Synthesis of Silica Nanoparticles from Malpe Beach Sand using Planetary Ball Mill Method

Radhip.N.R*1, Pradeep.N1, Abhishek Appaji M2 and S.Varadharajaperumal3

1Department of Nano Science and Technology, Mount Carmel College, Bengaluru, India.
2Department of Medical Electronics, BMS College of Engineering, Bengaluru, India.
3Center for Nano Science and Engineering, Indian Institute of Science, Bengaluru, India.

(Received on: June 8, 2015)

ABSTRACT

Silica nanoparticles have been prepared by planetary ball mill method at room temperature at different milling hours using Malpe beach sand, Karnataka, India. The result occurred in 150 hours milling time at 250 RPM to produce silica nanoparticle less than 100 nm in size. The synthesized Nanoparticles were characterized by Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Analysis (EDAX), X-Ray Diffraction (XRD). The XRD results confirmed amorphous of silica Nanoparticles. The morphology of all the samples was studied by Scanning Electron microscopy. The EDAX results was measured the atomic percentage of element Silicon and Oxygen and Aluminum and potassium.

Keywords: Silica Nanoparticles, planetary Ball mill, Malpe Sand.

1. INTRODUCTION

Nano Science and technology is an interdisciplinary field of Research including chemistry, microbiology, biotechnology, life science, engineering, food and nutrient, medical engineering and more. Sand particle diameter is affected by increasing ball milling time. Particle size decreases with increase the milling time. Silica nanoparticles are processed by top-down approach method from breaking up the bulk materials. Top-down approach is typically a ball milling process. The bigger particles are broken into very small particles to reach nano (10⁻⁹) scale. This ball milling process also called mechanical alloying which is
high energy milling process in which powder particles are subjected to repeated cold welding, fracturing and rewelding, the transfer of mechanical energy to the powder particles results in introduction of strain into the powder through generation of dislocations other defects which act as fast diffusion paths. During milling powder temperature occurs. All these effects lead to alloying of the blended elemental powders during the milling process\textsuperscript{1-3}. Silica also extracted from Rice Husk ash for drug Application\textsuperscript{4}, Lithium-ion Battery anodes\textsuperscript{5} and DNA and microarray detection\textsuperscript{6}. Silica nanoparticle can be prepared by many techniques such as sol-gel process\textsuperscript{7} novel method\textsuperscript{8} wet chemical synthesis\textsuperscript{9} and stober silica\textsuperscript{10}.

The objective of this research is to use a technique of transforming natural sand to sand silica nanoparticle by using a planetary ball milling with room temperature. So far very few researchers derived the silica nanoparticle from sand; literature survey reveals the milled sands from Indonesia and Egypt so far. According to our knowledge until now no one is derived silica nanoparticle from Indian sand especially from malpe beach sand from Karnataka, India, absence of applying additional heat and chemicals. The first comparative study on the different milling times of malpe beach sand. Such a new method synthesis of silica nanoparticle from Indian sand has not yet been reported. In this technique we synthesize Silicon nanoparticle from malpe beach sand and characterized by SEM, EDAX, and XRD.

2. MATERIALS AND METHODS

2.1 Preparation of silica Nanoparticles

Silica nanoparticles were prepared by ball milling method The planetary ball mill grinding jars and grinding balls used are from stainless steel with diameter of 10 mm with maximum Rotational speed of 250 RPM.

![Fig 1. Natural Sand before ball milling](image)
Figure 2 shows the malpe beach sand before ball milling. Sand was taken from Malpe beach, Karnataka, India. The sand was first being washed to remove impurities and dried in room temperature for 1 hour. Then, the sand size was measured using vernier to a size the range between 1mm to 3 mm. The sand was then pouring into the grinding jar with grinding balls and it’s milled for 50, 75,100,125,150 hours.

