrow distribution for particle size, small particle size, permeable and large surface area.

Nano silica are not toxic (low toxicity), this make it function with many polymers and a large range of molecules. . (R Yuvakkumar, 2014).It is highly reflective to ultra violet rays visible light and long wave

2.4 OTHER PROCESSES TO SYNTHESISE SILICA NANOPARTICLES

According to (Ara Begum, Hossain, Islam, & Azi, 2018), rice husk ash dissolves in alkaline solution. Due to its solubility has led to its use in the extraction of silica. The extraction of silica according to their studies was done by mixing 10% rice husk ash with 30% water and by heating, 5% of sodium hydroxide was added to this mixture. In addition, it was diluted

with 55% of water, the mixture was stirred. For a period of 24hrs the diluted solution was left to settle under room temperature. The diluted solution was left to settle.

Silica Nps are also synthesized by olivine dissolution, in which the olivine is dissolved in acids and olivine mineral is used to neutralise that acid (A.Lazzaro , 2010).The neutralization process produces silica, unreacted olivine, Mg or Fe salt solutions, and inert minerals. After the reaction is finished, sedimentation removes the inert minerals and unreacted olivine from the final suspension. Washing and sifting the mixture will remove any silica. After filtration, a cake with a 20% solid nano-silica content is produced.
The sol-gel process it's also another experiment used to synthesize silica nano particles. It involves the hydrolysis of metal alkoxides and also condensation of metal alkoxides such as TEOS or organic salts such as Na_2SiO_3 in the presence of HCl or base as a catalyst. There is formation of Silanol groups from the hydrolysis of TEOS, condensation between the silanol groups and ethoxy groups' forms' siloxxane bridges that form silicate a silica structure (Y. Ye et al , 2010).

Reverse micro emulsion is also used to synthesise silica nanoparticles. A surfactant molecule is dissolved in organic solvents which leads to formation of spherical micelles and in the presence of H_2O the polar head groups form micro cavities containing water (reverse micelles). The reverse micro elmusion produces spherical silica nps with diameters of tens to a few hundred nanometers. In production of silica nps, the nanoparticles can be grown inside the microcavties by c controlling addition of silicon alkoxides and catalyst into the medium with the reverse micelles. This process has got a disadvantage as it is expensive and it is difficult to remove the surfactants in the final products.

The flame synthesis can also be used to synthesise silica nanoparticles. This is the most economical way of producing silica nano particles in powder form. This method make use of high temperatures. Silicon dioxide Nps can be produced also through the use of high temperature flame decomposition of metal organic precursors, a process called chemical vapour condensation (M. Monshizadeh et al, 2010).In this method, the silica nps are produced by reacting $SiCl_4$ with H_2 and O_2 (Garret , 2007).Flame synthesis has got demerits of controlling the nature of the product that is the phase composition, particle size and morphology. The sol gel method this another method widely used for the synthesis of silica

nano particles. This method involves hydrolysis and condensation of metal alkoxides such as TEOS or organic salts such as sodium silicate in the presence of an acid such as HCl or base as a catalyst. Silanol groups are formed from the hydrolysis of TEOS, condensation between the ethoxy groups and silanol groups creates siloxxane bridges that form a silica structure (Y. Ye et al ,2010).

2.5 PROPERTIES OF SILICA NANOPARTICLES

2.5.1 THERMAL AND MECHANICAL PROPERTIES

The powdered nano particles can easily compact, the cindering temperatures also reduce to that of the conventional powders in the micro range. This is so due to the fact that surface area of nano particles provides higher particle contacts than conventional particles and molecules. Ceramic nps exhibit a reduced brittleness and more enhanced ductility making ceramics nanoparticles more reliable.

2.5.2 OPTICAL PROCESSES

The theoretical optical properties of silica have been characterized using the photoluminescence (PL) spectroscopy. The optical properties of silica nps can be enhanced through the incorporation with metal ions or other functionalized groups to give optical devices that are unique. Point defects may be classified into two; paramagnetic and diamagnetic. . The paramagnetic defects optical absorption which shows half-occupied energy level in the optical band gap, whole or electron transition to the valence band gap is possible. Diamagnetic defects possess an absorption band associated with whole transition to the conduction band, and their combination exhibits a diversity in absorption and photoluminescence (PL) bands in a wide range of wavelength, UV, and visible range. It was discovered by Chen that a unique blue shift in the blue band of the PL spectra while studying the PL behaviour of 7nm and 15nm silica NPs. The green bands in the Photoluminescence spectra were attributed to those species that are hydrogen-related and no bridging oxygen in the silica NPs.

2.5.3 PHYSIOCHEMICAL PROPERTIES

The silica Nps are chemically reactive and they play a major role in catalysis as they got adsorption properties. They got an ability that they can chemically adsorb and dissociate a wide range of hazardous organic molecules. Particle size of silica Nps is altered by the concentration of the silanol group, as the concentration of the silanol group increases the particle size of the nanoparticle decreases. There is experienced increase of the amount of the atoms that are residing on the surface with a decrease in particle size.

