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Synthesis and characterization of $BaSO_4$ nano particles using micro emulsion technique

V. Ramaswamy*, R.M. Vimalathithan[1] and V. Ponnusamy[2]

*[1] *Department of Physics, Annamalai university, Annamalainagar, Tamilnadu, India*
[2] *Department of Physics, Anna University, MIT campus, Chennai, Tamilnadu, India*

ABSTRACT

The micro emulsion technique has good micro mixing in the precipitation process. The micro emulsion precipitation process used in this work is a laboratory scale and well established production method for precipitation of nanoparticles. Recently the synthesis of inorganic nanoparticles has attracted great interest due to their potential use in various fields. Spherical shaped $BaSO_4$ nano particles with barite structure were synthesized in Tween-20 / n-hexanol / kerosene / water water-in-oil micro emulsion by mixing of separately prepared $BaCl_2$ and Na_2SO_4 respectively. Particle characterization was accomplished by Fourier transform Infrared spectroscopy (FTIR), X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive Spectrum (EDS) and Particle Size Analysis (PSA). The XRD, SEM and PSA results show that the size of the particles were in the range 50-70 nm.

Keywords: Barium sulphate, microemulsion, Tween-20, kerosene, Agglomeration.

INTRODUCTION

A major trend in research and development has been the shift of interest towards tiny particles. This can be summarized under the general term “nanotechnology”. In the engineering and materials science community, the term “ultra fine particles” and “submicron particles” were used before “nanoparticles” came into being. Nowadays, three main notations for nanoparticles are being used: nanoparticle, nanocrystal and nanocluster [1]. The typical application of nanoparticles can be found in many fields like heterogeneous catalysis, semiconductors, microelectronics, information storage, pharmaceuticals, paints and ceramics. Particles with a size between the 1 to 100nm have the special benefit that physical and chemical properties differ from the bulk material properties and are strongly size dependent [2]. Nano scale materials can be made up of numerous ways. A broad classification divides the synthesis methods into either bottom up or top down process [3]. Bottom up routes are more often used for preparing most of

the nano-scale materials due to the ability to generate uniform size, shape and distribution. Within that bottom up route, chemical/wet synthesis is widely used for nano particle generation. The chemical synthesis method has numerous advantages such as simple technique, inexpensive, less instrumentation, doping of foreign atoms was possible and large quantities of materials can be obtained [4]. This chemical method could be classified as sol-gel [5], chemical vapour deposition [6], reactive precipitation [7], micro emulsion [8, 9] and low- temperature wet/chemical synthesis [10] etc, Among these different methods, micro emulsion technique was used in the present study due to highly simple, less instrumentation, thermodynamically stable and environmentally risk less. Recently the synthesis of inorganic nano particles has attracted great interest due to their potential use in various fields. Barium sulphate commonly referred to as barite, is suitable for many diverse uses because of its high specific gravity(4.5),opaqueness to X-rays, inertness and whiteness [11].It is mainly used as radio contrast agent, filler in plastics, extender in paints, additive in pharmaceutical products and printing ink.

All researchers were used Triton X-100 and Marlipal 013/40 as a non-ionic surfactant in a chemically pure grade for the preparation of $BaSO_4$ nanoparticles. In this study, the application of the technical non-ionic surfactant Tween-20 was used .The advantages of using this surfactant as a stabilizer are related with the absence of electrostatic interaction between freshly formed solid surface and surfactant molecule and the effect of foreign ions on the formation of a new phase. It is also inexpensive and available in large quantity, easy to separate and therefore preferred for the upscale approach. At the same time here kerosene was used as an oil phase. The $BaSO_4$ nanoparticles were synthesized successfully and characterized using FTIR, XRD, SEM-EDS and particle size analysis.

MATERIALS AND METHODS

2.1. Chemicals

The raw materials used in the study were $BaCl_2$, Na_2SO_4, Tween-20 (non-ionic surfactant), n-hexanol, kerosene, ethanol, and acetone (all in analytical grade). These chemicals were used directly with out any further purification and supplied by the S-D fine chemicals Ltd. The distilled water was used as solvent.

