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Optical and Photoluminescence Studies of Synthetic Quartz Nanoparticles Prepared by High Energy Planetary Ball Milling Technique

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Abstract— *In the present study, synthetic quartz nanoparticles were prepared by mechanical media milling process in a high energy planetary ball mill using micron sized synthetic quartz as starting material, acetone as medium and stearic acid as surfactant. Additionally tungsten carbide balls were used as milling media at 350 rpm with BPR (ball to powder weight ratio) 10:1 for total duration of 48 hours. The sampling was carried out at 6, 12, 24 and 48 hours. The collected samples were washed with diluted HCL followed by distilled water and methanol to remove impurities and dried in oven at 50°C for 4 hours. The prepared synthetic quartz nanoparticles were characterized by particle size analyzer, scanning electron microscopy, X-ray diffraction, UV-visible spectrophotometry and fluorescence spectrophotometry. The crystalline size of prepared nano-synthetic quartz was found to be about 39.41 nm. In UV-spectrophotometric characterization of the synthetic quartz nanoparticles, the optical band gap was observed in the range of 4.4-5.1 eV. In photoluminescence (PL) analysis, five strong and broad emission peaks were detected at 240 nm, 295 nm, 448 nm, 480 nm and 665 nm. The PL spectra of the samples showed blue light emission in near blue region using UV-excitation wavelength at 220 nm. The obtained results can be further used for dosimetric application of synthetic quartz.*

Keywords— *Synthetic quartz nanoparticles, Photoluminescence, UV-visible spectroscopy.*

1. INTRODUCTION

Materials in nanometer size exhibit a remarkable amount of variation in electronic, magnetic, optical and chemical properties of a molecule that are significantly different from those of the bulk.[1-2] The desirability criteria of a nanomaterials are its submicron size, narrow size distribution, high level of dispersability and low extent of agglomeration.[3] Various methods have been used for preparation of ceramic or metal nanoparticles such as mechanical alloying, combustion synthesis, plasma forming, explosive forming, electro deposition, sol-gel technique and media milling/ball milling process.[1, 4-5] Among these methods, high energy ball milling (BM) is favourable technique widely used in production of nano particles due its simplicity, user friendly operation, low cost of production and applicability to any class of materials at large quantities.[6] Several workers have used BM method for the synthesis of nano-particles of different martials like Fe_2O_3-SnO_2, ZrO_2-Fe_2O_3, TiO_2-Fe_2O_3, TiO_2-WO_3, SiC, Si_3N_4 and quartz (SiO_2).[7-10] Literature revealed that nanoparticles of natural quartz have been prepared and their optical properties have been studied, [11-13] but no research work have been carried out on preparation of synthetic quartz nanoparticles and its optical properties. Natural quartz is a brittle, hard, water-resistant and chemically stable material with twins and other imperfections whereas laboratory grown synthetic quartz is pure, without any twins and impurities. Therefore, synthetic quartz is used for the optical studies under different physical conditions for dosimetric applications.

In present article, synthetic quartz nano-particles have been prepared by high energy planetary BM process for different durations. The characterization of nano-synthetic quartz material was carried out by particle size analyzer, XRD and SEM. The optical properties were studied by UV-Visible and photoluminescence (PL) analysis.

2. EXPERIMENTAL DETAILS

2.1 Sample Preparation

Synthetic quartz nanoparticles were prepared by a high energy planetary BM (FRITSCH Planetary Mono Mill PULVERISETTE 6 Classic Line, Germany) process using micron sized synthetic quartz as starting material, acetone as dispersing medium and stearic acid as surfactant. Additionally tungsten carbide balls were used as milling media at 350 rpm with BPR (ball to powder weight ratio) 10:1 for total duration of 48 hours. The sampling was carried out at 6 (SQ 1), 12 (SQ 2), 24 (SQ 3) and 48 (SQ 4) hours. The collected samples were washed with diluted HCL followed by distilled water and methanol to remove process impurities and dried in oven at 50°C for 4 hours. The dried samples were used for further investigations.

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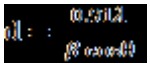
2.2 Characterization Techniques

Particle Size Analyzer (Malvern Zetasizer Nano ZS 90, Malvern Instruments, UK) was used for particle size measurement of prepared samples (SQ 1, SQ 2, SQ 3 and SQ 4). It works using a process called Dynamic Light Scattering (DLS) for size measurements. Dynamic Light Scattering measures Brownian motion and relates this to the average size of the particles.

