

# A STUDY ON CORRELATION AMONG PARTICLE SIZE, UV-ABSORPTION AND PHOTOLUMINESCENCE OF SYNTHETIC QUARTZ

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## Abstract

In this study, different micron size samples were prepared by grinding the synthetic quartz crystal using metallic mortar pestle and nano size samples were prepared by mechanical media milling process in a high energy planetary ball mill. The prepared samples were characterized by Particle size analyzer, Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), Fourier-transfer infrared spectroscopy (FTIR) and Energy Dispersive Spectroscopy (EDS). The micron and nano-sized synthetic quartz samples were further analysed using UV-visible spectrophotometry and fluorescence spectrophotometry. The impurities and defects were examined by FTIR. EDS was performed to detect any in-process impurity and to confirm that elemental composition of the prepared micron and nano sized samples. Particle size analyzer, TEM and XRD confirmed the nano size of prepared synthetic quartz samples. In UV-visible spectrophotometric study, one absorption peak was observed in all micron-size samples in 210-235nm range whereas in nano size sample, a broad absorption peak was seen in 210-480 nm range. In photoluminescence analysis, PL intensity systematically increased with decrease in particle size from 105 µm to 53 µm and reduced with further decrease in particle size from 53 µm to 39.41nm. **Keywords:** Synthetic quartz nanoparticles, UV-visible spectroscopy, Photoluminescence.

#### 1. INTRODUCTION

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. Synthetic quartz crystals also have attractive properties for development of various electronic and optical devices, where it is exposed to different optical backgrounds. In recent advancements in research and development, many applications are in control of particle size, morphology, size distribution and dispersion [1]. 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 [2]. So it is essential to study the variation in optical properties of synthetic quartz due to changes in particle size and morphology.

In present study, synthetic quartz samples in micronand nano-size have been prepared by mortar-pestle and high energy planetary BM process, respectively. The characterization of prepared synthetic quartz samples was carried out by particle size analyzer, TEM, XRD, FTIR and EDS. The optical properties were studied by UV-Visible spectrophotometric and photoluminescence (PL) analysis.

#### 2. EXPERIMENTAL DETAILS

#### 2.1 Sample Preparation

#### 2.1.1 Micro-sized sample preparation:

Micron-sized synthetic quartz samples were prepared by grinding the samples using metallic mortar-pestle. The crushed samples were passed through the different sieves having different mesh size (i.e. ASTM No.- 120, 140, 170, 200, 230, 270, 325, 400, 500), to achieve the samples of desired particle size [125-105µm (SQ1), 105-90µm (SQ2), 90-74µm (SQ3), 74-63µm (SQ4), 63-53µm (SQ5), 53-44µm (SQ6), 44-37µm (SQ7), 37-25µm (SQ8)].

### 2.1.2 Nano-sized sample preparation:

Synthetic quartz nano-size sample was 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 collected sample was 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 sample (SQ9) was used for further investigations.

## 2.2 Characterization Techniques

Particle Size Analyzer (Malvern Zetasizer Nano ZS 90, Malvern Instruments, UK) was used for particle size measurement of prepared sample.

The morphological investigations of optimized the prepared synthetic quartz-nano suspension (SQ-NS) was carried out by using a Transmission Electron Microscope (TEM PHILIPS, Technai-20, Japan). A drop of SQ-NS was placed on a coated carbon grid (300 mesh, 3mm) and air dried. The grid was then examined immediately under Transmission Electron Microscope.

The sample was further characterized by X-ray diffraction (XRD) using a X-ray Diffractometer (XRD, X-Pert-PRO, PANalytical, Netherland) with CuK $\alpha$ , 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]

where d is the mean grain size,  $\beta$  is the full width at half maximum (FWHM).  $\theta$  is the angle of the peak maximum, and  $\lambda$  (0.15406nm) is the Cu(K $\alpha$ ) wavelength.

All the prepared samples were characterized by Fourier transfer infrared spectroscopy using MIRacle10 SINGLE REFLECTION ATR SPECTROSCOPY SHIMADJU IRAffinity-1 (Serial no: A213751) for examination of the impurities and defects in the samples.

Energy dispersive X-ray spectroscopy (EDS JELO MODEL JSM5810LV) was used to detect any inprocess impurity and to confirm elemental composition in prepared synthetic quartz samples.

UV-Visible-double beam spectrometer (UV-1800 Shimadzu, Japan) with fixed slit width (2nm) was used for all absorbance measurements of prepared micron and nano sized synthetic quartz samples using 1.0 cm matched quartz cells. UV spectra were observed at room temperature for micron and nano size samples followed by beta radiation of 25.2 Gy. PL emission spectra of prepared micron and nano size synthetic quartz samples were recorded with excitation wavelength at 254nm followed by beta radiation of 25.2 Gy using Spectrofluorimeter (RF-5301PC Shimadzu, Japan).

#### **3. RESULT AND DISCUSSIONS**

The average particle size of prepared nano-size sample (SQ9) was measured using particle size analyser. It was observed that the average particle size of prepared nanosized synthetic quartz was 87nm.



Figure 1: particle size analyser showing average particle size of prepared nanosized synthetic quartz.