2.2. Experimental Procedure

Tween-20 was used as non-ionic surfactant and n-hexanol as co – surfactant. Kerosene was employed as an oil phase. 20ml of 0.5M $BaCl_2$ and 0.5M Na_2SO_4 were prepared separately. The micro emulsion system were prepared with the 10ml of Tween-20 was dissolved in 40ml of kerosene. Then n-hexanol was added to the obtained micro emulsion. The molar ratio of co-surfactant and surfactant ratio was 4:1. After stirring, the solution becomes transparent. Separately prepared salt solution was added drop wise to the surfactant mixture under vigorous stirring. After stirring over 5-10 minutes, the system was kept stable for complete precipitation. The solution was filtered to collect the solid materials. Then the precipitated particles were washed alternatively with ethanol and acetone for several times. The final product was dried in hot air oven for six hours at 100˚C. The powdered sample was taken for characterization.

2.3. Characterization

Sample was characterized by a number of different methodologies, including FTIR, XRD, SEM-EDS and particle size analyzer.

The powdered sample was mixed with KBr at various ratios 1:10, 1:20, 1:30, 1:40 and 1:50. The pellets were prepared and FTIR spectra were taken. The maximum absorption and large number of peaks are observed for the samples in the ratio of 1:30 (sample-KBr) and chosen for analysis. The Perkin Elmer RX1 series FTIR spectrometer was used for recording the IR spectra of the sample. The instrument wavelength ranges from 4000 to 400cm^{-1} with an accuracy of ±0.01cm^{-1} and resolution of ±4cm^{-1}. The instrument was calibrated with the spectra of a standard polystyrene film at room temperature. Every time, before the spectrum of the sample was obtained; the spectrum of the polystyrene film was obtained and checked for the accuracy and transmittance.

The X-ray pattern of the powdered sample were recorded at room temperature using XPERT-PRO PAN alytical having a curved graphite crystal diffracted monochromater, with a source of cu kα (λ=1.5406A°) at 40kV and 30mA,Ni filter and NaI(T1) scintillation detector cooled by liquid nitrogen. During the recording of the diffractrogram, a narrow silt of 0.1mm was used with a scanning speed of 1/2° per min and a time constant of 2 sec. The sample was firmly pressed into an aluminium holder, with an area of 30mm×30mm. The diffraction patterns were obtained over the 2θ values in the region 20˚ ≤2θ≤50˚.

The SEM microphotographs were recorded with an accelerating voltage 20kV at vaccum (HV) mode and secondary electron image using JEOL SEM model JSM 5610LV manufactured by Hitachi Ltd., TOKYO, Japan. Sample was placed in specimen stub using double sided adhesive carbon tape and the specimen were gold coated with the help of JEOL auto fine coater-JEOL 1600. Elemental analysis of samples was also made using Energy dispersive Spectrum (EDS) facility available with the SEM.

Particle size distribution was analyzed using Micro Track Blue Wave 3500 Model Particle Size Analyser with resolution of 10nm.

RESULTS AND DISCUSSION

3.1. Fourier transform Infrared (FTIR) studies

FTIR spectroscopy is a useful technique to characterize inorganic compounds. Figure (1) shows FTIR spectrum of prepared $BaSO_4$ particles.

