

# **PROJECT REPORT**

**On**

**“NANOSILICA PREPARATION BY PRECIPITATION METHOD  
IN PRESENCE OF POLYMERIC DISPERSING AGENT”**

Submitted by

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*In partial fulfillment for the award of the*

**Bachelor's Degree in Chemistry**



**POST GRADUATE AND RESEARCH  
DEPARTMENT OF CHEMISTRY**

**ST. TERESA'S COLLEGE (AUTONOMOUS)  
ERNAKULAM**

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**POST GRADUATE AND RESEARCH  
DEPARTMENT OF CHEMISTRY  
ST. TERESA'S COLLEGE (AUTONOMOUS)  
ERNAKULAM**



**B.Sc. CHEMISTRY PROJECT REPORT**

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Year of Work : 2018-2019**

This is to certify that the project “**NANOSILICA PREPARATION BY PRECIPITATION METHOD IN PRESENCE OF POLYMERIC DISPERSING AGENT**” is the work done by **Ashifa Ashraf, Akhila Mary Stephen, Aiswarya O. S.**

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**Submitted to the Examination of Bachelor's Degree in Chemistry**

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**CERTIFICATE**

This is to certify that the project work entitled **NANOSILICA PREPARATION BY PRECIPITATION METHOD IN PRESENCE OF POLYMERIC DISPERSING AGENT**” is the work done by **Ashifa Ashraf, Akhila Mary Stephen, Aiswarya O. S** under my guidance in the partial fulfilment of the award of the degree of Bachelor of Science in Chemistry at St. Teresa's College (Autonomous), Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

Dr. Saritha Chandran A

Project Guide

## **DECLARATION**

We hereby declare that the project work entitled “**NANOSILICA PREPARATION BY PRECIPITATION METHOD IN PRESENCE OF POLYMERIC DISPERSING AGENT**” submitted to Department of Chemistry, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by us under the guidance of Dr. Saritha Chandran A, Assistant Professor, Department of Chemistry, St. Teresa’s College (Autonomous), Ernakulam and this project work is submitted in the partial fulfillment of the requirements for the award of the degree of Bachelor of Science in Chemistry.

**Ashifa Ashraf**

**Akhila Mary Stephen**

**Aiswarya O. S.**

## *Acknowledgements*

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We take this opportunity to thank our friends and family who gave us confidence to complete this project work successfully.

Above all we thank God Almighty for showering his blessings on us in this endeavor.

*Ashifa Ashraf, Akhila Mary Stephen, Aiswarya O S*

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# Chapter 1

## Introduction

### 1.1 NANOCHEMISTRY

Nanochemistry is a branch of nanoscience, which deals with the chemical applications of nanomaterials in nanotechnology. Nanochemistry involves the study of the synthesis and characterization of materials of nanoscale size. Nanochemistry is a relatively new branch of chemistry concerned with the unique properties associated with assemblies of atoms or molecules of nanoscale (~1-100 nm), so the size of nanoparticles lies somewhere between individual atoms or molecules (the 'building blocks') and larger assemblies of bulk material which we are more familiar with. There are physical and chemical techniques in manipulating atoms to form molecules and nano scale assemblies. Physical techniques allow atoms to be manipulated and positioned to specific requirements for a prescribed use. Traditional chemical techniques arrange atoms in molecules using well characterized chemical reactions. [1]

Nanochemistry is the science of tools, technologies, and methodologies for novel chemical synthesis e.g. employing synthetic chemistry to make nanoscale building blocks of desired (prescribed) shape, size, composition and surface structure and possibly the potential to control the actual self-assembly of these building blocks to various desirable sizes. At this extremely small scale level, quantum effects can be significant, and scientifically very rewarding innovative ways of carrying out chemical reactions are possible. The small size of nanoparticles gives these particles



'unusual' structural and optical properties with applications in catalysis, electro-optical devices etc.

Nanoparticles are very tiny aggregations of atoms, but bigger than most molecules. There is no strict dividing line between nanoparticles and 'ordinary bulk' particles of a material such as baking powder or grains of sand, but particle size matters. Because nanoparticles can display properties significantly different from the bulk material and these properties can be exploited for many different uses. When we compare the size of nanoparticles to that of conventional industrially produced materials we find they have novel uses.

Nanoparticles have a high surface to volume ratio which has a dramatic effect on their properties compared to non-nanoscale, more bulky forms of the same material. Nanoparticles have a very high surface to volume ratio and this gives them special properties different from the bulk material. This difference in surface area / volume ratio for the particles of the material give nanoparticles extra chemical reactivity compared to the bulk material. Less of a material like a catalyst is needed in a chemical process, so catalysts based on nanoparticles are more efficient than those based on bulk material catalysts. [2]

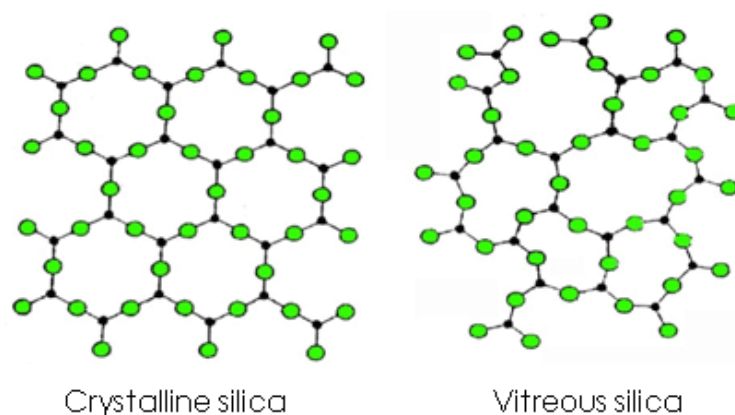
### **1.1.2 USES OF NANOPARTICLES**

- Nanoparticles have many applications in medicine for controlled drug delivery via fullerenes - an example of nanomedicine application.
- Nanotechnology is being applied to the production of synthetic skin and implant surgery.

- Nanomaterials that conduct electricity are being used in electronics as minute conductors to produce circuits for microchips.
- Materials in cosmetics, deodorants and sun creams may be of nanoparticle size and they are used to improve moisturizers without making them too oily.
- Nanoscale materials are being developed as new catalysts for fuel cells.
- Nanoparticle substances are incorporated in fabrics to prevent the growth of bacteria.

## **1.2 SILICA**

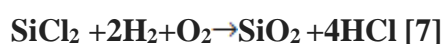
Silica is found most abundantly in the earth's crust. Silica contributes mass of 59% of earth's crust and 95% of rock. Silica is also known as silicon dioxide, compound of two most abundant elements, silicon and oxygen,  $\text{SiO}_2$ . It exists in two varieties, amorphous and crystalline. In crystalline forms the structures are characterized by tetrahedral configuration of atoms with the crystals whereas in amorphous forms the silicate subunits show no regular lattice pattern in the structure. The three main crystalline varieties of silica are: quartz (most abundant), tridymite and cristobalite. Other varieties include coesite, keatite and lechatelierite. The solid amorphous states are opal, flint, vitreous silica or diatomaceous silica. In amorphous forms, the silicate subunits show no regular pattern. [3].



