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CHARACTERIZATION OF SILICA NANO-PARTICLES SYNTHESIZED BY THERMO-MECHANICAL ROUTE

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ABSTRACT

Ball milling is a modest and valuable processing technique that is employed in the production of nanocrystals and nanoparticles. Thesilica (SiO_2) nanoparticles were synthesized from colloidal silica via thermos mechanical route method using ball mill. The parameters, which are affecting the milling process has been thoroughly calculated such as the milling period and the initial size of silicaparticles. The results indicated that the silicananoparticle with particle size 30 nm-50 nm could be successfully prepared from crystal silica particles of size 0.5-2.5 mm. To obtain these silica nanoparticles, 25 gm silicaparticles, 50 ml of toluene and 15 hours of milling at 300 rpm were used. The silica nanoparticles were characterized by HR-SEM (High Resolution Scanning Electron Microscopy), XRD (X-Ray Diffraction) and EDS (Energy Dispersive Spectroscopy). The nature of silica nanoparticles was evaluated using XRD and EDS. The morphology of particles and particle size was determined by HR-SEM. The results obtained showed semispherical (cauliflower) shape ofsilica nanoparticles.

Keywords: Ball Milling, HR-SEM, Silica Nanoparticles, Thermo-Mechanical, XRD.

I.INTRODUCTION

Nanoparticles arehaving characteristic dimension (diameter for spherical particles) of less than 100 nm in any of the dimension[1-2]. The silica (SiO_2)nanoparticles can be produced by chemical processes, precipitation processes, stober process,solid state process, hydro-thermal process, sol-gel process, ball mill process, etc.[3-4]. It is reported in [5] that chemical synthesis of silicananoparticlesproduces high contamination in the final products. The synthesis of silicananoparticles through ball mill is a "top-down" process which produces low contamination in the final product. In the "top-down" process, the energy has imparted to a course grained material to reduce the particle size. This process is also termed as mechanical alloying and it is high energy milling process in which powder particles are subjected to repeated cold welding, fracturing and re-welding[6-8]. The size and shape of silicananoparticles may be controlled by additives such as electrolytes, surfactants, organic acids, etc. [9]. Nanomaterials show exceptional physical and chemical properties, and impart boosts to engineered materials. Thesilica nanoparticles have major application in construction composites, pharmaceutics, electronics, catalysis's, additives for rubber, plastic non-toxic platforms, and in several biomedical applications (drug delivery and dental)[10-12].The particles sizes are becoming smaller with increase of milling time and speed.The method used for synthesizing the silica nanoparticles in this study is thermo-mechanical. The main precursor was colloidal silica, various processes also been used like ball milling, calcination, refluxing during the nano particle synthesis.

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II.. Materials and Method

The materials used in synthesizing silicananoparticles were colloidal silica solution (30% weight suspension in H_2O, Sigma-Aldrich), ethanol (99% purity, SRL Pvt. Ltd.), dehydrated toluene (99.5% purity, Kanto Kagaku Co., Ltd.) and Dodecyltrichlorosilane (Tokyo Chemical Industry Co., Ltd.). The silica nanoparticles were prepared by thermo-mechanical method using a ball mill. The planetary ball mill with grinding balls (stainless steel balls of diameter 6, 7, and 8 mm) were used for milling silica particles with maximum rotational speed of 300 rpm.

(a) **(b)**

Fig. 1. (a) Crystal silica (b) Silica nano powder.

The colloidal silica is dried up to 12 hours at 100ºC in oven but to remove moisture content in the sample again it is kept in furnace for 3 hours at the rate 15ºC/minuteat 600 ºC to get the dried crystal silica (0.5 mm to 2.5 mm) as shown in Fig.1(a). Further, the crystal silica was mixed with dehydrated toluene (as wet media)and milled for 15 hours to get the silica nanoparticles."Retsch-PM400" planetary ball mill and stainless steel grinding balls (AISI 304 Technocon Engineers, India) are used. The balls have variable diameter (6, 7 and 8 mm) and grinding jar with maximum rotational speed of 300 rpm. The detailed parameters used during milling process are as follows given in Table 1. It is then again mixed the sample with dodecyltrichlorosilane for reflexing to get the precipitate silica. Finally, the sample has been crushed in agate jar by hand milling and get the pure silica nanoparticle. The schematic flow diagram to synthesize silicananoparticles is given in Fig. 2.

