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Synthesis and characterization of spherical silica nanoparticles by Sol-Gel method

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Abstract: Silica nanoparticles were synthesized by sol gel method from tetraethyl orthosilicate (TEOS), ethanol (C_2H_5OH), water (H_2O) and ammonoium hydroxide (NH_4OH)as catalyst. The morphology and structure of colloidal silica particles formed depend on the molar ratio of reagents. The XRD patterns show the amorphous nature of the particles. FTIR spectra confirm the presence of functional group in the bulk and at the surface of the silica particles. SEM image shows that spherical structure of silica nano particles, whose particle is varied by using different molar ratio TEOS, C_2H_5OH and NH_3. The FTIR and EDAX analyses prove the successful synthesis of silica material.

1. Introduction

Silica nanoparticles are widely used in industrials such as electronic devices, insulator, catalysis or pharmaceuticals [1, 2] due to their attractive properties in optical properties.The most popular process of obtaining silica nanoparticles is through sol gel technique [3-7]. It involves the simultaneous hydrolysis and condensation reaction of the metal alkoxide. The resultants desired particles size and morphology of silica particles are produced through controlling parameters such as concentration of alkoxide, amount of water and concentration of ammonia or acid and solvent and aging time.

2. Experimental methods

2.1. Preparation of silica (SiO_2) nano powder

Chemicals used in this experiment are Tetraethyl Orthosilicate (TEOS), concentrated Ammonia (NH_3) and Ethanol (C_2H_5OH) solution. Tetraethyl Orthosilicate (TEOS) is used as the silica source. Aqueous ammonia solution was used as the catalyst. All the chemicals are purchased from Aldrich without further purification. Distilled water was used throughout the experimentSilica nanoparticles were synthesized using a standard procedure with experimental conditions provided in Table 1. The product was grained to get the silica nanoparticle.

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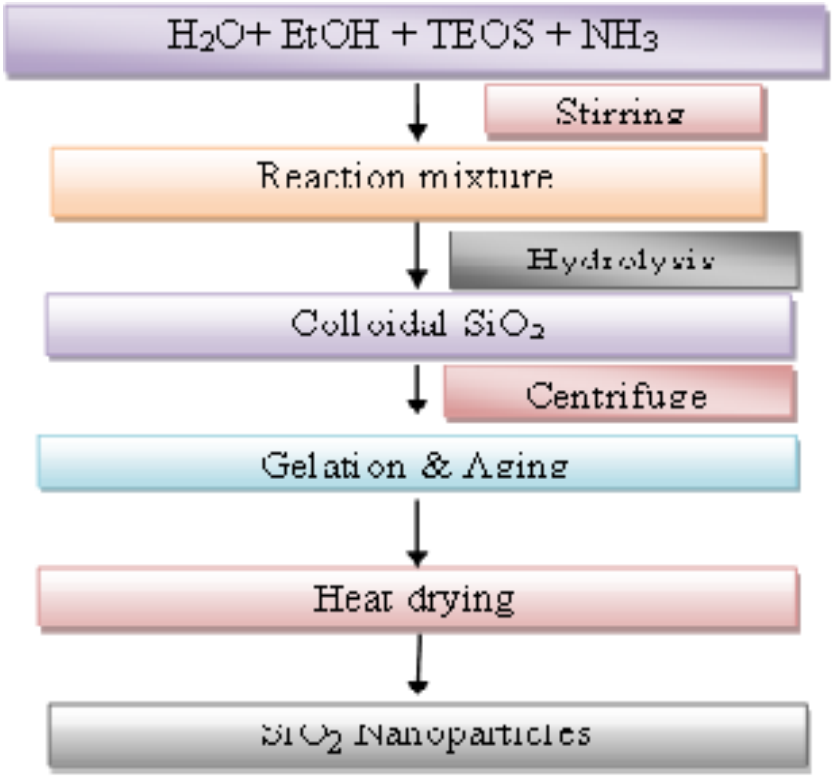

Flow chart for the Synthesis of silica nanoparticles by sol-gel method

Table 1: Molar ratios for the preparation of silica nanoparticles

Sample	Molar Ratio			
	H_2O	TEOS	NH_3	EtOH
a.	1	5	7	14
b.	1	5	9	7
c.	1	7	7	7

2.2. Characterization

The characterization of nanoparticles is done by using different techniques. The crystalline structure, morphology and compositional analysis of the prepared samples are examined by that the X-ray Diffraction (XRD), Scanning Electron Microscope(SEM), and Energy Dispersive Analysis using X-rays (EDAX) respectively. Nature of bonding and the chemical composition were analyzed by Fourier Transfer Infrared spectra (FTIR).