2.6 APPLICATIONS OF SILICA Nps

Silica nano particles are for many marital purposes to humanity, this is due to their stability to heat high abrasion resistance, mechanical strength, and reduced shrinkage. The silica Nps have got a high surface to volume ratio due to particle size and they exhibit good optical transparency

2.6.1 Use in Concretes:

Silica Nps are used in concrete due to its finer particles and pozzanic behaviour are valuable in high performance of concretes.(J. H Brouwers , 2010).Nano silica improves the bonding between the cement mixture and easily aggregates ,therefore a concrete with high flexural strength and compressive strength can be produced using nano-silica. Silica fume is used to produce a highly enhanced concrete with high early strength and compressive strength can be produced using nano silica.

2.6.2 Used in car and tyre manufacture:

A car and tyre company called TATA INDUSTRIES is employing the use of nano silica in its tyre manufacturing process.

The function and benefits of the nano silica in the manufacture:

Benefits	Function	Customer application
Nano sized particle	Reinforcement	Reinforcing filler
High surface area	Filler Abrasion resistant	Construction industry
Mesopores	Hydrophobic Hydrophilic	High temperatures, insulators and industrial absorbents
Functionalized	Other functions	New applications

Table 2.1: The function and benefits of the nano silica in the manufacture:

TYRES:

The use of functional silica in tyre enhances the efficiency significantly:

- Reinforcing filler: It acts as a reinforcing filler in the tyre tread which reduces rolling resistance and friction .It reduces wear and tear thereby, improving durability
- Friction reduction: Reduces the friction between rubber molecules in the tyre this leads to less energy loss through the tread, thereby delivering better fuel consumption
- Increases hardness: adding silica to rubber increases it hardness and makes it less susceptible to deformation thereby decreasing its rolling resistance
- Enhanced wet handling: delivers better wet and snow traction, improved rolling resistance(TATA Chemical limited)

Tyre compounding studies done with silica sample show better characteristics in physical, dynamic stiffness leading to improved hardness, tensile strength, tear strength and elongation (TATA, 2016)

2.6.3 POLYMER NANOCOMPOSITES

The nanoparticles are used in making Nano composites which enables them to play a role in aerospace, automotive, engineering and electronic sectors. Silica Nps reinforcement in polymer matrixes can lead to property improvement. The nano composites of silica polymers got the ability to attain homogeneous filler dispersion which determines its overall performance (L. Rahman et al , 2012)

2.6.4 Other Applications of the NPs:

- Basis for biomedical research as it is stable(L. Tang, 2013)
- As abrasives in the electronic industry
- As a strengthening filler for concrete and other construction composites
- Can be widely used in ceramics porcelain
- Used in plastics manufacture

2.7 CHARACTERISATION METHODS:

2.7.1 Scanning Electron Microscopy

According to (Kannan, 2018), A scanning electron microscope functions similarly to an optical microscope, except instead of using light to "image" the sample and learn more about its structure and makeup, it instead employs a concentrated electron beam. It was further argued that the development of the electron microscope was undertaken to address the issues related to the use of light microscopy. The principles of electron microscope indicate that it has much higher magnification and resolution compared to the light microscope and thus is much reliable. The higher magnification and resolution help in the study of the smallest objects. (Berhanu, 2018), suggested that the resolution depends on the dimensions of the electron spot, the electrons' wavelength and the dimensions of the interaction volume. The electron beam is formed from an electron source and is accelerated on the sample using a positive potential. The electron beam is pinched and focused into a thin, monochromatic beam that is pinched and focused using a metal shutter and magnetic lens. The electrons in the beam interact with the atoms in the sample to generate a signal containing information about their surface topography, composition, and other electrical properties. These interactions and effects are captured and converted into images (Kannan, 2018)

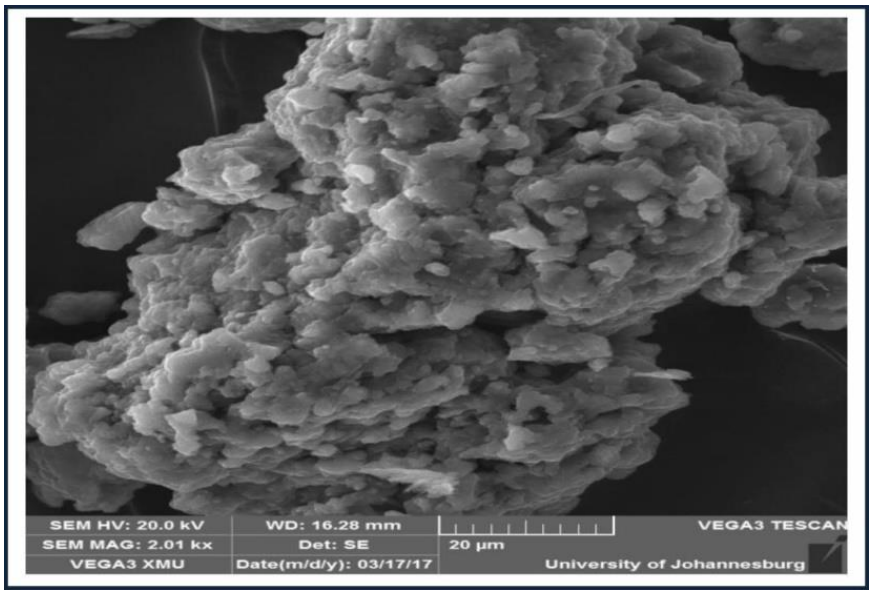

FIG 2.2 SEM image of silica

2.7.2TRANSMISSION ELECTRON MICROSCOPE

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image. The specimen is typically an ultrathin section less than 100 nm thick or a suspension on a grid. An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic paper, or another imaging device

Due to the short de Broglie wavelength of electrons, transmission electron microscopes can image at a substantially better resolution than light microscopes. Since a single column of atoms is thousands of times smaller than a resolvable object observed in a light microscope, this allows the equipment to capture extremely fine detail. In the physical, chemical, and biological sciences, transmission electron microscopy is a crucial analytical technique. TEMs are useful in the study of cancer, viruses, and materials, as well as in the study of pollution, nanotechnology, and semiconductors, as well as in other disciplines including palaeontology and palynology.