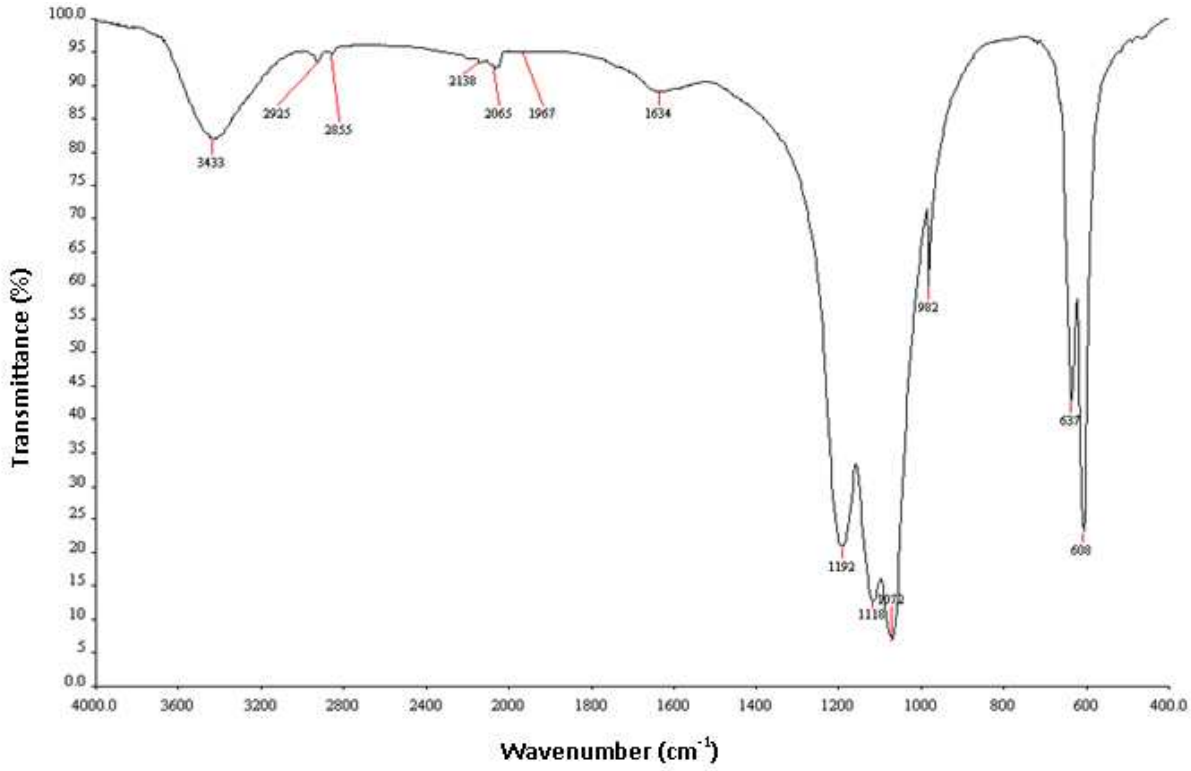


Fig. 1. FTIR spectrum of synthesized Barium sulphate particles

The observed wave numbers are 3433, 2925, 2855,2138, 2065, 1634, 1192, 1118, 1072, 982, 637 and 608cm^{-1}. According to Alder and Kerr, (1965) [12], the sulfur-oxygen (S-O) stretching of inorganic sulfates are found in the region 1179 - 1083cm^{-1}. The bands centered at 1192 to 1072cm^{-1} and the shoulder at 982cm^{-1} is the symmetrical vibration of SO_4^{2-}. From this observation the slight shift in the peak position may be attributed to the smaller particle size. According to Shen et al. (2007) [13], the peaks at 608 and 637cm^{-1} are due to the out-of-plane bending vibration of the SO_4^{2-}. Generally, bending bands are shaper than the stretching bands, which is observed in the above spectrum. The absorption peaks appeared at about 3433 and 1634cm^{-1} are due to the stretching and deformation of adsorbed water molecule. The weak absorption band at 2855 and 2925cm^{-1} could be assigned to the symmetric and asymmetric vibrations of $–CH_2$ and $-CH_3$ groups. According to Manam and Das, (2009) [14], the peaks near 2000cm^{-1} are overtones and combination bands of the lower wave number of sulfur-oxygen stretching and bending vibrations and these peaks do not affect the identification of the substance involved in the experiment.

3.2. X-Ray Diffraction

X-ray diffraction study was effectively used to identify the crystal structure and determine the particle size [15].