*Figure 1.1 Two types of silica*

In silicon dioxide, silicon atom uses d orbital for bonding and hence exists as infinite three dimensional structures and it is a high melting solid. Silica which is amorphous contains about 4% water and can be represented as  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ . It consists of silicon and oxygen arranged in a tetrahedral structure. Surface silanol concentration (silanol groups- Si-OH) influences the degree of hydration. Water content in silica can affect processing and vulcanization of rubber. Absorbed water can decrease cure time, tensile strength and also abrasion resistance. The hydroxyl groups on the surface of silica control surface acidity. This intrinsic acidity can influence vulcanization [4-6]. These sites affect the rubber filler interaction.

Silicon dioxide is obtained by mixing and purification of the resulting mineral. Quartz comprises more than 10% by mass of earth's crust. It is also produced almost always via the processing of quartz, synthetically on very large scale. Pyrogenic silica is very fine particulate for colloidal form of silicon dioxide. It is prepared by burning  $\text{SiCl}_4$  in an oxygen rich hydrocarbon flame to produce a smoke of  $\text{SiO}_2$ .



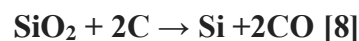
Amorphous silica gel is produced by the acidification of sodium silicate. The initially formed gelatinous precipitate is then washed and dehydrated to produce colourless micro porous silica. Idealized equation involving a trisilicate and sulphuric acid is



Thin films of silica grow spontaneously on silica wafers via thermal oxidation. This leads to the formation of shallower layer of native oxide. The native oxide layer can be beneficial in microelectronics where it acts as electric insulators with high chemical stability in electrical applications. It can protect the silicon, store charge block current and even act as a controlled pathway to limit current flow.

### 1.2.1 USES

- ❖ Used for the production of Portland cement.
- ❖ Silica is used in the production of glass for windows, drinking glass, etc.
- ❖ Used for the manufacturing of optical fibers for telecommunication.
- ❖ Used as raw materials for ceramics such as earthen wares, stone ware and porcelain.
- ❖ Used for the production of elemental silicon. This process involves carbothermic reduction in an electric arc furnace.



- ❖ Used as an additive in production of food, where it is used as a flow agent in powdered food.
- ❖ Colloidal silica is also used as a wine, beer and juice fining agent.

- ❖ In pharmaceutical products, silica aids powder flow when tablets are formed.
- ❖ Silica based aero gel was used in star duct spacecraft to collect extraterrestrial particles.
- ❖ Used in the extraction of DNA and RNA due to its ability to bind with nucleic acid.
- ❖ Used in toothpastes as a hard abrasive to remove tooth plaque.
- ❖ Used for the production of high thermal temperature protection fabric.

### 1.2.2 PREPARATION AND CHARACTERISATION OF SILICA

Acidification of alkali silicate solutions under controlled conditions produces precipitated silica.



Colloidal pyrogenic silica is produced by reaction of silicon tetrachloride at high temperature with water.



The reaction products are quenched immediately after coming out of the burner. But pyrogenic silica is too active and expensive.

Precipitated silica is silicon dioxide containing about 10-14% water. They are used as reinforcing fillers for making composites of high tensile strength, tear resistance, abrasion resistance and hardness. It is being used in the manufacture of translucent and coloured products, shoe soles, tires and other mechanical rubber goods. Fumed or pyrogenic silica is silicon dioxide containing less than 2% combined water. These are highly

reinforcing fillers of very small particle size giving high tensile strength, tear resistance, and abrasion resistance particularly to silicon rubbers [10].

Characterization of silica filler is also based on particle size and specific surface area. Surface area measurement is usually done by nitrogen adsorption [BET] method. Also pH, chemical composition and oil absorption are specified. The smallest physically observable primary particle for precipitated silica is about 15– 20  $\mu\text{m}$  and for fumed silica it is about 15 $\mu\text{m}$  in size. The surface forces of all small primary particles are so high that many particles agglomerate to form the so-called secondary particles.

### 1.3 NANOSILICA

Nano silica consists of spherical particles having a diameter less than 100 nm. Chemically speaking, they are made of silicon and oxygen atoms (fig: 1.2)

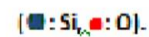
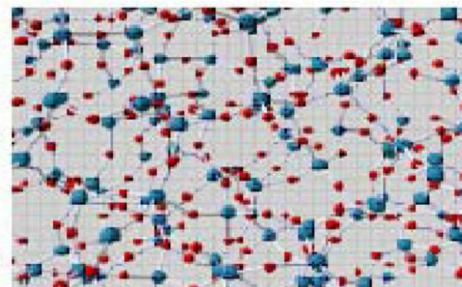


Figure 1.2 Chemical structure of nanosilica

Silica synthesis evolved during last decades from thermal hydrolysis silane resulting in non dispersible aggregated nanoparticles to sol-gel process resulting in well-defined nanoparticles highly compatible with the targeted matrix. Processes enabling chemically tuned and well integrated particles together with the nano scale effect are a high way to high performance nano composite materials having enhanced mechanical properties.

### **1.3.1 ADVANTAGES OF NANOSILICA OVER SILICA**

Silicon is one of the most abundant element in the earth's crust. It has been used for decades in the semiconductors and photovoltaic industries. Relative to some other semiconductor materials, silicon is safe, benign and inexpensive. When silica is compared with nanosilica, it is found that nanosilica is applied to high efficiency photovoltaic system and high capacity lithium ion battery. Nanosilica has several advantages over its regular, bulk cousin and other methods for micro or nanostructuring. Specifically, relative to bulk silicon, nano silicon's optical properties can be turned to dramatically reduce reflection and increase absorption, which are critical parameters for a high efficiency solar cell. It does not possess health hazards like the crystalline quartz which has been associated with silicosis.

### 1.3.2 SYNTHESIS OF NANOSILICA

Some of the methods used to synthesize silica nanoparticles are reverse microemulsion, flame synthesis and sol-gel process. In reverse microemulsion, the surfactant molecules dissolved in organic solvents forms spherical micelles. In the presence of water, the polar head groups organize themselves to form microcavities containing water, which is often called as reverse micelles. Silica nanoparticles can be grown inside the micro cavities by carefully controlling the addition of silicon alkoxides and catalyst into the medium containing reverse micelles. The major drawbacks of the reverse micro emulsion approach are high cost and difficulties in the removal of surfactants in the final products [12].

Silica nanoparticles can also be produced through high temperature flame decomposition of metal-organic precursors. This process is also referred to as chemical vapor condensation (CVC). In a typical CVC process, silica nanoparticles are produced by reacting silicon tetrachloride,  $\text{SiCl}_4$  with hydrogen and oxygen. Difficulty in controlling the particle size, morphology, and phase composition is the main disadvantage of the flame synthesis. Nevertheless, this is the prominent commercial method used in the synthesis of nanosilica[13].