2.7.3 XRAY DEFRACTOMETER

An XRD instrument is used in the characterisation of crystalline materials and the study of space between atoms (atomic spacing), this is done by the use of x-ray beams focused on the crystalline matter and in turn generates a diffraction pattern. The XRD determines the atomic size, atomic scale difference and chemical bonds present in the material. The percentage composition of elements present in analysed using EDX (Energy Dispersive X-ray Spectroscopy) for silica NPs it should show only the peaks of silicon and oxygen (J. Chrusciel et al, 2003)

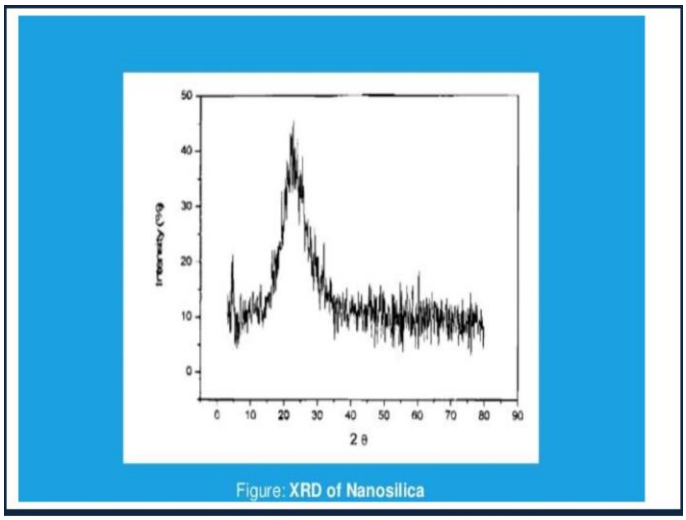

Fig 2.3 XRD PATTERN OF SILICA

2.7.4 Fourier Transform Infrared Spectroscopy (FTIR)

It works by creating an infrared absorption spectrum, which confirms the purity and type of surface functionalities present on the surface of the silica nanoparticles. FTIR spectra are used to identify chemical bonds in molecules. A solid, liquid, or gas's emission or absorption of infrared light is obtained by the FTIR. The raw material data are transformed into actual spectrum using the FTIR.

2.7.5 EDX (Energy Dispersive X-ray Spectosctropy)

Energy dispersive X-ray spectroscopy (EDS, EDX, XEDS or EDXS), sometimes called Energy dispersive X-ray microanalysis (EDXMA), energy dispersive X-ray analysis (EDXA). It’s an analytical technique used for the elemental analysis or chemical characterization of a sample. Its characterization is due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.

Proposed EDX sample results

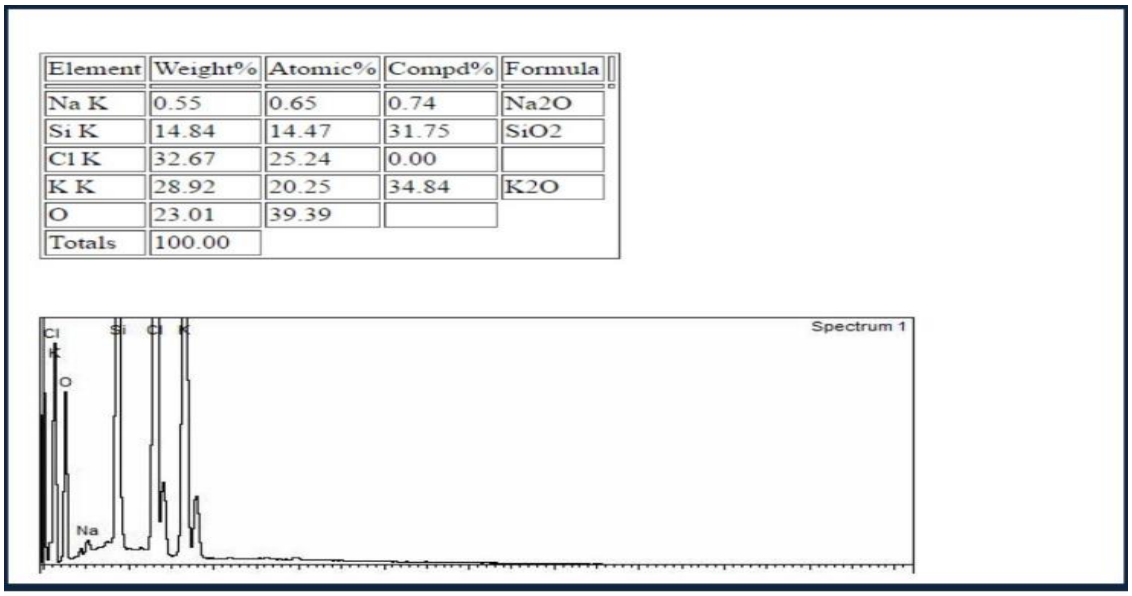

Fig 2.4 EDX PATTERN FOR SILICA

2.7.6POWDER XRAY DIFFRACTION

An X-ray diffracts in a manner specific to the structure when it is shone on a crystal. Instead of using a single crystal, the diffraction pattern in powder X-ray diffraction is derived from the material's powder. Since powder diffraction does not call for the creation of individual crystals, it is frequently simpler and more practical than single crystal diffraction. Instead than obtaining a diffraction pattern from a single crystal, which may not accurately reflect the entire material