Table 1 Parameter used during milling.

S. No.	Parameter	Value
1.	Balls/powder weight ratio	6:1
2.	Balls diameter (mm)	6, 7 and 8
3.	Atmosphere	Air
4.	Grinding medium	Wet (toluene)
5.	Mass of ball (gm)	150
6.	Milling time	15 hours (pause mode every 1.5 hours)
7.	Type of mill	Dual drive planetary mill

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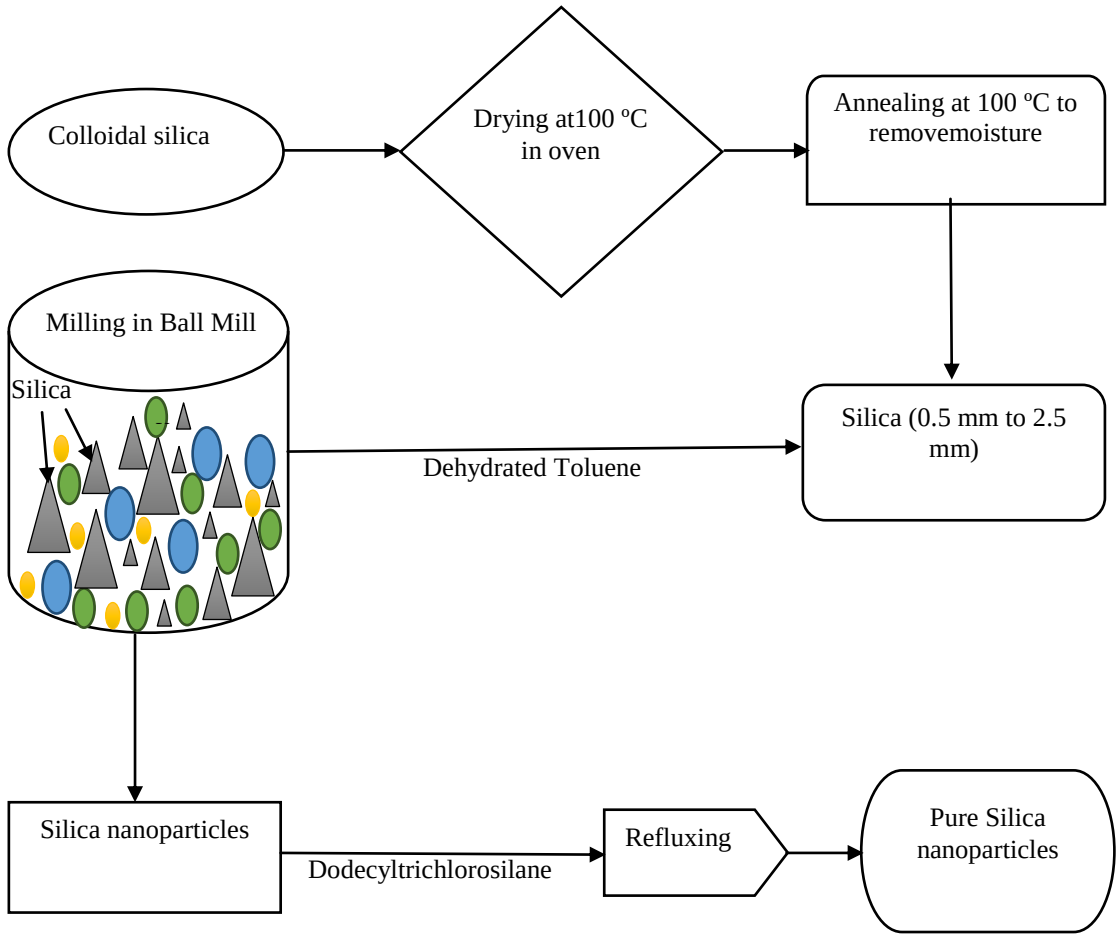

Fig. 2. Flowchart for synthesizing pure silica nanoparticles.