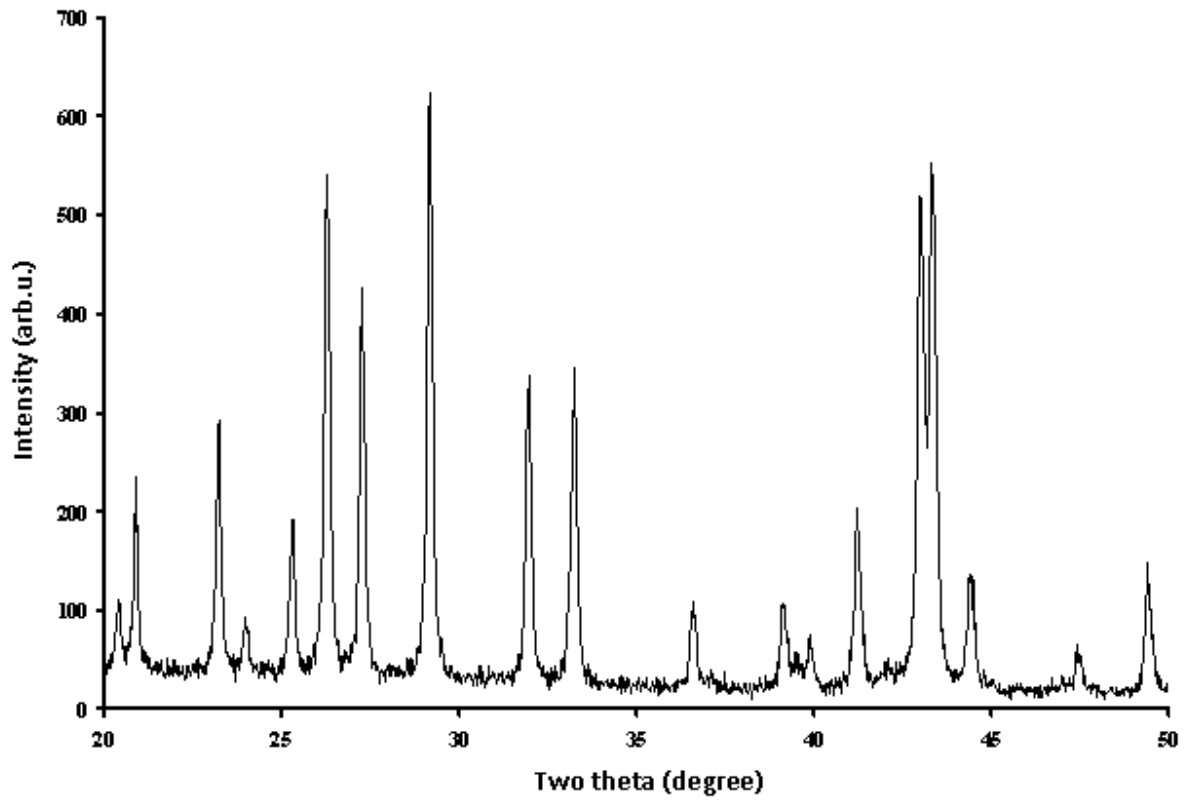


Fig. 2. The XRD pattern of synthesized $BaSO_4$ particles

Figure (2) shows the typical XRD pattern of $BaSO_4$ particles prepared through micro emulsion technique. All the diffraction peaks could be matched with the reference of the barite structure (JCPDS card no. : 24-1035) [13 and 15].The diffraction peaks of (h k l) values (101),(111),(021),(121),(002)and (212) were the characteristics of orthorhombic $BaSO_4$ crystals, only $BaSO_4$ peaks were observed in the XRD spectra which indicate that the prepared sample had high purity in nature.

The crystallite size was calculated using the Debye–Scherrer equation. The diameter of the $BaSO_4$ was calculated as follows

Where D, λ, β and θ are the average $D = \frac{0.94\lambda}{\beta \cos\theta}$ diameter, the X-ray wavelength, half width of the peak (full width at half maximum) and the Bragg's diffraction angle respectively. The crystalline sizes of the $BaSO_4$ are estimated for high intensity peaks. According to Scherrer equation, the average particle size of $BaSO_4$ is range between 50-70nm.