3. Results and Discussion

3.1. X-ray diffraction analysis

The crystal structures and phases of all the synthesized nanomaterials were ascertained from the XRD pattern. The figure shows that the XRD patterns of silica nanopowder prepared by sol-gel method for different molar ratios of ammonia, TEOS and ethanol concentration. From the three graphs (Fig 1) shows that the particles are amorphous in nature. The intense peak at $\theta = 23^{\circ}$ indicates, that the silica particles are formed by small nanocrystals. The broadening of peak is high owing to the smaller grain size effect. Other peaks are not present which represents the amorphous in nature due to the smaller particle size effect and incomplete inner structure of the nanoparticles [8]. XRD peaks which represent that silica nanoparticles structure is not changed entirely with small variation in ammonia, TEOS and ethanol concentration. By changing the different concentration, there is no phase change which represents the high purity of the silica nanoparticles. This demonstrates that high percentages of these particles are amorphous [9].

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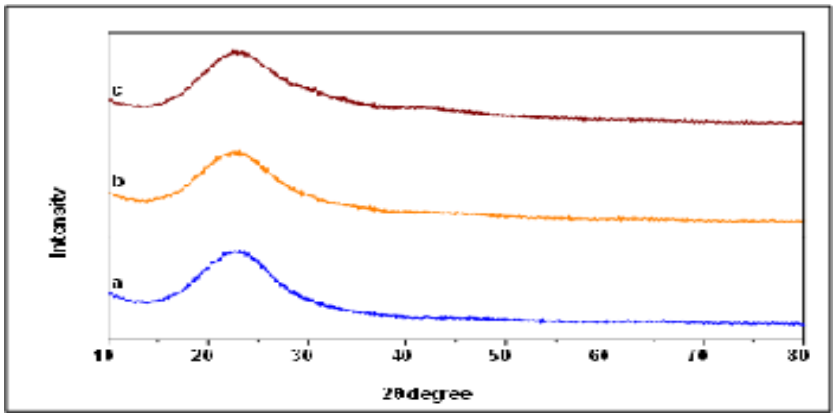

Fig 1: XRD spectrum of silica nanoparticle at different molar ratio of water, TEOS, NH_3, EtOH a) 1:5:7:14 b) 1:5:9:14 c)1:7:7:7

3.2. Morphological analysis (SEM) & Compositional analysis (EDAX)

The surface morphology of the silica nanopowder is analyzed using scanning electron microscope at the molar ratios of Water: TEOS: NH_3: EtOH as shown in Figure (2-4). The reaction was performed at lower water molecules in the solution to avoid aggregation of the silica nanoparticles at constant temperature. Therefore, hydrolysis and condensation of the reaction is carried out in alcohol with the presence of basic medium to get uniform distribution of silica nanoparticles. Figures 2-4 shows the spherical and agglomerated silica nanoparticles, which were obtained using different molar ratios of reagents and solvents.

The figure 2 shows that for preparing smaller silica nanoparticles, the reaction mixture should have higher concentration of ethanol and lower concentration of ammonia. Aggregation is always energetically favoured over nanoparticles since it minimizes surface areas and saturates the bonding and co-ordination sites and therefore, in order to prevent the nanoparticles from further growth or aggregation, the particle surfaces should be saturated immediately after nucleation by electrostatic or steric stabilization. Thus, the ammonia and solvent molecule of ethanol plays an important role to produce small size of silica nanoparticles with monodispersed spherical shape.

The figure 3 shows increase in particle size compared to sample 1. The base medium of ammonia is increased where as the amount of ethanol is decreased compared to sample 1. When the concentration of ammonia was changed corresponding decrease in the amount of solvent, many particles agglomerated, although few smaller spheres starts to form bigger silica nanoparticles. With the increase in the amount of ammonia, the sizes of the particle gradually increased and produce irregular spherical nanoparticles with high aggregation effect. The irregular shape of silica particles is obtained due to the fast nucleation process which is difficult to control the reaction by high concentration of basic medium and less solvent effect. And also decrease in the amount of ethanol the particle size increases [10]. Hence the SEM shows an increase in the size and irregular shape of the silica nanoparticles compared to SEM 1.