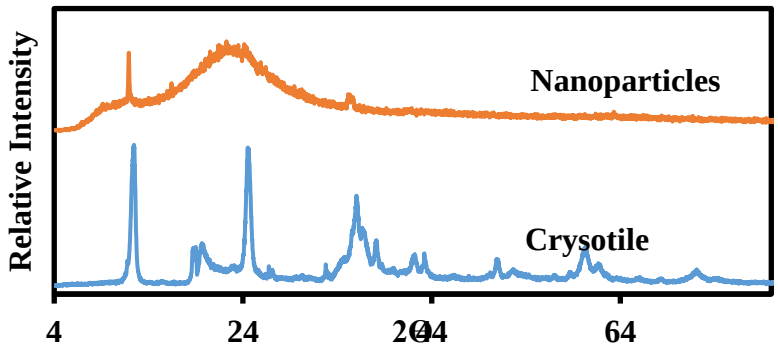

Fig 2.5 PXRD Pattern for silica and chrysotile

BRAGG,S LAW

When X-rays hit a crystal's surface, atoms partially scatter the light. The X-ray that is not scattered passes through to the layer of atoms below, where it is again partially scattered and partially continues on to the next layer. Similar to how a grating diffracts a beam of light, this results in an overall diffraction pattern. The sample must be crystalline and the distance between atom layers must be close to the radiation wavelength for an X-ray to diffract.

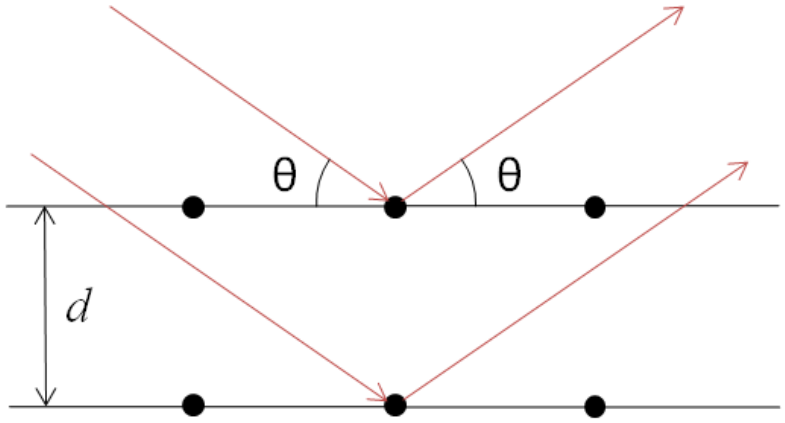

Fig 2.6 Xray diffraction

The diffraction pattern exhibits a peak when beams diffracted by two separate layers are in phase; however, when they are out of phase, destructive interference appears and there is no peak. Diffraction peaks only occur if

$$\sin\theta = n\lambda \backslash 2d$$

where

- θ is the angle of incidence of the X-ray,
- n is an integer,
- λ is the wavelength, and
- d is the spacing between atom layers.

Only crystalline solids will diffract; amorphous materials will not appear in a diffraction pattern because diffraction requires a highly regular structure.

XRF method

According to (Khalid, Khan, Alam, & Anwar, 2015), the emission of distinctive or secondary X-rays from a material that has been excited by high-energy electrons or other X-ray or -ray

photons is known as X-ray fluorescence. If the incident particle is powerful enough, it can knock an orbital electron out of the target atom's inner shell. To fill the void, one of the electrons from the higher shells jumps to the inner shell, producing a photon with energy equal to the difference between the two shells' binding energies. The emission spectrum of X-rays at discrete energy is produced by X-ray fluorescence. These emission spectral lines are referred to be characteristic or fluorescence X-rays since they are dependent on the target element (Khalid, Khan, Alam, & Anwar, 2015) . By comparing the peak's energy to the element's binding energy, we can utilize these spectra to identify the elements.

CHAPTER 3
RESEARCH METHODOLOGY

3.1 INTRODUCTION

This chapter focuses on the methods that were taken to achieve the set objectives. The methods followed includes, preparation of the samples, synthesis of silica Nps from *chrysotile* band finally the methods of characterisation of silica Nps.

3.2 MATERIALS AND REAGENTS

Asbestos (Chrysotile) was collected from Zvishavane, Midlands province. . SkyLabs in South Africa and Bindura University Laboratory in Zimbabwe provided the Hydrochloric acid and distilled water. FTIR and XRD were used to characterize the silica nanoparticles.