3.3. Particle Size Distribution

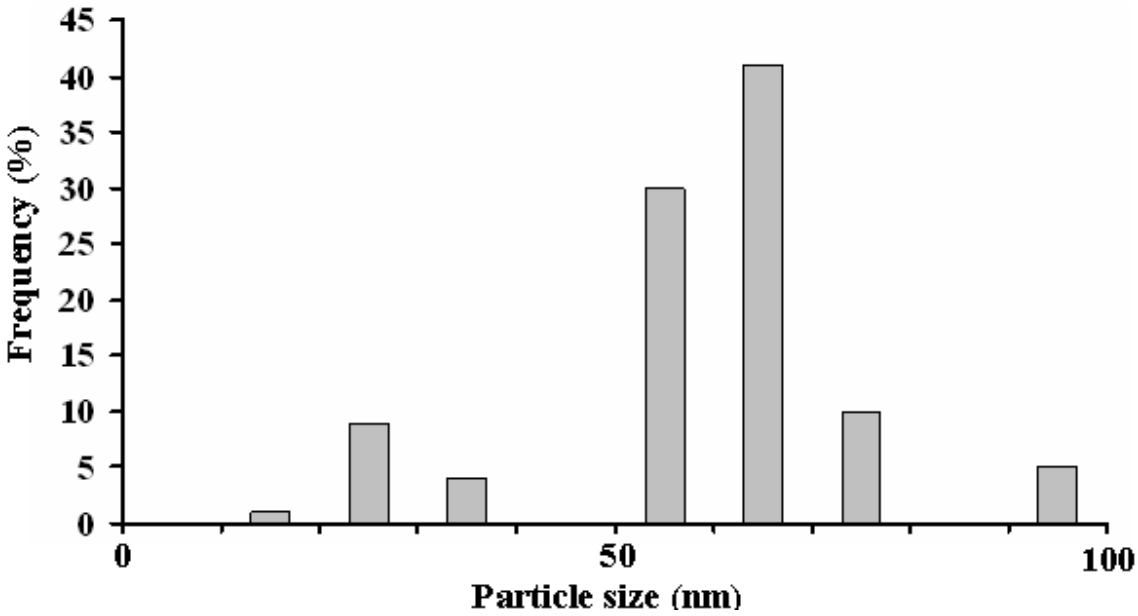
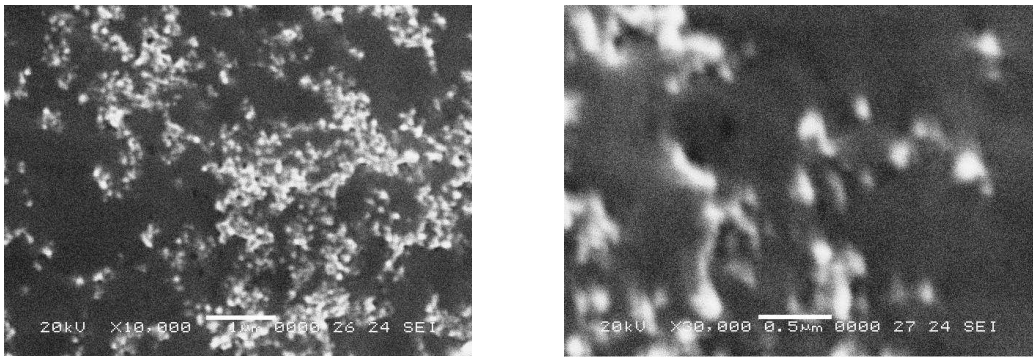


Fig. 3. The particle size distribution for $BaSO_4$ nanoparticles

Particle size distribution was analysed using Micro Track Blue Wave 3500 model particle size analyser with resolution of 10nm and is shown in figure (3). The properties of nanoparticles are significantly depend on the particle size distribution. Especially evenly distributed particle size is of interest due to the size dependent physical and chemical properties of nanoparticles. Most of the particles (41%) have size in the range 66nm. This is in good agreement with the XRD result.

3.4. SEM-EDS

Scanning electron microscopy was used to analyze the morphology and size of the synthesized $BaSO_4$ particles.



4(a) **4(b)**

Fig. 4. The SEM image of the Barium sulphate nanoparticles

Figure (4) shows the SEM images of the $BaSO_4$ nano-particles. The morphology of the $BaSO_4$ nanoparticles shows that the particles are in spherical shape. According to Limin Qi et al. (1996),

[11] the water content greatly affects the shape of the $BaSO_4$ particles. When the water content is maximum, cubic particles can be obtained. However, in the present study, the particles are in the spherical shape which shows that the water content is minimum. The spherical nanoparticles, which are uniform in size, shape and arranged systematically. Particles seen in SEM figures 4(a & b) are appeared to be ultra-fine and having agglomeration. The surfactant plays an important role on the surface of the particles to breakage into smaller particles. From SEM images, it was clearly seen that the size of the particle was less than 100nm range.