A cheap and environment friendly route towards the synthesis of nanosilica hybrid composites has been presented by Tapasi *et al.* This is a sol-gel method in which the acid plays a catalytic role in enhancing sol-gel condensation of silicon alkoxides with poly (vinyl alcohol). First colloidal stable silica was prepared by the acid neutralization with an objective to increase the gelatin time and decrease rate of self condensation of silica. At lower PVA concentration, co-condensation occurs [14].



Nanosilica can be synthesized by precipitation method using sodium silicate and hydrochloric acid in presence of a polymeric dispersing agent. Poly (vinyl alcohol), starch, carboxy methyl cellulose and poly (ethylene glycol) were found to be good dispersing agents because they are macromolecules and contain a large number of hydroxyl groups per molecule.

The interaction between hydroxyl groups of dispersing agents and the hydroxyl groups of silica would result in co-condensation. The synthesized silica after complete drying is to be calcinated to remove the dispersing matrix and to obtain pure silica. The presence of organic molecule eliminates the need for usage of coupling agent during nano composite preparation.

### **1.3.3 DISPERSING AGENTS USED IN THE SYNTHESIS OF NANOSILICA**

A dispersing agent is a non surface active polymer or a surface-active substance added to a suspension, usually a colloid, to improve the separation of particles to prevent settling or clumping. The dispersing agents that are used in the synthesis of nanosilica are given below [11].

#### **(A) Starch**

Starch consists of two types of molecules; amylose (normally 20 to 30%) and amylopectin (normally 70 to 80%). Both consist of polymers of  $\alpha$ -D

glucose units in the  $4C_1$  conformation. In amylose these are linked – (1, 6)-branch points. Both depend on the surface of the starch, for example; amylomaizes contain over 50% amylose whereas ‘waxy’ maize has almost none (~3%).

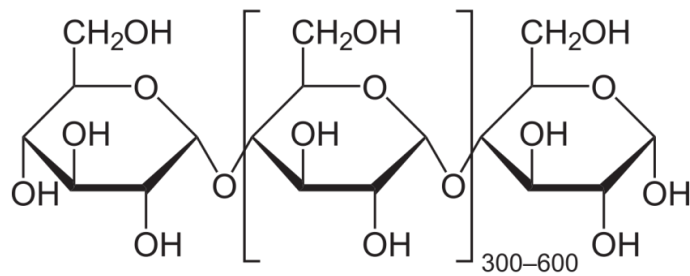


Figure 1.3 Structure of amylose

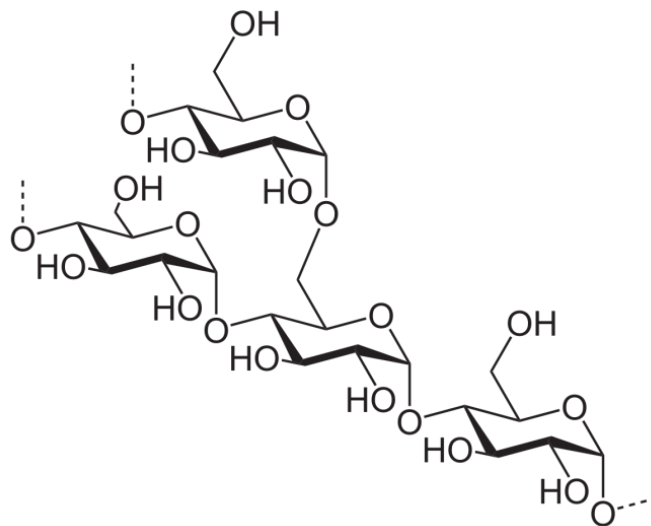
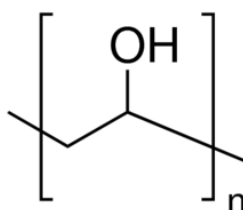


Figure 1.4 Structure of amylopectin

**(B) Poly (vinyl alcohol)**

Poly (vinyl alcohol) is a water soluble polymer having the following structure:-



It dissolves slowly in cold water, but more rapidly at elevated temperature and can usually be dissolved completely above 90°C. The aqueous solutions are not particularly stable especially if traces of acids or bases present results in irreversible gelation reactions. For example, cross-linking can occur at either linkage, resulting in increased viscosity through the formation of insoluble products.

**(C) Carboxy Methyl Cellulose (CMC)**

Carboxy Methyl Cellulose (CMC) is a derivative of cellulose formed by its reaction with alkali and chloro acetic acid. Sodium carboxymethyl cellulose is an anionic water soluble polymer. The CMC structure is based on the  $\beta$  (14)-D glucopyranose polymer of cellulose. Different preparations may have different degrees of substitution, but it is generally in the range 0.5-0.95 derivatives per monomer unit. CMC molecules are somewhat shorter, on average than native cellulose with uneven derivitization giving areas of high and low substitution.

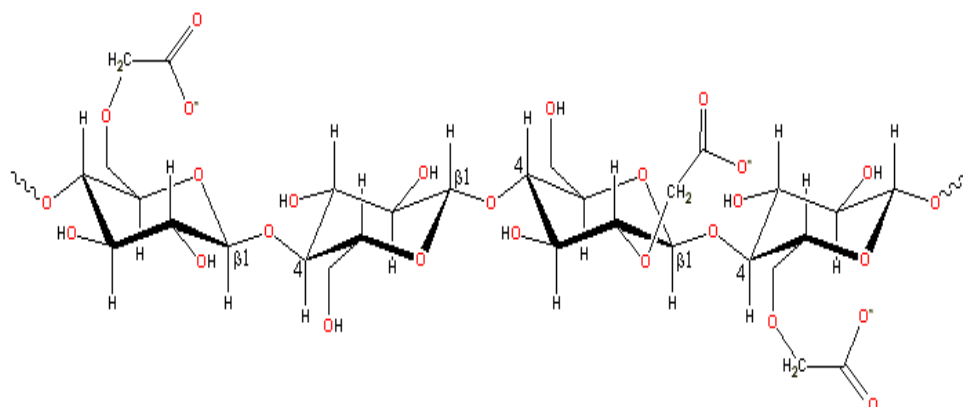


Figure 1.5 Structural unit *o*-carboxy methyl cellulose

This substitution is mostly 2-*o*- and 6-*o*- linked followed in order of importance by 2,6-di-*o*- then 3-*o*-, 3,6-di-*o*- lastly 2,3,6-tri-*o*- linked. It appears that the substitution reaction is slightly cooperative (within residues) rather than random process giving slightly higher than expected unsubstituted and trisubstituted areas. CMC molecules are most extended at low concentrations but at higher concentrations, the molecules overlap and coil up and then at higher concentrations, entangle to become a thermo reversible gel. Increasing ionic strength and reducing pH decrease the viscosity as they cause the polymer to become more coiled.

#### (D) POLY (ETHYLENE GLYCOL)

Poly ethylene glycol is a polymer of ethylene oxide obtained by polymerization reaction in presence of water. It is a kind of liquid or a low melting solid depending on their molecular weight. Poly ethylene glycol is soluble in water, methanol, ethanol, acetonitrile, benzene and dichloromethane, and insoluble in diethyl ether and hexane. It is a

polyether having straight chain and branched chain. It is hydrophilic in nature since it can form hydrogen bond with water and hence helps in separation of the substances.

