Research Journal of Chemical Sciences _____ ISSN 2231-606X
Vol. **4(5),** 52-55, May **(2014)** *Res.J. Chem. Sci.*

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

PatelB.H.* **and Patel P.N.**
Department of Textile Chemistry, Faculty of Technology and Engineering, The M S University of Baroda, Vadodara 390 001, INDIA

Available online at: www.isca.in, www.isca.me
Received 10thMarch2014, revised25thApril 2014, accepted15thMay 2014

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[1,2]. In European Commission 2004[3], 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[4]. 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[5,6]. Gradually various methods i.e. aerosol, combustion flame, laser ablation, chemical vapor condensation, spray pyrolysis, electrospray and plasma spray[7-15]. Additional techniques reported for the synthesis of silica nanoparticle are sonochemical, cavitations and microemulsion processing and also high-energy ball milling[16-20]. Majority of these processes for the synthesis of nano particles are criticized for high energy consumption and process complicity[21-25]. In the present work an attempt has been made to synthesized SiO_2 nanoparticles by the leaching of sodium meta-silicate ($Na_20_3Si.9H_2O$) 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 elementally analyzed confirmed by Oxford-Inca software.

Material and Methods

Materials: Chemicals: Sodium meta silicate ($Na_20_3Si.9H_2O$, 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 ($CH_2CHCO\ NH_2$, 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 with drop-wise addition (~ 2 drops/s) of 2% HF, until a clear solution obtained. The solution was than 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 1000°C for 1 hour.

Characterization of nano SiO_2 particles: The size and shape of prepared nanoparticleswas 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 1shows 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
Results of particle size analysis

Rms radius moments(nm)		Geometric radius moments(nm)	
Rn (num. avg.mean)	n/a	Rn	n/a
Rw (wt.avg.mean)	n/a	Rw	n/a
Rz (z.avg.mean)	n/a	Rz	n/a
R(avg)		R(avg)	88.0 ± 0.1 nm

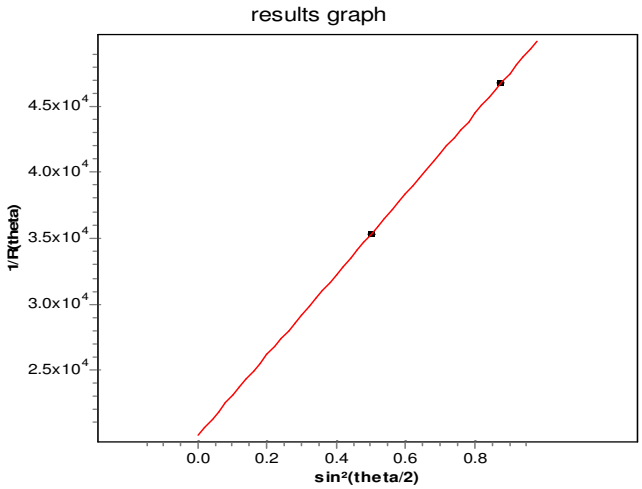

Figure-1
Statistical particle size analysis by static light scattering

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.

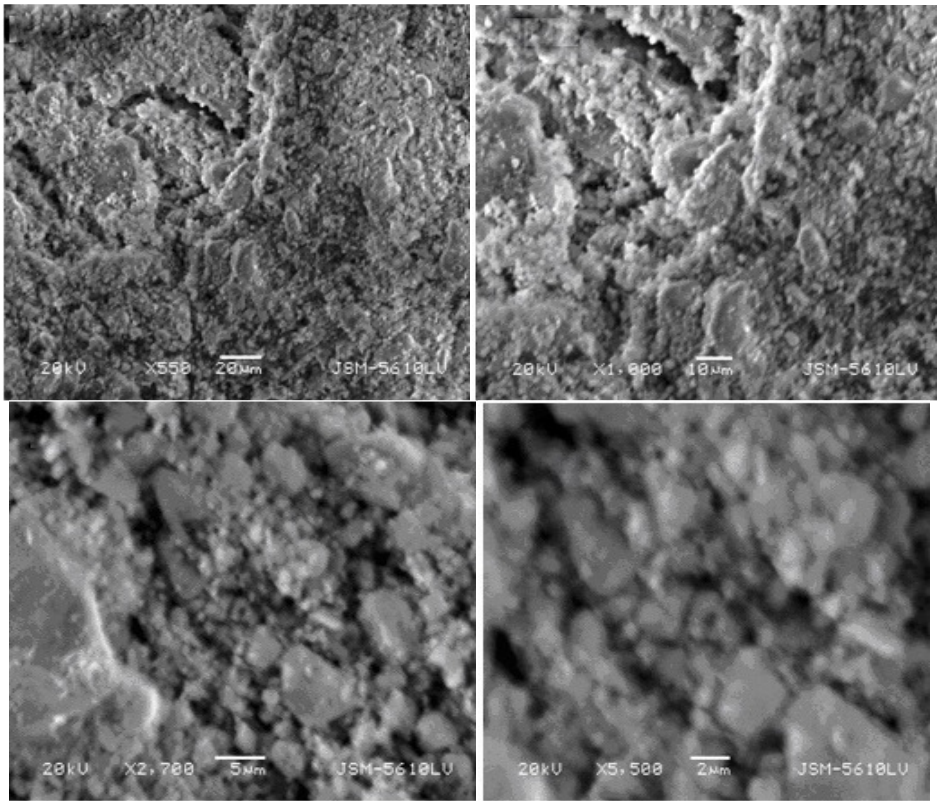

Figure-2
Scanning electron microphotographs of silica nano particle

Infrared spectral analysis of silica nano particles: IR Spectra of silica nano particle after calcinations at 800^{O}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.