3.3 REAGENTS AND APPARATUS

- **C**hrysotile
- Distilled water
- Hydrochloric acid
- Nitric Acid
- Phosphoric acid
- Stove
- Condenser
- Oven
- Filter papers
- Erlenmeyer flask
- Analytical Balance
- FTIR
- XRD

3.4 EXPERIMENT PROCEDURE

1. Weigh 10grams of Chrysotile into an Erlenmeyer flask

2. Add 200ml of 2M HCl to the Chrysotile

3. Connect a condenser to the Erlenmeyer flask and reflux for 4hours with running distilled water through the pipe

4. After 4hours filter the mixture to remove HCl

5. Wash the product with distilled water

6. Filter again to remove excess water

7. After washing place the product into an oven at 100degrees celcius for 24hours

9. Repeat the experiment using Nitric Acid and Phosphoric acid to determine the best acid for the experiment

REFLUX APPARATUS

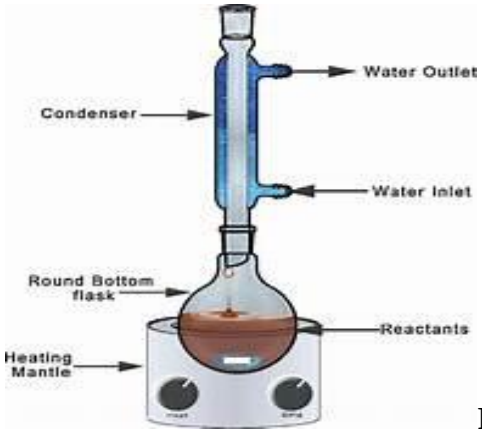

FIG 3.1 Reflux apparatus

3.5 SUMMARY OF THE PRODUCTION OF SILICA NANOPARTICLES

FIG 3.2 Summary for production of silica

3.7 Characterisation Methods

3.7.1POWDER XRAY DIFFRACTION

An X-ray diffracts in a manner specific to the structure when it is shone on a crystal. Instead of using a single crystal, the diffraction pattern in powder X-ray diffraction is derived from the material's powder. Since powder diffraction does not call for the creation of individual crystals, it is frequently simpler and more practical than single crystal diffraction. Instead than obtaining a diffraction pattern from a single crystal, which may not accurately reflect the entire material, powder X-ray diffraction (XRD) also obtains a diffraction pattern for the bulk material of a crystalline solid. Intensity is shown against the detector's angle in a diffraction pattern

Fig 3.3 PXRD instrumentation

3.7.2Fourier Transform Infrared Spectroscopy

The ability possessed by FTIR to detect functional groups present in a sample has made FTIR a method for characterizing different samples to validate presence of required compound. The type of the FTIR used is the German Nicorette Thermo Fisher FTIR. Structural determination of the elemental silica was carried out, this was done by crushing the silica granules into a powder. Ethanol was used to clean the sensor at the FTIR. A small amount of powder was placed on the iD7ATR Diamond for analysis. The machine was manipulated accordingly and the spectrum was generated as expected.

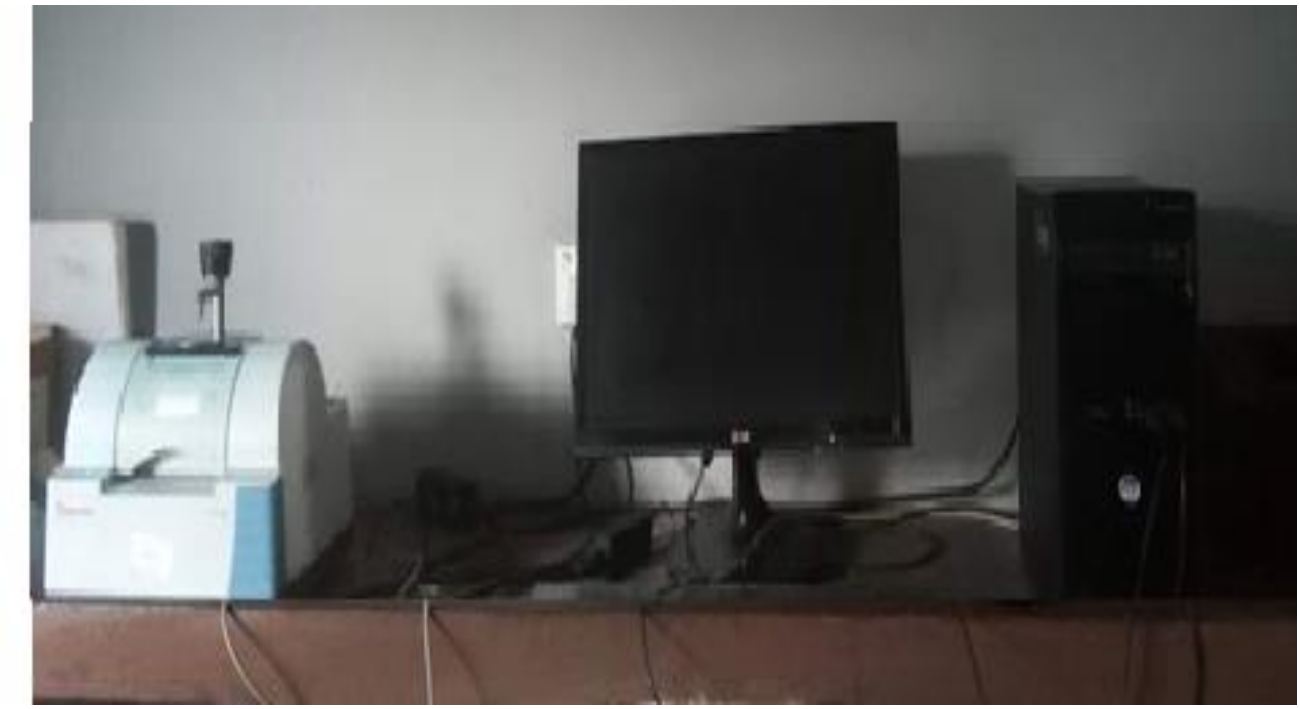

Figure 3.5: Fourier Transform Infrared Spectroscopy

3.7.3 Determination of Bulk Density

The bulk density of the silica particles was measured using the principles stated by (Lin, Wang, & Selomulya, 2022) and (Smithers, 2022). 5g of sample was measured and 12.5 mls of water was used. The bulk density was calculated using the following equation: $\rho = \frac{m}{V_0}$

where M =mass in grams, V_0 =untapped apparent volume in millilitres and ρ= bulk density