III. CHARACTERIZATION OF SILICA NANOPARTICLES

Crystallite size and morphology ofsilicananoparticle have been investigated through XRD and HR-SEM,respectively. XRD pattern shows single phase silica amorphous nanoparticles. The XRD results have been compared with Maud analysis which confirms the crystallite size and lattice parameter of silica nanoparticles. HR-SEM confirms the semi-spherical shape(cauliflower) having some agglomeration. The elemental analysis has also been done by using EDS unit.

3.1 X-ray Diffraction (XRD)Analysis

The crystallinity of particles was determined by X-ray diffraction (XRD) "Shimadzu, Japan XRD- 7000". XRD analysis is the most useful technique for identification of crystalline structure. The X-ray pattern of silica nanoparticles is shown in Fig. 3(a) Which confirms that the powder isamorphous silica with few impurities. The highest peak ofamorphous silica nanoparticle at 21.54º angle has been observed with a wide range of angle 2θ = 21º to 24º.The silicananoparticles characteristic with crystallite size of 33.45 nm. These XRD resultsalso indicates that the synthesis of silica nanoparticles by thermo-mechanical method is significant. Maud analysis Fig. 3(b) has also been done for confirming the result obtained from X-Ray analysis. Rietveld analysis confirms the crystallite size and comparison has been shown in Table 2.

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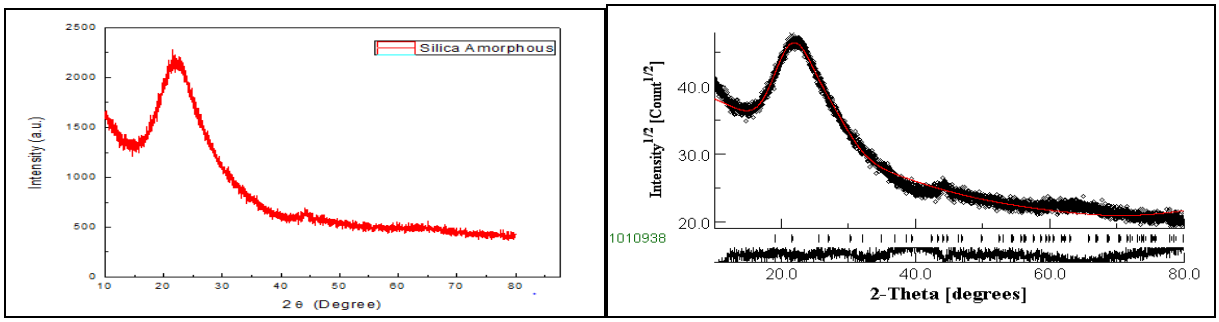


(a) **(b)**

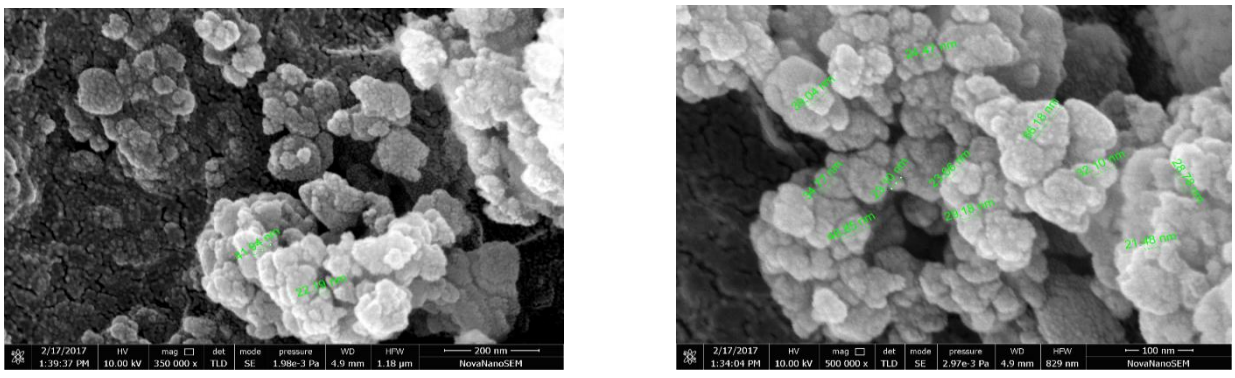
Fig. 3(a) XRD result (b) Maud analysis.