The morphology of the prepared synthetic quartz nano-particles (SQ 1, SQ 2, SQ 3 and SQ 4) was observed using a Scanning Electron Microscope 205 (SEM, JSM-5610, JEOL Ltd. Tokyo, Japan). The sample was mounted directly onto the SEM sample holder using double-sided sticking carbon tape and images were recorded at the required magnification at the acceleration voltage of 15 kV.

The SQ 4 sample was further characterized by X-ray diffraction (XRD) using a X-ray Diffractometer (XRD, X-Pert-PRO, PANalytical, Netherland) with CuKα, operating at 45kV and 40 mA. The crystallite size of the milled powder was determined by X-ray line broadening and calculated using the Scherrer equation [1]



(1)

Where d is the mean grain size, β is the full width at half maximum (FWHM). θ is the angle of the peak maximum, and λ (0.15406nm) is the Cu(Kα) wavelength.

UV-Visible-double beam spectrometer (UV-1800 Shimadzu, Japan) with fixed slit width (2nm) was used for all absorbance measurements of prepared nanoparticles (SQ 1, SQ 2, SQ 3 and SQ 4) using 1.0 cm matched quartz cells.

PL emission spectra of prepared nanosized synthetic quartz samples were measured with excitation wavelength at 220nm using Spectrofluorimeter (RF-5301 Shimadzu, Japan).

3. RESULT AND DISCUSSIONS

The average particle size of SQ 1, SQ 2, SQ 3 and SQ 4 were measured by using particle size analyser and it has been observed that the average particle size of synthetic quartz decrease with increase in milling time. The Variation in particle size of synthetic quartz with milling time is depicted in table 1.

The SEM images of SQ 1, SQ 2, SQ 3 and SQ 4 samples are presented in Figure 1(A), 1(B), 1(C) and 1(D) respectively. As per the images it can be observed that the particles are agglomerated and the shapes of the particles are irregular. The actual size cannot be determined by SEM due to limitations of resolutions of the instrument but it can be easily predicted that all the samples are in nano-metric range.

Table 1: average particle size and band gap energy analysis of synthetic quartz nanoparticles.

Samples	*Milling time (hours)*	*Avg. particle Size (nm)*	*Band gap energy (eV)*
SQ 1	6	490	5.10
SQ 2	12	422	4.80
SQ 3	24	133	4.65
SQ 4	48	87	4.40

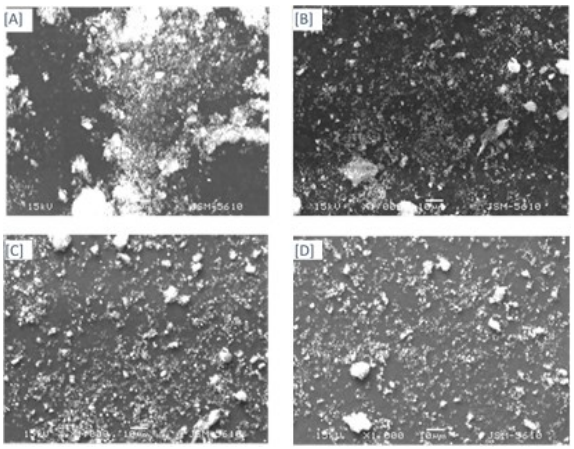


Fig. 1: The Scanning Electron microscope photograph of synthetic quartz nanoparticles at 1000X magnification. (A) SEM image of SQ 1, (B) SEM image of SQ 2, (C) SEM image of SQ 3, (D) SEM image of SQ 4.

Figure 2 shows the XRD pattern of the prepared synthetic quartz sample milled for 48 hours (SQ 4). The crystalline size of SQ 4 was calculated using Debye Scherer formula and was found 39.41nm.

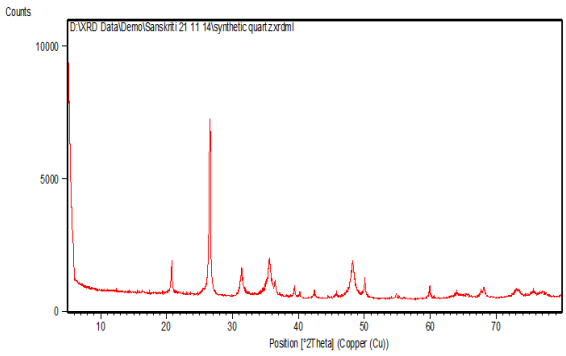


Fig. 2: XRD pattern of 48 hours milled synthetic quartz nanoparticles (SQ 4).