Figure 4 shows the silica nanoparticles were synthesized with same molar ratio of TEOS, Ammonia and Ethanol giving rise to larger silica nanoparticles with a broad distribution of particle sizes. Uniform orientation of the silica nanoparticles without aggregation is obtained is due to the covalent bond between the neighboring nanoparticles. Synthesis time was necessary because TEOS must be added very slowly to avoid any second nucleation or chemical aggregation. Second or multi nucleation could be avoided by three ways:1) increase of the ionic force in order to reduce the number of nucleation centers, 2) increase of particle surface in solution, 3) limiting TEOS concentration. Different concentration of TEOS, ammonia and ethanol, spherical silica nanoparticles is formed in different size which is not only alter the particle size and shape, also dramatically affects the optical properties of resultant nanoparticles.Finally we concluded, the particle size decreases with increasing molar ratio of ethanol and the particle size increases with increasing molar ratio of TEOS and ammonia.

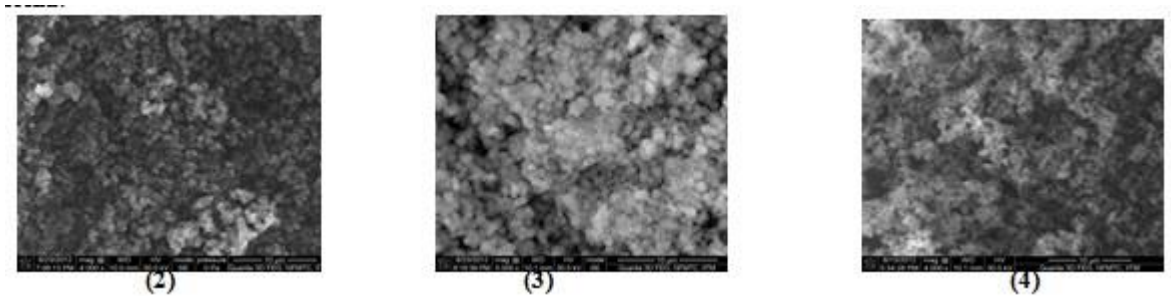

Fig 2, 3 and 4 shows SEM micrographs of silica nanoparticles obtained from a molarratio of Water: TEOS: NH_3: EtOH a) 1:5:7:14 b) 1:5:9:7 c) 1:7:7:7.

The elements of silica nanoparticles are confirmed by EDAX analysis, this is shown in figures 5,6 and 7. The

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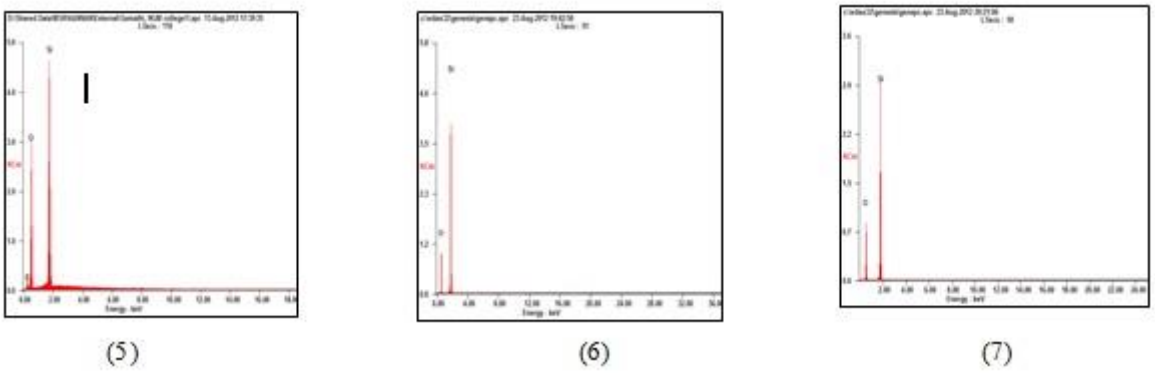
Composition is given in table 2 [11].