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

This chapter's sole aim is to focus on the results produced during the experiments done to produce the required product. The results produced will be presented in different ways (tables, diagrams etc.) in a step fashioned way as the way the experiment progressed.

4.2CHARACTERIZATION OF NANO SILICA

The FTIR (Fourier Transform Infrared Spectroscopy) spectra are used to identify chemical bonds in a molecule through producing an infrared absorption spectrum, confirm the purity and nature of surface functionalities present on the surface of the silica NPs. It obtains an infrared spectrum absorption or emission of a solid, liquid or gas. Fourier transform is required to convert the raw data into actual spectrum. The percentage composition of elements present is analysed using EDX (Energy Dispersive X-ray Spectroscopy), for silica NPs it should show only the peaks of silicon and oxygen (J. Chruściel et al, 2003).Instruments such as TEM, SEM and PXRD are also used

Table4.2: Table of the effect of acid used

Mass of Chrysotile used	Leaching acid	Leaching temperature	Leaching Time	Mass of Silica Particles Produced/g	Observations
10grams	Hydrochloric acid	100 degrees	4hrs	5	The nano produced was very white and fine. Larger quantities of silica particles produced
10grams	Phosphoric acid	100 Degrees	4hrs	4.6	The nano produced was white and fine. Large quantity of silica particles produced which is less

					than that from hydrochloric acid leached.
10grams	Nitric acid	100 degrees	4hrs	4	The nano produced was white and the quantity of silica produced was larger but less than that from hydrochloric and phosphoric acid leaching.

The type of acid used in the leaching of the samples plays an important role in the production of higher grade. From the table 4.2, it can be seen that the silica particles that were produced from the sample that was HCl leached had an intense white colour compared to that of phosphoric acid and nitric acid, despite the fact that they are all acids, hydrochloric acidic is slightly acidic compared to the other two. The leaching power of Hydrochloric acid is greater than that of the other two because of its smaller structure thus it can penetrate the sample easily without much steric hindrance and removing more impurities. The acid dissociation constant of nitric acid is less than that of phosphoric acid and thus although its particles were white, the whiteness was less than that of HCl and phosphoric acid.

EFFECT OF ACID LEACHING TIME

The effect of different acid leaching times (30 min, 60 min, 90 min, 120 min, 150 min, 180 min, 240 min, and 300 min) on the leaching levels of chrysotile was investigated by using 10grams of Chrysotile and using HCl as it proved to be the best acid during leaching of asbestos. The results are shown in the graph below. With the extension of time, Asbestos kept on reacting with hydrochloric acid, so the leaching level increased with the increase in reaction time. The leaching level of asbestos reached the best when the reaction time was 240 min, and the increase in the leaching level was no longer significant when the acid leaching time was extended.