The optical absorption spectra SQ 1, SQ 2, SQ 3 and SQ 4 are shown in Figure 3. The absorption range was observed from 400 nm to 220 nm. According to theories, this absorption pattern is due to the transition or excitation of electron from the valance band to the conduction band and which can be used to calculate the value of the optical energy band gap of the prepared synthetic quartz nanoparticles.

From the absorption peak the optical energy band gaps of SQ 1, SQ 2, SQ 3 and SQ 4 have been calculated using the formula

$$(\alpha h\nu)^{1/n} = A\,(h\nu - E_g) \qquad (2)$$

Where hν is incident photon energy, α is absorption coefficient A is constant and E_g is the band gap energy of material and the exponent n depends on the type of transition.

Figure 4 shows that the band gap energy of the synthetic quartz samples is found in the range 4.4-5.1 eV. The band gap energies of SQ 1, SQ 2, SQ 3 and SQ 4 are summarized in Table1.

The PL spectra of synthetic quartz nanoparticles (SQ 1, SQ 2, SQ 3 and SQ 4) are shown in Fig. 5. In PL spectra, five strong and broad emission peaks were detected at 240 nm, 295 nm, 448 nm, 480 nm and 665 nm under UV excitation of 220 nm wavelength for all samples. PL spectra of the samples showed high intense blue light emission in near blue region at 448 nm. The band gap energies in the range of 4.4-5.1 eV also give the confirmation for emission of highest intense peak at 448 nm.

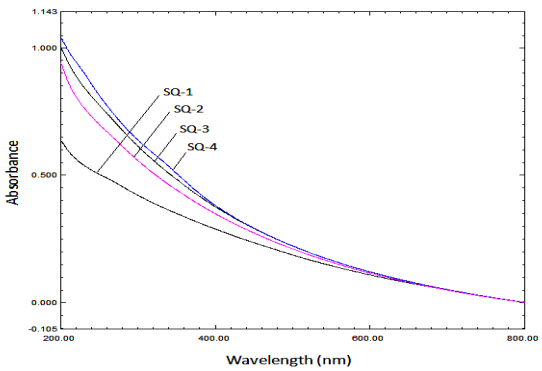


Fig. 3: Absorption spectrum of synthetic quartz nanoparticles (SQ 1, SQ 2, SQ 3 and SQ 4).

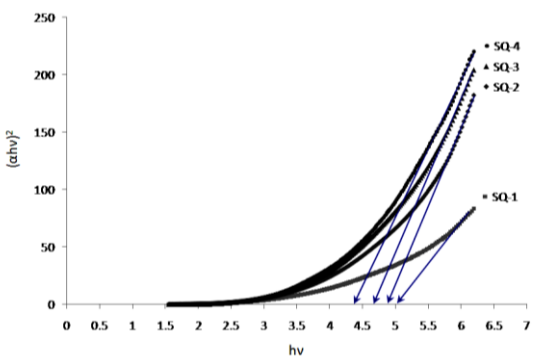


Fig. 4: Calculation of optical band gap energy for SQ 1, SQ 2, SQ 3 and SQ 4.

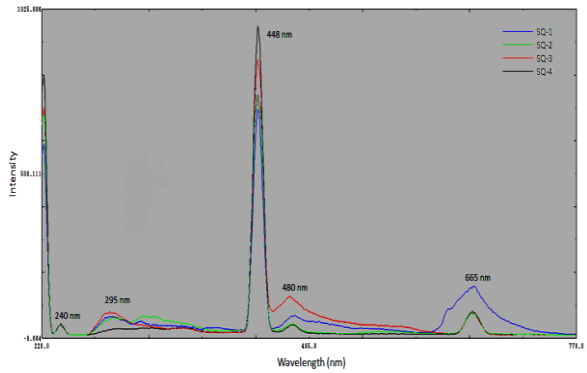


Fig. 5: PL emission from the synthetic quartz nanoparticles (SQ 1, SQ 2, SQ 3 and SQ 4).

4. CONCLUSION

Synthetic quartz nanoparticles were efficiently prepared by media milling process in a high energy planetary ball mill. The prepared synthetic quartz nanoparticles were characterized by particle size analyser, SEM and XRD and the results obtained were found in good agreement with each other. The optical properties of synthetic quartz nanoparticles were investigated by UV-Visible and PL analysis. The UV absorption pattern of prepared nanosized samples gave the optical band gap energies in the range of 4.4–5.1eV which correlates with maximum excitonic emission at 448nm near blue region in PL spectra. The obtained results can be further used for dosimetric application of synthetic quartz.

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