The TEM image of prepared sample is presented in Figure 2. As per the image, it can be observed that the particles are in nano-metric range.



Figure 2: The Transmission Electron microscope photograph of synthetic quartz nano-size sample (SQ9).



Figure 3: XRD pattern of 48 hours milled synthetic quartz nanoparticles (SQ9).

Figure 3 shows the XRD pattern of the prepared synthetic quartz sample milled for 48 hours. The



crystalline size of prepared synthetic quartz was calculated using Debye Scherer formula and was found 39.41nm.



Figure 4: FTIR Spectra of prepared micron and nano-sized synthetic quartz samples.

Figure 4 shows the IR spectra of prepared micron and nano sized synthetic quartz samples. From these spectra, a series of absorption peak can be observed from 600 cm-1 to 4000 cm-1. To be precise, a broadband between 3200 cm<sup>-1</sup> to 3600 cm<sup>-1</sup> is corresponded to the O-H stretching mode of hydroxyl group, whereas the peak observed between 900 cm<sup>-1</sup> to 1100 cm<sup>-1</sup> is assigned to silicate ion [3]. It can be observed that as the size decreased, the hydroxyl group content increased in the prepared samples.



Figure 5 shows the EDS images of prepared synthetic quartz samples. From the images, it can be observed that the elemental composition of characterized synthetic quartz sample is SiO<sub>2</sub>.

The optical absorption spectra of micro and nano size samples are shown in Figure 6 and Figure 7. In UV spectra, only one absorption peak was observed in all micron-size samples in 210-235nm range where as in nano size sample, a broad absorption peak was seen in 210-480nm range. This absorption peak was assigned as due to the presence of E1centres (defects associated with oxygen vacancies) in synthetic quartz samples [4]. A broad band observed in nano synthetic quartz sample was due to increase volume to surface ratio at nano level.

The PL spectra of micro and nano-size synthetic quartz samples are shown in Figure 8. In PL spectra, it was noticed that PL intensity systematically increased with decrease in particle size up to 53 µm and reduced with further decrease in particle size from 53 µm to 39.41nm. The observed peaks at 371 nm, 385 nm, 400 nm have their origin from oxygen vacancy, [AlO<sub>4</sub>/Li+] centre, intrinsic emission respectively, while 451nm, 471nm, 482nm, 493nm peaks are due to intrinsic defect, self-trapped excitons and [AIO4]<sub>0</sub> centre. The intensity is affected by the concentration of both impurity and intrinsic defects [6]. For example, surface impurities such as hydroxyl are known to quench the exciton luminescence [5]. From the correlation between PL and defect showed by FTIR, It can be noted that hydroxyl group acts as non-radiative recombination center, when particle size decreases from 53µm up to 39.41nm. The PL intensity monotonically decreased with increase in particle size (range 53-



105  $\mu$ m); this may be due to the integrated intensities of emissions from excitons and defects; but it is partially limited by non-radiative processes [5].



Figure 6 Absorption spectrum of micro-size synthetic quartz samples.



Figure 7: Absorption spectrum of nano size synthetic quartz sample.



Figure 8: PL emission spectra of prepared micron and nano sized the synthetic quartz samples.

## 4. CONCLUSION

In present study, different micron- sized and nano size synthetic quartz samples were successfully prepared. The result obtained from characterization of nano-sized synthetic quartz sample using Particle size analyzer, TEM and XRD confirmed the nanosize of the same. EDS proved the elemental composition (i.e. SiO<sub>2</sub>) of the prepared samples. EDS also confirmed the presence of some in-process impurities which were in acceptable amount. In UV-visible spectrophotometric study of micron and nano synthetic quartz, only one absorption peak was observed in all micron-size samples which was due to presence of E1-centres (defects associated with oxygen vacancies). A broad band observed in nano sized sample was due to increase in volume to surface ratio at nano level. In nano samples, PL intensity systematically increased with decrease in particle size up to 53 µm and reduced with decrease in particle size from 53 µm to 39.41 nm. The PL intensity is affected by the concentration of both impurity and intrinsic defects as observed in FTIR spectra. In micro-sized samples, the increase in PL intensity with decrease in particle size down to 53 µm may be due to the integrated emission intensities of excitons and defects; however it is partially limited by non-radiative processes.

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## REFERENCES

- 1. E. G. Goh et al. I Scripta Materialia 78-79 (2014) 49-52.
- **2.** B. Bhushan, Handbook of Nanotechnology, Springer – Verlag, New York (2004).
- J. Coates, Interpretation of Infrared Spectra, A Practical Approach, Encyclopedia of Analytical Chemistry, R.A. Meyers (Ed.), pp. 10815–10837, Ó John Wiley & Sons Ltd, Chichester, 2000.
- 4. Y H Gandhi, Y D Kale, T R Joshi, Indian Journal of Pure & Applied Physics Vol.37, August 1999,pp. 600-603.
- G. Xiong, J. Pal, J. G. Serrano, K. B. Ucer, and R. T. Williams, phys. stat. sol. (c) 3, No. 10, 3577– 3581 (2006).
- 6. J. Gotze, M. Plotze, D. Habermann, Miner. Pet. 71, 225–250 (2001).