(5) (6) (7)

Fig 5,6 and 7 shows EDAX spectrum of Si particles of molar ratio a)1:5:7:14 b)1:5:9:7c)1:7:7:7

Table 2: EDAX result of silica nanoparticleswith different molar ratio

S.No	Molar Ratio				Elements	Atomic %
	H_2O	TEOS	NH_3	EtOH		
a.	1	5	7	14	Si : O	24.43 : 75.70
b.	1	5	9	7	Si : O	36.12 : 63.88
c.	1	7	7	7	Si : O	31.15 : 68.85

3.3 Fourier Transform Infrared Spectroscopy

FTIR results of present study in figure 8 shows the spectrum of silica nanoparticles at different molar ratio of Water, TEOS, NH_3, EtOH. The Band values of Sample a, b and c are summarized and are given in Table 3.[12-15].

Table 3: FT-IR spectra of Sample a, b and c

Name of the Sample	Adsorption bands (cm^{-1})	Functional groups
Sample a	457	Si-O-Si bond of bending vibration
	798	symmetric vibration of Si-O
	950	stretching vibration of Si-OH
	1633	bending vibration of water
	2856 and 2960	stretching and bending vibrations of C-H bond
	3448	H-bonded silanol OH group
Sample b	476	Si-O-Si bond of bending vibration
	800	symmetric vibration of Si-O
	950	SiO_2 stretching vibration of Si-OH bond
	1633	bending vibration of molecular water
	2856 and 2957	stretching and bending vibrations of C-H bonds
	3632	H-bonded silanol OH groups
Sample c	464	Si-O-Si bond shows bending vibration
	798	symmetric vibration of Si-O
	950.	SiO_2 stretching vibration of Si-OH bond
	1632	bending vibration of molecular water
	2856 and 2957	stretching and bending vibrations of C-H bond
	3454	H-bonded silanol OH group

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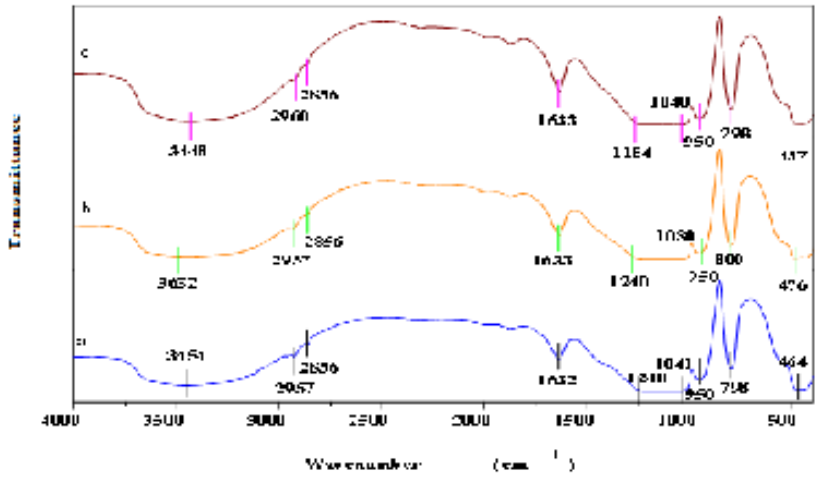

Fig 8: FTIR spectrum of Si-nanoparticles of molar ratio a) 1:5:7:14 b) 1:5:9:7c) 1:7:7:7.

Conclusion

We have successfully synthesized monodisperse silica spheres with the size ranging from >100 nm through sol-gel method. The reaction parameters can be used effectively for the synthesis of spherical silica nanoparticles at different molar ratio of Water, TEOS, NH_3, EtOH. The influence of the matrix obtained from the TEOS precursors and ammonia plays a major role in the evolution of the processes. The morphology and average diameter of colloidal silica particles depend on the proportion of reactants. XRD pattern shows the amorphous nature of silica nanoparticles. The SEM image shows the spherical structure of nanoparticles, whose particle size decreased with increasing molar ratio of ethanol in sample a. The particle size increased with increasing molar ratio of ammonia and decreasing molar ratio of EtOH in sample b. The particle size is increased for equal molar ratio of TEOS, NH_3, EtOH, in sample c. The FTIR and EDAX analyses prove the successful synthesis of the silica material. The resultant spherical silica nanoparticles synthesized can be used for various photocatalytic activity, assembly of photonic structures and these microspheres have potential for biomedical applications.

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