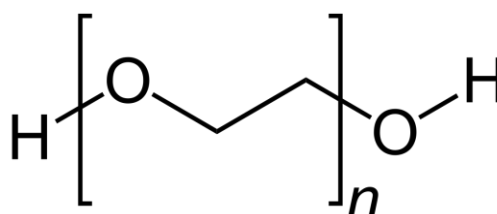


Figure 1.6 structure of poly ethylene glycol

#### 1.3.4 APPLICATIONS OF NANOSILICA

- Nanosilica can be compounded with rubber and can be used for making shoe out soles and rubber footwear components due to high abrasion resistance, good grip and non dusting qualities of nanosilica .
- Due to their optimal property, nanosilica is used in battery separators.
- Nanosilica enhances polymer property like flame retardancy, barrier properties/transparency and starch resistance.
- Nanosilica is also used as micro cellular product, like shoes, mat etc. due to their foaming control.
- In optic electronics nanosilica is used in protective coatings and encapsulations of electro optically active materials.
- It is used in drug encapsulation as catalytic vehicles and supports, preparations, particular calibration standards, chromatography, other separations and

- It has been found that silica aero gel coated on surface of granular activated carbon has four times more ability to remove uranium, chromium and arsenic from water supplies.
- Nano-sized silica abrasives are being researched for use in chemical – mechanical polishing of copper, to minimized mechanical stress during polishing and reduce defects such as surface scratches, copper peeling, dishing and erosion.
- Nano silica filled room temperature vulcanized silicone rubber has higher erosion resistance, lower hydrophilicity and lesser surface roughness and which is used as insulators in coastal areas.
- The film of nanosilica known as Xerocoat, when applied to glass/mirror cuts unwanted reflections from glass, letting more light through ,hence reduces fogging and improves vision.
- Nanosilica used in rubber products repellant properties of fabric by creating nanowhiskers.
- Nanosilica particles are also used for intercellular sensing of oxygen, pH and ionic species.

#### **1.4 SCOPE AND OBJECTIVES OF THE PRESENT WORK**

Silica nanoparticles are well known for their variety of applications in various fields including biomedicine, such as; colloidal nanosilica can be used in relatively small quantities as cofillers together with large size fillers to achieve high packing capacity in hybride type composite. This has been attributed to their size, stability, low toxicity, tensibility, easily modifiable capabilities and ability to be functionalized with a range of molecules and polymers because of the presence of functional groups and

their biocompatibility. With the growing demand of nanosilica, economic competition and ecological pressure, there is an increasing need to synthesis naosilica from a cost effective source and by a cheap method.

The objective of the current work is to synthesize nanosilica from a cheap source such as sodium silicate and make the process of preparation of nanosilica less expensive. The concept of condensation of silica particle into a dispersed polymeric matrix by the catalytic action of an acid was adopted from the work of Ktoky and Doloui [22].The study also aims to explore the possibility of different polymeric dispersing medium in the synthesis of nanosilica. During this reaction, the product formed will be silica and not polymer silica nanocomposite. Then by keeping the silica concentration low, it was expected that the precipitated silica would be collected in the polymer matrix and hence its agglomeration would be prevented. The addition of the dispersing agent would produce a matrix into which synthesized silica would be incorporated, thus producing silica in the nanoscale.

# Chapter 2

## Materials and Methods

This chapter gives a brief description of the materials and experimental procedures adopted for the present investigation.

### 2.1 MATERIALS

#### 2.1.1 Sodium silicate

Sodium silicate penta hydrate manufactured by Nice Chemicals (P) Ltd, Kochi, Kerala

#### 2.1.2 Hydrochloric acid

A.R HCl manufactured by Nice Chemicals (P) Ltd, Kochi, Kerala with an acidimetric assay of 36.46% was used.

#### 2.1.3 Soluble starch

Soluble starch manufactured by Laboratory Chemicals, Indian Drugs and pharmaceuticals, Hyderabad was used.

#### 2.1.4 Poly vinyl alcohol

Cold water soluble poly vinyl alcohol manufactured by Nice Chemicals (P) Ltd, Kochi, Kerala

### 2.2 EXPERIMENTAL METHODS

#### 2.2.1 Synthesis of Nanosilica

##### (A) Experimental set up



The experimental setup consists of:

- Reactor

The experiments were carried out in small scale so 500 ml Borosil beaker was used as the reactor.

- Stirring Cum Heating Arrangement

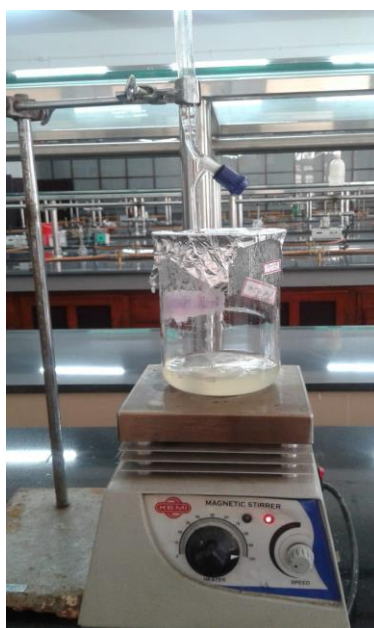
Magnetic stirrer was used for stirring. Heating was done using a hot plate which was set at 60°C.

- Filtration Assembly

Vacuum filtration was done using Buchner funnel, suction flask, tubing and a vacuum pump.

- Drying Assembly

Hot air oven with a temperature setting adjustable to 300°C was used.



*Fig2.1 Experimental setup*

**(B) Procedure**

100ml of 15% sodium silicate solution was prepared and transferred into a beaker. 1% starch solution was prepared by dissolving 1gm starch in 100ml of water and 5ml of this solution was added to the sodium silicate solution. This mixture is slowly stirred on a magnetic stirrer and heated on a hot plate until it attained a temperature of 100°C. 1N HCl was added from a burette. The pH was maintained between 1 and 2. During the addition of HCl, instantaneous gelation occurred sometimes and stirring became difficult. So distilled water was added to keep the reaction in a slurry form. The reaction mixture was stirred continuously for a period of two hours and the temperature was maintained at 60°C. These reaction conditions enabled the uniform distribution of the dispersing agent, starch in the reaction solution, so that it could act as a matrix to collect the formed particles. In the absence of heating, the dispersing agents agglomerated in the reaction medium. It also enabled the conversion of silicic acid, formed by the reaction between HCl and sodium silicate into silica.

The reaction between sodium silicate and HCl is,



The acid plays a catalytic role in enhancing the condensation of silicon oxides within the dispersing agent's matrix. It is expected that the addition of the above dispersing agent would produce a matrix into which synthesized silica would be incorporated, thus producing silica in the nanoscale. The interaction between the hydroxyl groups of silica would result in condensation. Hydrogen bonding between the polymer and the developing polysilicate networks leads to system homogeneity.