3. RESULT AND DISCUSSION

3.1 X-Ray Diffraction (XRD)

An x-ray diffraction pattern shows the crystalline structure the SiO$_2$ nano powder prepared by malpe sand. Different milling time was used at 50, 75,100,125,150 hours. The peaks are observed in XRD orientation (100), (101), (220), (110), (102), (111), (200), (201), (112), (003), (202), (211), (113), (203), (104), (302), (220), (213).These above orientation indicate the milled powder is SiO$_2$ material. The peaks in the diffractogram were in agreement with of silicon dioxide as given in the JCPDS database and relevant literature. Typical silicon dioxide peak is observed. The average crystallite size was calculated from the full width at half maximum (FWHM) of the X-ray diffraction peak using Scherer’s equation (1) shown below.

$$D=0.9\lambda/W \cos\theta$$  \hspace{1cm} (1)

The index peak orientation of 101 at 26° of two theta was taken for crystallite size calculation. Compared to the XRD pattern of 50 hours milled powders to 150 milled powder shows broadened peak intensity which means the powder size is effectively reduced by milling. All the peaks are sharp indicates that the powder is polycrystalline nature no indication of the amorphous structure. The crystalline size respect to the milling time shown in Table: 1, during ball milling the intense mechanical deformation experienced by the powder leads to formation of strains. Figure: 4 shows the variation of lattice strain and crystallite size of the silicon dioxide powder for different milling time. A linear reduction in crystallite size is observed along with the milling time when milling hours is increased particle size is decreased. The lattice strain is increasing with increasing of milling time. This lattice strain was increased from 0.0022 to 0.0037 respectively with milling time of 50hours to 150hours.

3.2 Scanning Electron Microscope (SEM)

The SEM was used to follow the changes in the morphology of the ball milled sand powder. Figure: 4. The SEM micrograph indicates the prepared silica powder is irregular in shape and size. The morphology is rough in all the samples. The particle sizes were between few nm to few hundred nanometers in all samples. The particle sizes are become smaller with increase of milling time. At 50 hours sample, shows the different sizes of particles in nanometer range this indicates the milling is not enough to get nanoparticle. 100 hours shows
the fine particles along with the few nanometer size particles. The particle size was fine and smooth in 125 hours comparable with 100 hours milled powder. At this stage the particle were finely grinded it’s clearly seen that at 150 hours milled powder shown agglomeration of particle with fine particles. However, the samples prepared with ball milling shows particles size less than 500 nm.

![Figure 2. X-Ray Spectrograph of Malpe sand with different milling times](image)

Table 1.

<table>
<thead>
<tr>
<th>Milling (Hours)</th>
<th>Crystallite size (nm)</th>
<th>Lattice strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>71</td>
<td>0.0022</td>
</tr>
<tr>
<td>75</td>
<td>50</td>
<td>0.0031</td>
</tr>
<tr>
<td>100</td>
<td>47</td>
<td>0.0033</td>
</tr>
<tr>
<td>125</td>
<td>44</td>
<td>0.0035</td>
</tr>
<tr>
<td>150</td>
<td>42</td>
<td>0.0037</td>
</tr>
</tbody>
</table>

![Figure 3. Graph of Crystallite size and Lattice strain milling hours](image)

3.3 Energy Dispersive X-Ray Analysis (EDX)

Figure 5 shows EDAX spectra of different milling times of maple beach sand Silicon (Si) and Oxygen (O) and with low percentage of Aluminum (Al) and Potassium (K). The SiO$_2$ was confirmed by EDAX analysis of the malpe beach sand. The Al and K are natural ingredients for sands. There is no, other contamination at 150 hours milled sand 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 all samples.
Fig 4. Scanning Electron Microscope Images of Different hours ball milled sand
(a) 50hours, (b) 75hours, (c) 100hours,
(d) 125hours, (e) 150hours.
Fig 5. EDAX Images of Different hours ball milled sand
(a) 50hours, (b) 75hours, (c) 100hours
(d) 125hours (e) 150hours
4. CONCLUSIONS

The silica nanoparticles with a diameter ~100 nm and regular spherical structure are synthesized successfully by economical and easy ball mill technique form malpe beach sand. From the XRD data is confirmed the SiO$_2$ nanoparticle. The SEM images indicate that silica nanoparticle rough and different shape of Morphology. The EDAX data shows the percentage of Si, O$_2$, Al, K nearly all the samples.

Acknowledgement

The work reported in this paper is supported by B M S College of Engineering, Bengaluru through Centre of Excellence in Advanced Materials Research, TECHNICAL EDUCATION QUALITY IMPROVEMENT PROGRAMME [TEQIP-II] of the MHRD, Government of India.

REFERENCE

5. Mingyuan Ge1, Jiepeng Rong, Xin Fang, Anyi Zhang, Yunhao Lu, and Chongwu Zhou, Scalable preparation of porous silicon nanoparticles and their application for lithium-ion Battery anodes, Nano Research 2013, 6(3) :174-181.
10. Ismail A.M.Ibrahim, A.A.F.Zikry, Mohamed A.sharaf, “preparation of spherical silica