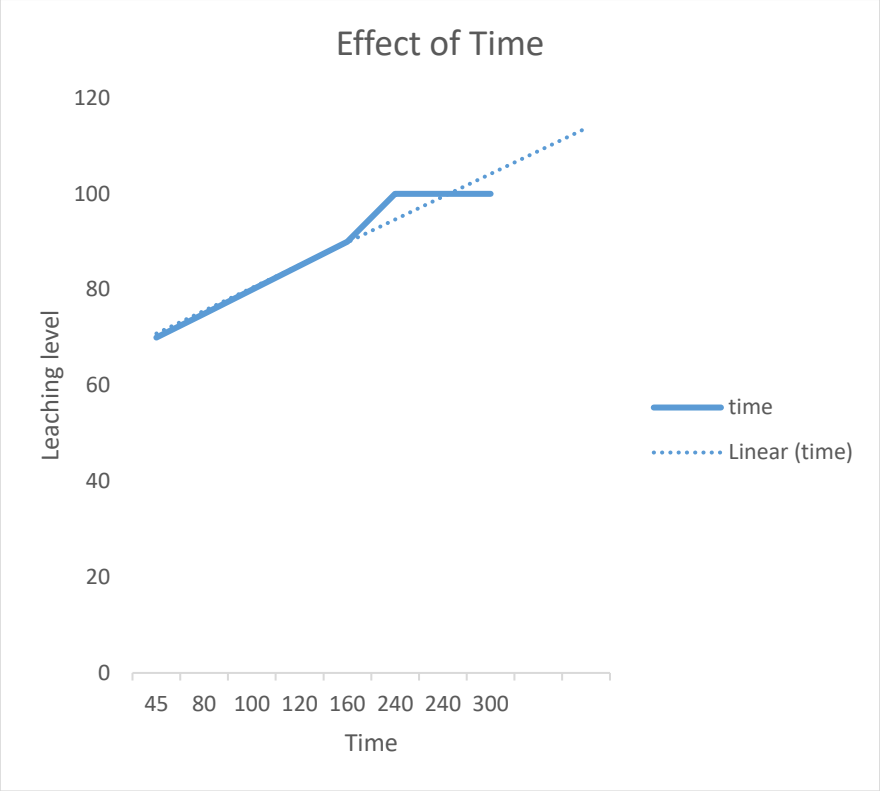

FIG 4.1 Graph for the effect of Time

EFFECT OF ACID LEACHING TEMPERATURE

The reaction time was determined to be 240 min, and the best acid was HCl. The effect of different acid leaching temperatures (45 °C, 55 °C, 65 °C, 75 °C, 85 °C, 100 °C) on the leaching level of asbestos was investigated, and the results are shown in the figure below. At 100 °C, the asbestos leaching level was the best. The higher the temperature, the greater the rate of the chemical reaction.

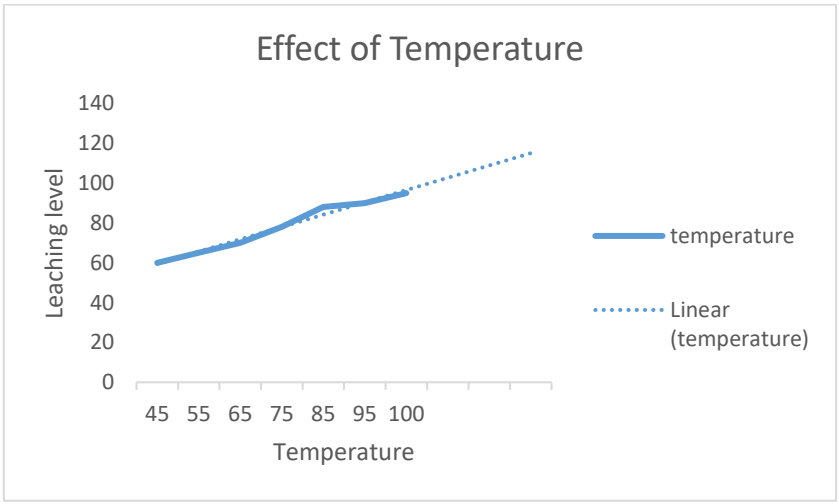

FIG 4.2 Graph for the effect of Temperature

EFFECT OF HCl CONCENTRATION

The acid leaching temperature selected was 100 °C, and the leaching time was 4hours The effect of different hydrochloric acid concentrations (0.5M , 1M , 1.5M , 2M) on the leaching ratios of asbestos were investigated, and the results are shown in the Figure below. As seen in the Figure below , the leaching levels of asbestos increase rapidly with increasing hydrochloric acid concentration, and a continued increase in concentration increases HCl volatilization. When the concentration of hydrochloric acid was 2M , the leaching ratios of asbestos were at best. However, higher hydrochloric acid concentrations at high temperatures tended to cause other volatilization of HCl, resulting in a wastage of hydrochloric acid, so 2M hydrochloric acid was chosen as the suitable concentration for acid leaching.

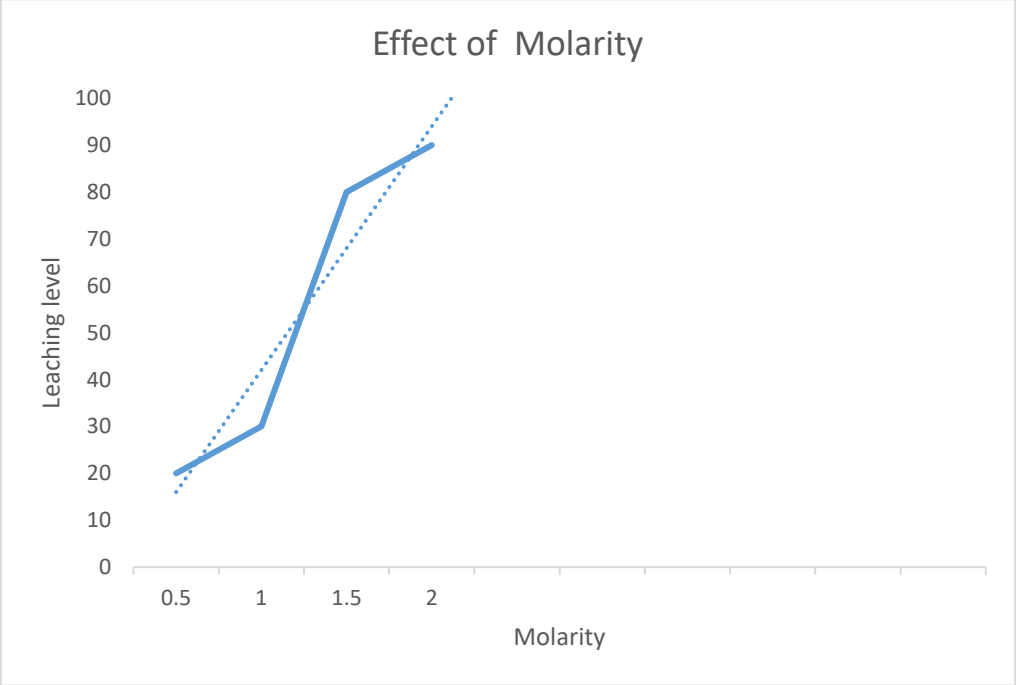

FIG 4.3 Graph for the effect of Molarity

4.3 Moisture absorption Capacity of Silica particles

$$\textbf{Moisture absorption capacity} = \frac{\textit{weight increase after moisture absorption}}{\textit{sample weight}} \times 100$$

There was a general increase in the moisture absorption capacity of the silica particles with time. The general increase decreased with time as the silica particles become saturated with the amount of moisture.