After completion of reaction, the slurry was kept at room temperature for 24 hours. It was then filtered by vacuum filtration. A suction flask with Buchner funnel and tubing to vacuum were used. Two filter paper cut exactly to the inner diameter of Buchner funnel was placed in the funnel. Vacuum pump was started;

the slurry was slowly poured in to the funnel, always maintaining a slurry level of not more than 50% of the funnel height. After completely emptying out the slurry into the funnel, the washing was started by adding distilled water into the funnel. The washings continued until all the sodium chloride is removed. After filtration cum washing, the cake collected on the filter paper in the Buchner funnel was scraped cut using a scraper and spread evenly on a glass plate. This was then placed in a hot air oven at a temperature of 60°C for 24 hours. The cake thus obtained was to obtain fine powder of silica. The synthesized silica after completely drying was calcinated in muffle furnace at 600°C for 6 hours to remove the dispersing agent's matrix. The pure silica particle obtained was then characterized using IR and XRD. Preparation of nanosilica was repeated using 3% and 5% of starch solution and 1%, 3% and 5% of poly vinyl alcohol solution as the dispersing agent.



*Before calcination*



*After calcination*

*Fig2.2 Nanosilica*

## **2.2.2 Characterization**

### **2.2.2.1 Infra red spectroscopy (IR)**

The IR spectrum of the synthesized silica was recorded with Fourier Transform Infrared Spectroscopy, Thermo Nicolet, Avatar 370 operating in the range 4000-400  $\text{cm}^{-1}$  with a DTGS detector.



*Figure 2.1 IR Spectroscopy*

#### **2.2.2.2 X-RAY diffraction (XRD)**

The XRD analysis was carried out with X-Ray Diffractometer, Bruker AXS D8 Advance model, employing CuK $\alpha$  radiation ( $\Lambda=1.54\text{\AA}$ ). The particle size of the samples was calculated using the Debye-Sherrer formula [23-24]

$$D=0.9\Lambda/ \beta \cos\theta \dots 2.3$$

where,  $d$  is the particle size,  $\Lambda$  is the wavelength of the incident X-ray beam,  $\beta$  is the full width at half maximum (FWHM) of an  $hkl$  X-Ray diffraction peak at  $2\theta$  value [25] and  $\theta$  is half of the angle  $2\theta$  corresponding to the peak.

# Chapter 3

## Results and discussion

### 3.1 INFRARED SPECTROSCOPY

Figure 3.1 to 3.3 shows the IR spectrum of synthesized silica with 1, 3 and 5% starch as dispersing medium, respectively.