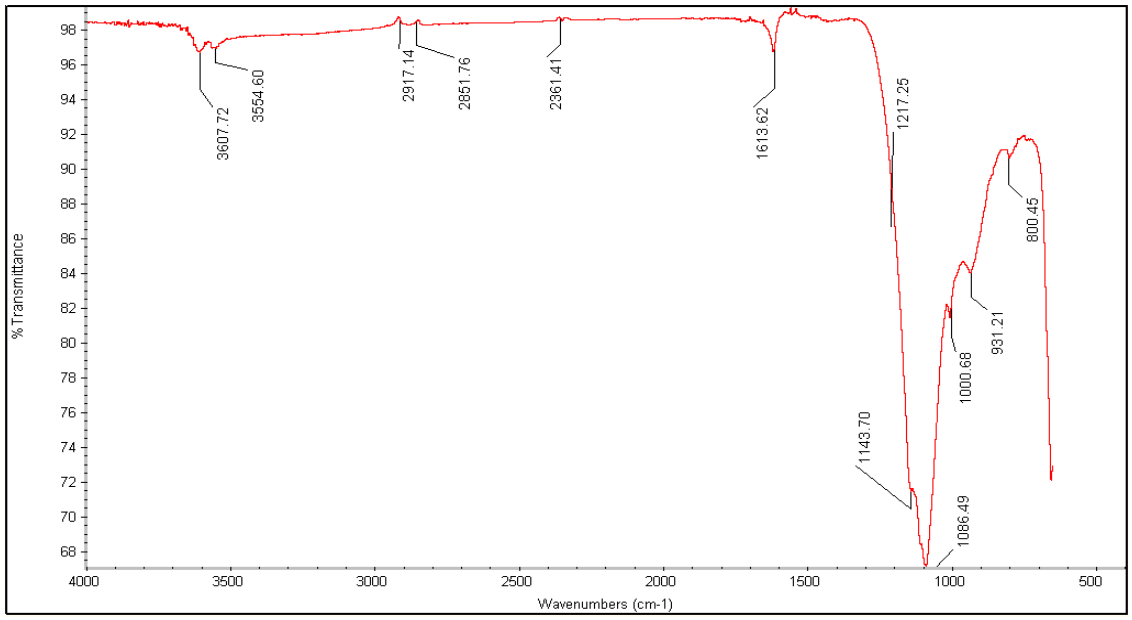

Figure-3
IR Characteristic spectra of silica nano particles

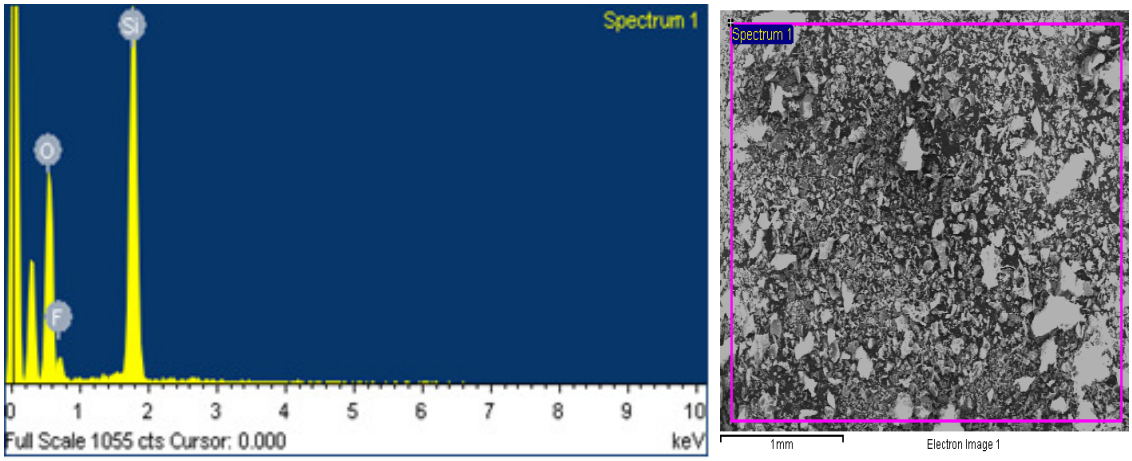

Figure-4
Elemental analysis of silica nano particle by Oxford-Inca software on scanning electron microscope

