Synthesis and Characterization of Silica Nano-Particles by Acid Leaching Technique

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Abstract
Silica nano particles have been prepared by hydro fluoric acid leaching of sodium meta-silicate solution characterised its surface morphology by SEM image. The size and size distribution of these nanoparticles has also been examined using static light scattering instrument; the average diameter of the particles is found 176 nm. The FTIR spectrum of the powder confirms the presence of silica.

Keywords: Acid leaching, characterization, FTIR spectroscopy, silica nano.

Introduction
In nature silica is available in form of sand, quartz and cell walls of diatoms. Due to its inherent properties, it is widely used as flow agent in powdered foods, hygroscopic agent, it can also be used in filtration and cement industry. In European Commission 2004, it has been reported that when the size of particle is reduced to nano scale, the number of atoms are higher on the surface than inside the particle, and atoms on the surface may behave differently than those atoms inside the particle. At this minute scale, nanoparticles are affected by quantum effects and other physical and chemical properties in ways that are not significant at larger scales, reported by Breggin and Pendergrass. Using modern technology, therefore, it is possible to expose matter to extreme conditions (such as extreme cold or a vacuum) which can then change its properties.

There are several methods for creating nano-particles, including both attrition and pyrolysis. Initially evaporation and condensation method was reported by Gleiter in 1989 and Siegel in 1991, 1994. Gradually various methods i.e. aerosol, combustion flame, laser ablation, chemical vapor condensation, spray pyrolysis, electrospray and plasma spray. Additional techniques reported for the synthesis of silica nanoparticle are sonochemical, cavitations and microemulsion processing and also high-energy ball milling. Majority of these processes for the synthesis of nano particles are criticized for high energy consumption and process complicity. In the present work an attempt has been made to synthesized SiO$_2$ nanoparticles by the leaching of sodium meta-silicate (Na$_2$O,Si,9H$_2$O) with hydro fluoric acid. The synthesized nano particles were characterized using static light scattering instrument, scanning electron microscope (SEM) and FTIR spectroscopy for their size, shape and chemical composition respectively. Silica nano particles were further elementary analyzed confirmed by Oxford-Inca software.

Material and Methods
Materials: Chemicals: Sodium meta silicate (Na$_2$O,Si,9H$_2$O, MW 284) of analytical grade purity were procured from Loba chemie Pvt.Ltd. Hydro fluoric acid (40%) (HF MW 20.01) and Poly acryl amide (CH$_2$CHCO NH$_3$, MW 71.08) of analytical grade purity were procured from SuLab reagents.

Experimental methods: Preparation of nano SiO$_2$ particles: Silica nano particle was prepared by drop-wise addition (~ 2 drops/s) of 2.5% HCl in diluted sodium meta silicate (10gm in 1000ml water) solution with constant stirring for 250 rpm at 60°C until a cloudy, viscous gel was formed. 1gm of silica gel in 100ml water was stirred continuously for 250 rpm at 60°C until a cloudy, viscous gel was formed. 1gm of silica gel in 100ml water was stirred continuously for 250 rpm at 60°C with drop-wise addition (~ 2 drops/s) of 2% HF, until a clear solution obtained. The solution was then filtered using Watt Man filter paper no 1. The filtrate was then evaporated to dryness resulted in white powder which was thoroughly washed with distilled water and dried in oven at 80°C. The process was repeated 4 to 5 times. The product was then dried in oven at 100°C for more than 24 hours, and calcinations in air at 100°C for 1 hour.

Characterization of nano SiO$_2$ particles: The size and shape of prepared nanoparticles was analysed on Static Light Scattering (MiniDawn, ASTRA5.3.2, Wyatt Technology Corporation, France), observed on scanning electron microscope (SEM) instrument (Model JSM5610LV, version 1.0, Jeol, Japan) and the presence of silica was confirmed by FTIR Spectroscopy (Nicolet is10 FT-IR Spectrometer, Thermo Scientific, Japan).

Results and Discussion
After the optimization of synthesis process the further experiment were carried out using sodium meta silicate as precursor and their size has been reduced by using HF. The as
synthesized particles have been characterized for their size, shape and their elemental analysis.

**Size and shape of silica nano particles: Size by static light scattering:** The shape size and size distribution of the prepared silica particles was recorded using static light scattering instrument. Figure 1 and table 1 shows the result of geometric radius and size of silica nanoparticles dispersed in water medium. The geometric radius of silica nanoparticle was shown in form of R avg. of the tested sample. The average diameter of silica nano particle was found 176 nm prepared by sol-gel technique with the use of HF.

**Table-1**

<table>
<thead>
<tr>
<th>Rms radius moments(nm)</th>
<th>Geometric radius moments(nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rn (num. avg.mean)</td>
<td>n/a Rn n/a</td>
</tr>
<tr>
<td>Rw (wt.avg.mean)</td>
<td>n/a Rw n/a</td>
</tr>
<tr>
<td>Rz (z.avg.mean)</td>
<td>n/a Rz n/a</td>
</tr>
<tr>
<td>R(avg)</td>
<td>R(avg) 88.0 ± 0.1 nm</td>
</tr>
</tbody>
</table>

**Figure-1**

Statistical particle size analysis by static light scattering

**Shape by scanning electron microscopy:** Scanning electron micrographs of silica nanoparticle deposited on carbon coated aluminium sheet were shown in figure 2. From the scale given in photograph, the average size of prepared silica nanoparticles were about 100-180 nm, which was in good agreement with the size determined by the static light scattering instrument. It can also be seen that the shape of synthesized silica particles were spherical.

**Figure-2**

Scanning electron microphotographs of silica nano particle
Infrared spectral analysis of silica nano particles: IR Spectra of silica nano particle after calcinations at 800°C temperature is shown in figure 3. From the figure two main characteristic peaks of Si-O-Si bonds vibration modes were observed around 800 & 1086 cm⁻¹, which are attributed to Si-O bending vibration band and Si-O-Si antisymmetric stretching vibration band respectively. The broad peaks between 3000 to 4000 cm⁻¹ is characteristic to the OH group, which exists in water.

Elemental analysis of silica nano particles: Elemental analyses of silica nanoparticles were performed in the scanning electron microscope using oxford-Inca software. The result observed in this test is shown in figure 4. Presence of silica is confirmed by the elemental analysis curve. The presences of oxygen in results (figure 4 and table 2) indicate that the silica is in the form of oxide or dioxide.

![Figure-3](image1)

**Figure-3**

**IR Characteristic spectra of silica nano particles**

![Figure-4](image2)

**Figure-4**

**Elemental analysis of silica nano particle by Oxford-Inca software on scanning electron microscope**

**Table-2**

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O K</td>
<td>51.03</td>
<td>61.90</td>
</tr>
<tr>
<td>F K</td>
<td>12.91</td>
<td>13.18</td>
</tr>
<tr>
<td>Si K</td>
<td>36.06</td>
<td>24.92</td>
</tr>
</tbody>
</table>
Conclusion
The silica nano particles with a diameter ~180 nm and regular spherical structure are synthesized successfully by economical and easy acid leaching technique, using the sodium metasilicate as a precursor. The R-avg. size of the synthesized silica nano particles is found to be 100-180nm. The SEM image indicates that silica nanoparticles produced are of spherical shape. The FTIR spectrum of the powder confirms the presence of silica; EDS (by oxford-Inca software) results further confirm the existence of silica in the synthesized powder.

References