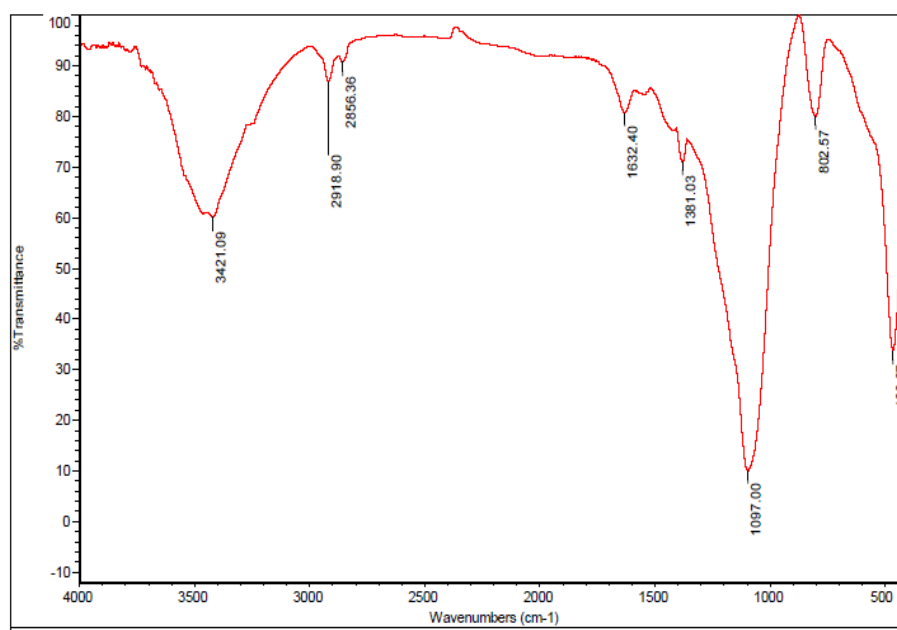


Fig 3.1 IR spectrum of the silica with 1% starch as dispersing medium

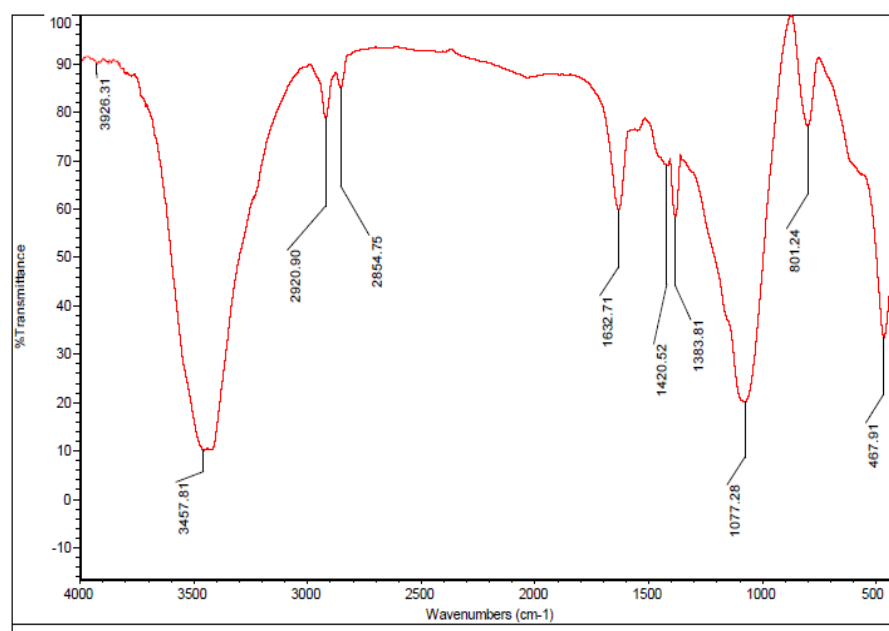


Fig 3.2 IR spectrum of the silica with 3% starch as dispersing medium

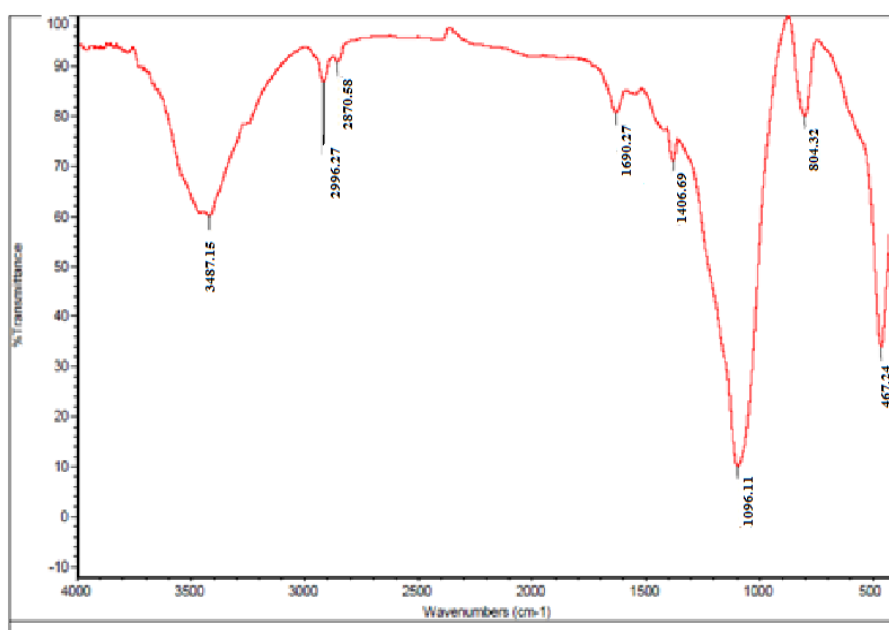
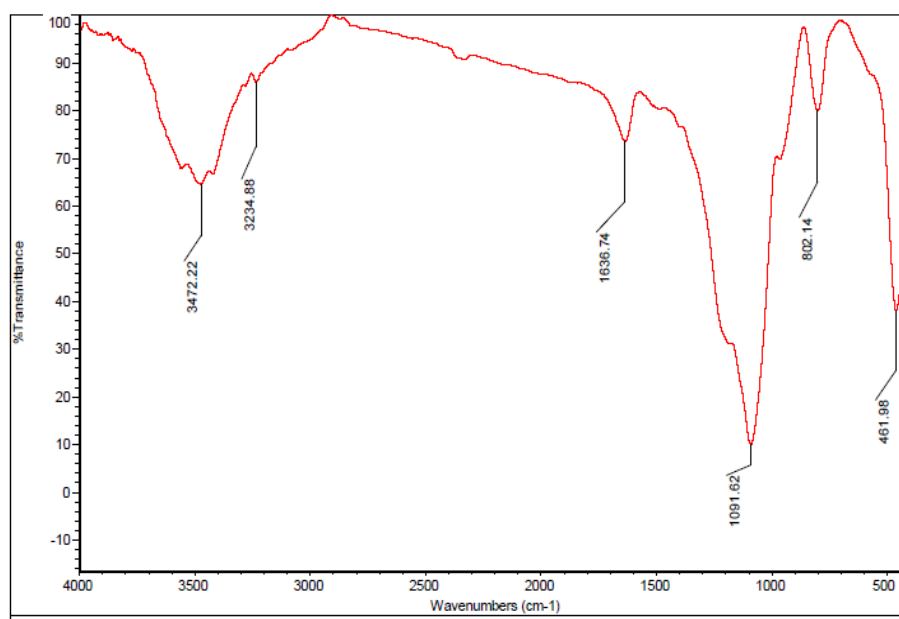


Fig 3.3 IR spectrum of the silica with 5% starch as dispersing medium

The broad band between 3000 and 3750  $\text{cm}^{-1}$  in the spectra is due to silanol -OH groups and adsorbed water. The predominant peak between 1097-1077  $\text{cm}^{-1}$  for 1%, 3% and 5% of starch as dispersing medium, is due to siloxane bonds (Si-O-Si). The peaks between 1000 and 700  $\text{cm}^{-1}$  are attributed to vibration modes of the gel network.

It can be concluded that the synthesized silica consist predominantly of silicon oxide. The -OH stretching peak for the sample with 1%, 3% and 5% starch as dispersing medium is at 3421.09, 3457.81 and 3487.15  $\text{cm}^{-1}$  respectively.

Figure 3.4 to 3.6 shows the IR spectrum of synthesized silica with 1, 3 and 5% poly vinyl alcohol as dispersing medium, respectively.



*Fig 3.4 IR spectrum of the silica with 1% polyvinyl alcohol as dispersing medium*