Table-2
Results of elemental analysis of silica nano particles

Element	Weight%	Atomic%
O K	51.O3	61.90
F K	12.91	13.18
Si K	36.O6	24.92

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 meta silicate as a precursor.The R-avg. size of the synthesized silica nano particle 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

1. PatelB.H. and Patel P.N., Synthesis and application of nano-sized SiO_2 to textiles: a review, *International Dyer,* May, 35-39, **(2012)**

2. Patel B.H. and Chattopadhyay D.P., Nano-particles & their uses in textiles, *The Indian Textile Journal*, **118**, 23-31 **(2007)**

3. European Commission (EC), Nanotechnology: Innovation for tomorrow's world EUR 21151EN 2004, Available: www.cordis.lu/nanotechnology**(2004)**

4. Breggin L. and Pendergrass J., Where does the nano go? End of life regulation of nanotechnologies. Woodrow Wilson International Center for Scholars,*Project on Emerging Nanotechnologies*, PEN 10, July (**2007)**

5. Gleiter H., Nanocrystalline materials, *Prog. Mater. Sci.* **33**, 223–315 **(1989)**

6. Siegel R.W., Nanomaterials: synthesis, properties and applications, *Ann.Rev. Mater. Sci.*, **21**, 559 **(1991)**

7. Uyeda R., Crystallography, methods of preparation and technological applications, *Prog. Mater. Sci.*, **35**, 1-96 **(1991)**

8. Friedlander S.K., Synthesis of nanoparticles and their agglomerates: Aerosol reactors,*In Rand D status and trends*, ed. Siegel et al., **(1998)**

9. Information from website; http://www.wtec.org/loyola/nano/02_04.htm**(2004)**

10. Rao et al, Nanostructured materials production by hypersonic plasma particle Deposition, *Analytical Chemistry*, **69**, 471-477 **(1997)**

11. Becker et al, Metal nanoparticles generated by laser ablation, *Nanostruct. Mater.,***10**, 853 **(1998)**

12. Kear B.H., Sadangi R.K. and Liao S.C., Synthesis of WC/Co/diamond nanocomposites, *In Proc. of the Joint NSF-NIST Conf. on Nanoparticles***(1997)**

13. Messing G.L., Zhang S. Selvaraj U. Santoro R.J. and Ni T., Synthesis of composite particles by spray pyrolysis, In Proc. of the Joint NSF-NIST Conf. on Ultrafine Particle Engineering,May 25-27, Arlington, VA, **(1994)**

14. Mora et al, Electrospray atomizers and ultrafine particles, In Proc. Joint NSF-NIST Conf. on UltraFineParticle Engineering, May 25-27, 1994, Arlington, VA, **(1994)**

15. Berndt C.C., Karthikeyan J., Chraska T. and King A.H., Plasma spray synthesis of nanozirconia powder, In Proc. of the Joint NSF-NIST Conf. on Nanoparticles, **(1997)**

16. ChaudhariS.B., Mandot A.A. and PatelB.H., Functionalized nano-finishing to textiles using Ag nano-colloids, *Melliand International*, **15(5-6),** 214-216 **(2009)**

17. ChaudhariS.B., Mandot A.A. and PatelB.H., Effect of nano TiO_2 pretreatment on functional properties of cotton fabric, *Int. Jr. of Engg. Res. & Dev.*, **1(9),** 24-29 **(2012)**

18. Mandot A.A., ChaudhariS.B. and PatelB.H., Nanocomposite: Manufacturing and applications in textiles, *Melliand International*, **18(3),** 188-189 **(2012)**

19. Bhattacharya S.S. and ChaudhariS.B., Change in physico-mechanical and thermal properties of polyamide / silica nanocomposite film, *Int. Jr. of Engg. Res. & Dev.*, **7(6),** 1-5 **(2013)**

20. Chattopadhyay D.P. and Patel B.H., Modification of Cotton Textiles with Nanostructural Zinc Particles, *Journal of Natural Fibers*, **8(1),** 39–47 **(2011)**

21. Ozuomba J.O. and Ekpunobi A.J., Sol-Gel Derived Carbon Electrode for Dye-Sensitized Solar Cells,*Res.J.chem.sci.*,**1(8)**, 72-75**(2011)**

22. Kumar Harish, Rani Renu and Salar Raj Kumar, Synthesis of Nickel Hydroxide Nanoparticles by Reverse Micelle Method and its Antimicrobial Activity,*Res.J.chem.sci.,***1(9)**, 42-48**(2011)**

23. Pawar M.J., Nimbalkar V.B., Synthesis and phenol degradation activity of Zn and Cr doped TiO_2 Nanoparticles,*Res.J.chem.sci.,* **2(1),** 32-37**(2012)**

24. Bansal P., Jaggi N. and Rohilla S.K., "Green" Synthesis of CdS nanoparticles and effect of capping agent concentration on crystallite size, *Res.J.chem.sci.,* **2(8)**, 69-71**(2012)**

25. Samira Bagheri, Chandrappa K.G. and Sharifah Bee Abd Hamid, Generation of Hematite Nanoparticles via Sol-Gel Method, *Res. J. Chem. Sci.***, 3(7)**,62-68**(2013)**