Table 4.2 Moisture absorbed by silica

Days	Initial	1	2	3	5	6	7
Moisture absorbed in g/day	0.00	0.49	0.32	0.28	0.22	0.18	0.18
% moisture gained	0.00	20.3	27.8	42.3	53.2	64.5	66.9

4.4 PXRD ANALYSIS

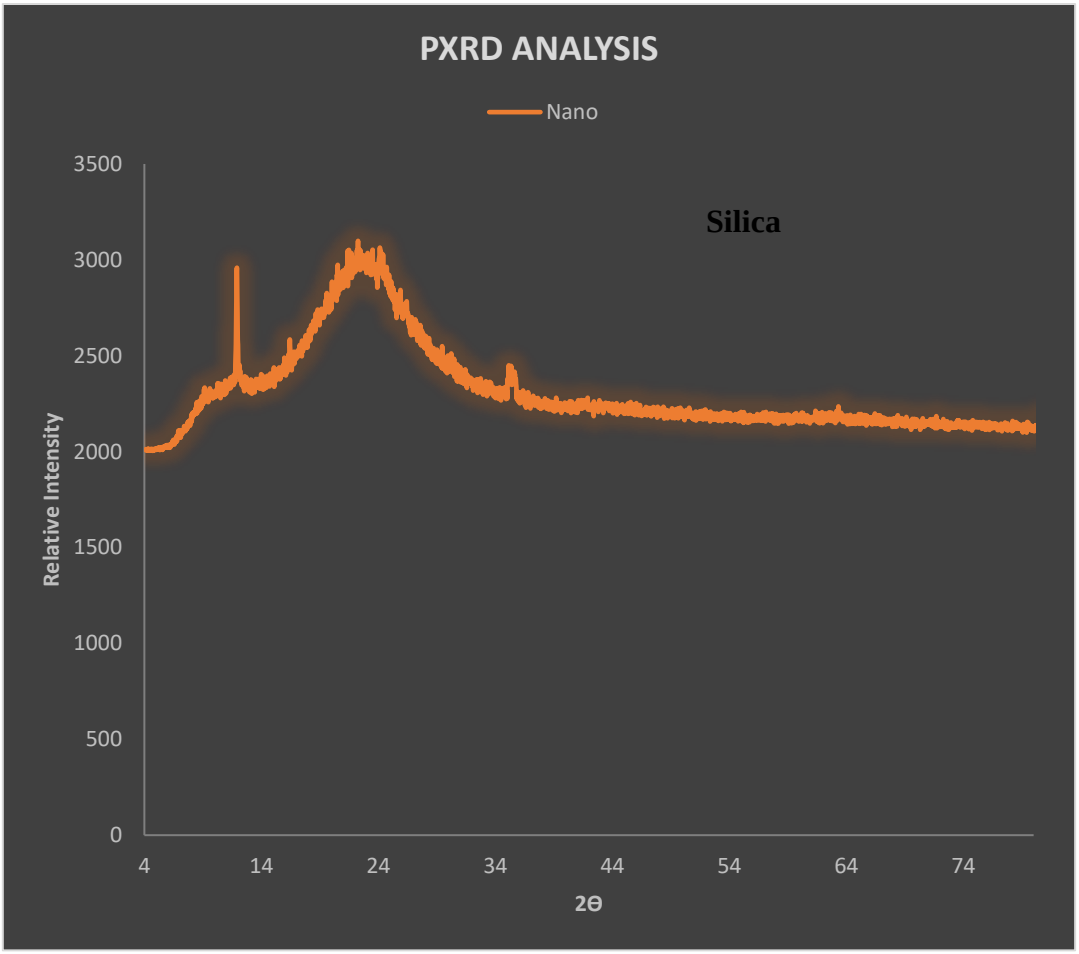

Fig 4.4 PXRD Results

X ray diffractions confirmed the amorphous nature of the prepared silica samples as shown in the diagram above. The broad peak at angle 14-27 reveals the amorphous nature of the silica nanoparticles. The spectrum also showed that there were some crystalline impurities in our silica samples.

CHAPTER 5

CONCLUSION

5.1 RECCOMENDATIONS

There is high need to remove impurities, some impurities were due to the lack of certain reagents and material. There is also need for larger Erlenmeyer flasks and larger heating mantles and also large condensers so as to carry the experiment on a large scale to improve the yield and the purity of the silica nanoparticles. There is also need for necessary equipment such as XRF, TEM, SEM and FTIR so as to characterize the nanoparticles.

5.2 CONCLUSION

The experiment was successful for the production of silica nanoparticles from asbestos and investigations of varying parameters. Nano silica is a useful product in the modern world due to its unique characteristics. Nano silica has many uses both industrially and domestically, as it can be used in cements, plastics and tyre formation. It was observed that HCl was the best acid for leaching chrysotile as it produced a greater yield than the other acids. It was also observed that 4Hours was the best time to produced good results during leaching. Molarity of 2M was the best during the leaching process.

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