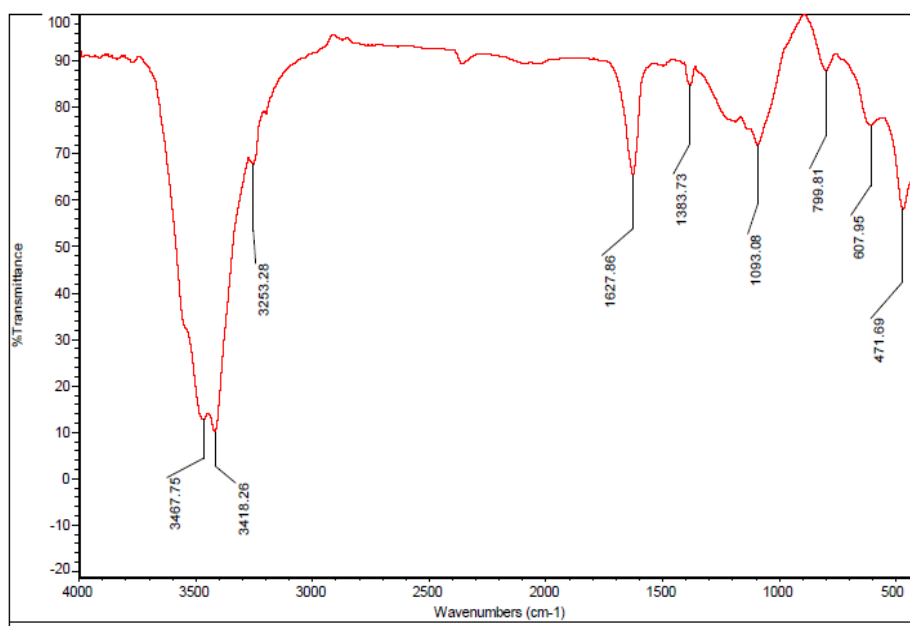


Fig 3.5 IR spectrum of the silica with 3%polyvinylalcohol as dispersing medium

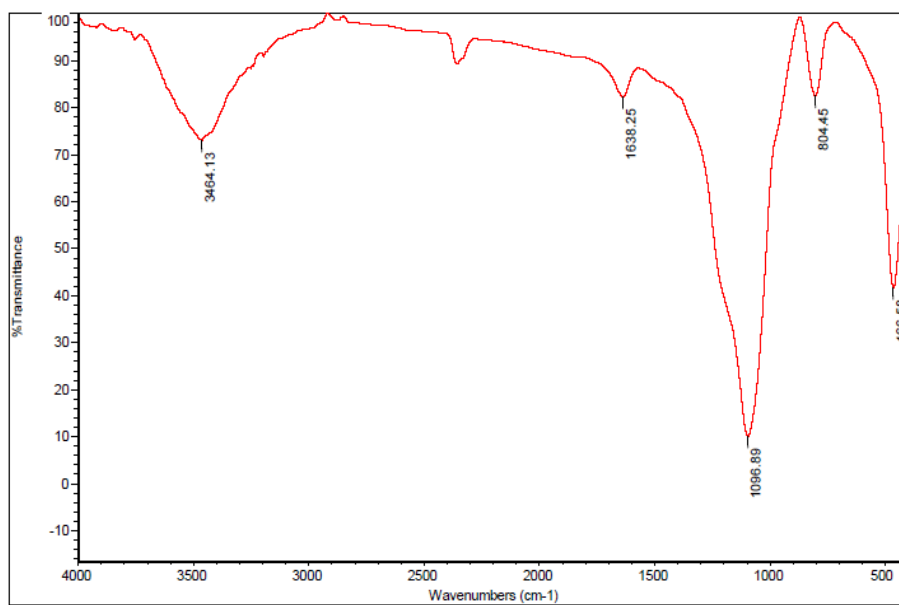


Fig 3.6 IR spectrum of the silica with 5%polyvinylalcohol as dispersing medium

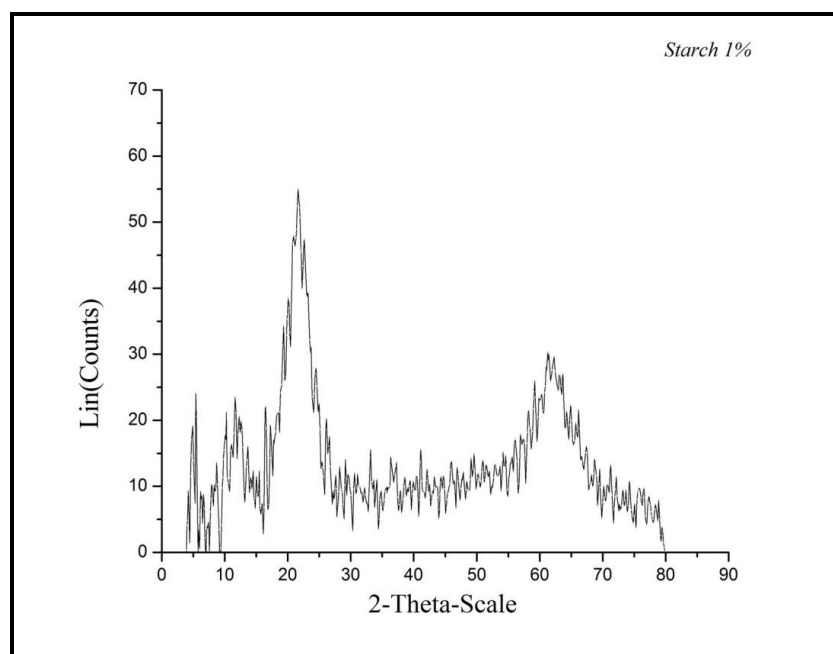


The broad band between 3000 and 3750  $\text{cm}^{-1}$  in the spectra is due to silanol -OH groups and adsorbed water. The predominant peak around 1096  $\text{cm}^{-1}$  for 1%, 3% and 5% of polyvinyl alcohol as dispersing medium, is due to siloxane bonds (Si-O-Si). The peaks between 1000 and 700  $\text{cm}^{-1}$  are attributed to vibration modes of the gel network.

It can be concluded that the synthesized silica consist predominantly of silicon oxide. The -OH stretching peak for the sample with 1%, 3% and 5% polyvinyl alcohol as dispersing medium is at 3472.22, 3467.75 and 3464.13  $\text{cm}^{-1}$  respectively.