Table 2 Comparison of results obtained from XRD and Maud analysis.

Analysis	Crystallite size
XRD Results	33.45 nm
Maud Results	40.92 nm

3.2 High Resolution Scanning Electron Microscopy(HR-SEM)

The surface morphology and size of nanoparticles wereaccessed byHR-SEM "SUPRA 40, ZEISS". The samples were Au sputtering prior to the observation. The SEMX as shown in Figs. 3 (a-b). silica nanoparticles were observed in the size range 22-42 nm (Fig 3 a) and 22-47 nm (Fig 3 b) for above magnification range respectively. The HR-SEM micrograph indicates that the prepared SiO_2nanoparticles are irregular in shape and size. The morphology of silica nanoparticles is semi-spherical(cauliflower) in the sample. The HR-SEM analysis has revealed that the average particle size is 38 nm.



(a) (b)

Fig. 3 (a) HR-SEM analysis having magnification range of 350000 X and (b) HR-SEM analysis having magnification range of 500000 X.

3.3 EnergyDispersive Spectroscopy (EDS)

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The elemental analysis was achieved by HR-SEM with EDS unit. Thedata obtained from EDS analysis and EDS graph of silica nanoparticles are shown in Table 3 and Fig.4. The EDAX spectra of milled silica having four elements as Silicon (Si), Oxygen (O), some low percentage of Carbon (C) and Gold (Au). The SiO_2 was confirmed by EDAX analysis of thesilica nanoparticle sample. There is no, other contamination at 15 hours milled silica powders. More importantly ball milling is performed under atmosphere condition only at longer time, so that the EDAX shows the Oxygen atomic percentages twice that of silicon atomics percentage. The data confirmed the presence of silicon and oxygen in the sample.

Table 3 Data obtained from EDS analysis

S. No.	Element	Weight%	Atomic%
1.	C	10.31	13.90
2.	O	54.21	56.20
3.	Si	27.80	28.64
4.	Au	7.68	1.26

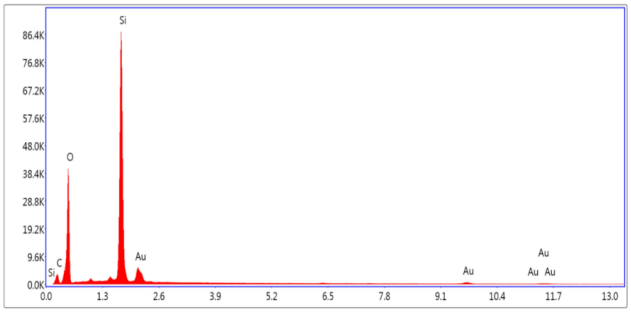

Fig .4 EDS of Silica nanoparticle.

V. CONCLUSION

Silica nanoparticles were synthesized by thermo-mechanical route and has been confirmed by XRD patterns which shows amorphous silica nanoparticles. The XRD results also showed that the crystallite size of amorphous silica is 33.45 nm which has also been confirmed by Reitveld analysis of Maud software. The HR-SEM shows the morphology of amorphous silica nanoparticles were semi-spherical (cauliflower) with an average particle size of 38 nm. The elemental analysis by HR-SEM with EDS has confirmed that the silica is the major part in the sample with very small contamination of Carbon also.

VI.ACKNOWLEDGEMENT

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