### 3.2 X-RAY DIFFRACTION

X ray diffraction of the silica samples are shown in fig 3.7-3.12



*Fig 3.7 XRD pattern of the silica with 1%starch as dispersing medium*

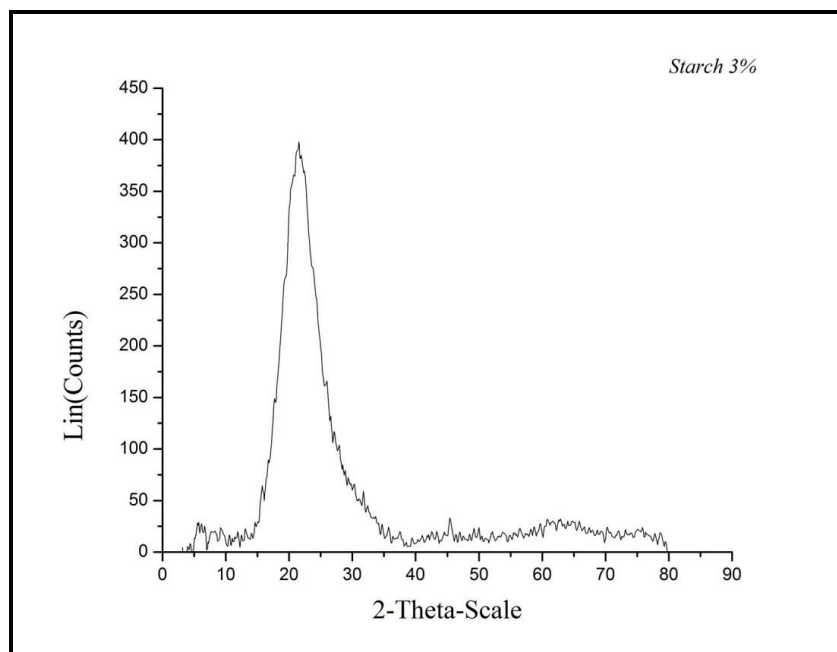


Fig 3.8 XRD pattern of the silica with 3%starch as dispersing medium

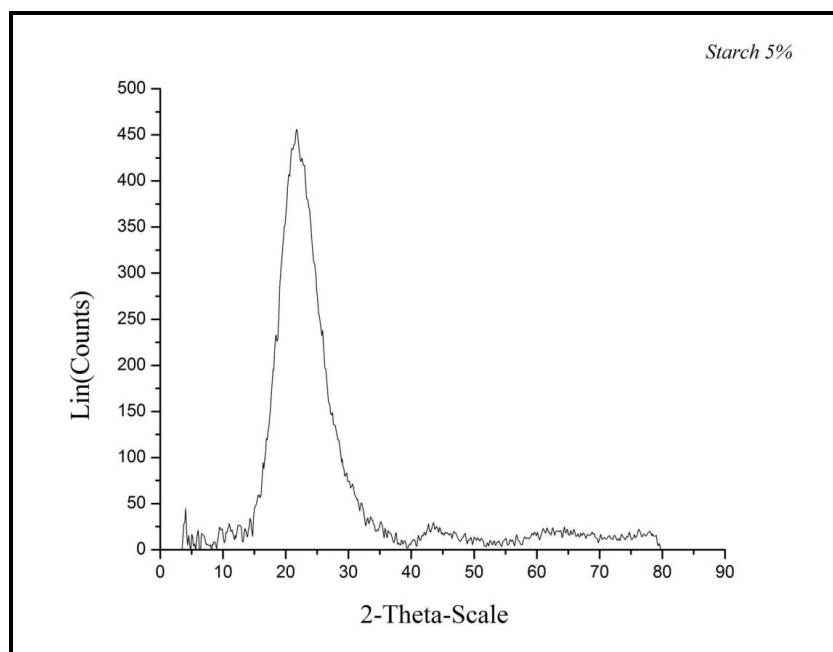
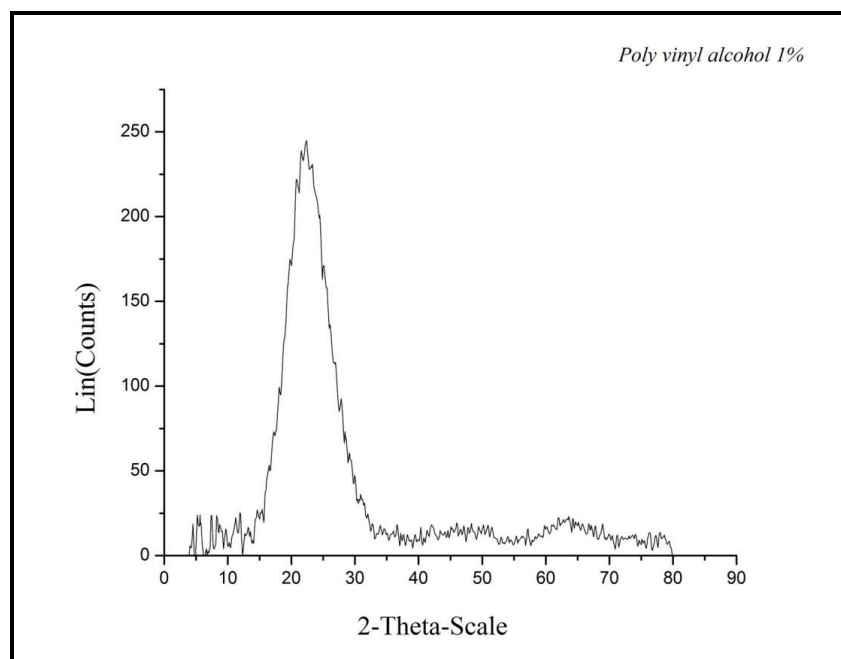
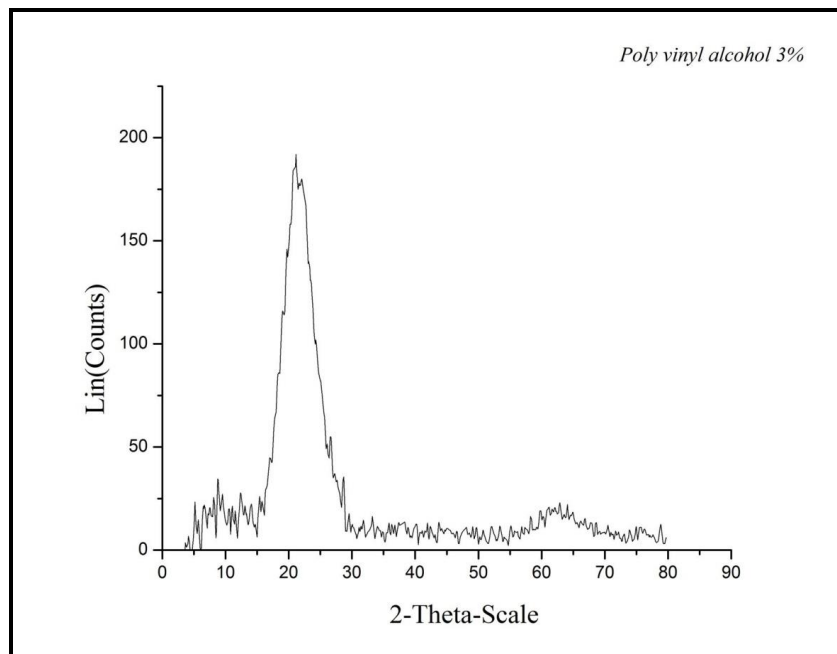


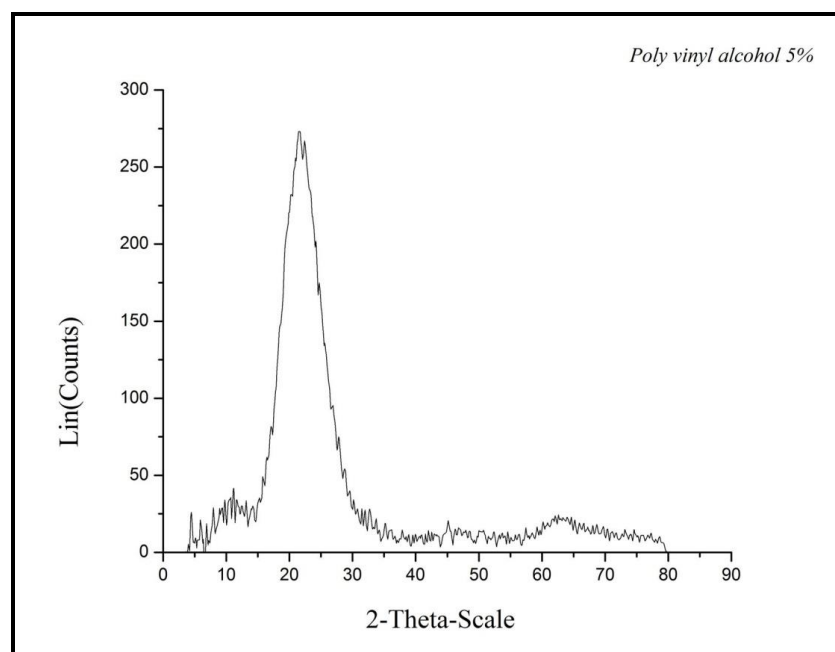
Fig 3.9 XRD pattern of the silica with 5%starch as dispersing medium



*Fig 3.10 XRD pattern of the silica with 1%polyvinylalcohol as dispersing medium*



*Fig 3.11 XRD pattern of the silica with 3%polyvinylalcohol as dispersing medium*



*Fig 3.12 XRD pattern of the silica with 5%polyvinylalcohol as dispersing medium*

Strong broad peaks between a  $2\theta$  of  $21-26^\circ$  are seen. These strong peaks are characteristics of amorphous  $\text{SiO}_2$ . This shows that the synthesized silica is in an amorphous state. The full width at half maximum (beta) was used to determine the particle of the sample using the Debye-Scherrer formula. The average particle size,  $C_s$ , of the silica sample was found to be 4.272, 2.666 and 2.569 nm respectively for 1%, 3% and 5% of starch and 2.355, 2.398 and 2.454 nm for 1%, 3% and 5% of polyvinyl alcohol as dispersing agents.

# Chapter 4

## Conclusions

Nanosilica can be successfully prepared by precipitation method under controlled conditions using starch and poly vinyl alcohol as dispersing agents. XRD results show that synthesized silica is predominantly amorphous. Nanosilica prepared by this method has a particle size lower than that of the reported values of commercially available silica. This is confirmed from IR and XRD data. The optimum concentration of starch as dispersing medium is 5% and poly vinyl alcohol as dispersing agent is 1%, as the sample shows least particle size of 2.569 nm and 2.355 nm, respectively.

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