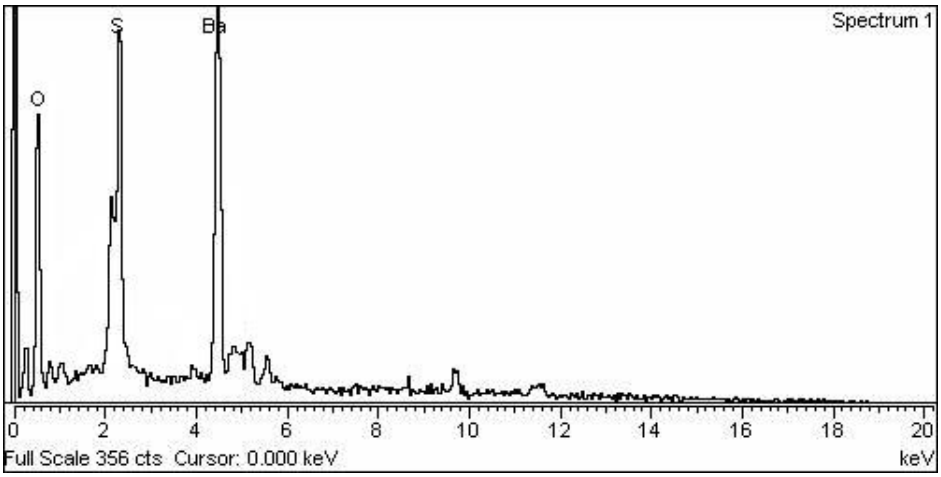


Fig. 5. The EDS spectrum of the Barium sulphate nanoparticles

Figure (5) shows the energy dispersive spectrum of the $BaSO_4$ particles which clearly show the presence of Ba, S, and O elements.

CONCLUSION

The $BaSO_4$ nano particles were prepared successfully by the chemical reaction in the Tween-20 / n-hexanol / kerosene / water micro emulsion system using $BaCl_2$ and Na_2SO_4 as reagents. With the best of our knowledge, no work has been done so far in the preparation of $BaSO_4$ nano particles using Tween-20 and kerosene as surfactant and oil phase respectively. FTIR study clearly reveals the functional group of $BaSO_4$ nanoparticles. In addition, XRD analysis shows that the synthesized $BaSO_4$ is in orthorhombic structure. The average particle size of $BaSO_4$ was determined as 50-70nm using Scherrer equation. From the particle size analyzer, the particle sizes were in the range 59-68nm. It is in good agreement with XRD analysis. The SEM study dictates the morphology of the $BaSO_4$ nanoparticles which were in spherical shape and also particle size was less than 100nm. The shape of the particles gave the information about the content of water. The EDS spectrum shows its purity of synthesized sample. Further, the particle size can also be reduced by adjusting the initial reagents concentration, pH value, and temperature. From the overall point of view, the micro emulsion technique is one of the well established technique for the synthesis of $BaSO_4$ nano materials.

REFERENCES

[1] F. Einar Kruis, Rakesh K. Joshi,*China Particuology*, **2005**, 3, 99-104.
[2] B.Niemann, F. Rauscher, D. Adityawarnan, A. Voist, K. Sundmacher, *Chem. Eng. Process*, **2006**, 45, 917-935.
[3] R. Kelsall, I. Hamley, M. Geoghegan, *Nanoscale Science and Technology*, Wiley and Sons, Weinheim, **2005**.
[4] Sulabha K.Kulkarni, Nanotechnology: Principles and Practices, Capital Pub. Com., New Delhi, **2006**.
[5] A. Chatterjee, D. Chakravorty, *J. Mater. Sci.*,**1992**, 27, 4115.
[6] F.E. Kruis, H. Fissan, A. Peled, *J. Aerosol Sci*., **1998**, 27, 511.
[7] Y.F. Gu, S. Wang, L.M. Hu, A.I. Zhang, *J. East-Chin. Inst. Chem. Technol*., **1993**, 15,550.
[8] J.A. Perez, M.A. Quintela, J. Mira, J. Rivas, S.W. Charles, *J. Phys. Chem*. B, **1997**, 101, 8045.
[9] D. Adityawarman, A. Voigt, P. Viet, K. Sundmacher, *Chem. Eng. Sci*., **2005**, 69, 3373- 3381.
[10] H. Chen, H. Chang, *Ceramics International*, **2005**, 31, 795-802.
[11] L. Qi, J. Ma, H. Cheng, Z. Zhao, Colloids Surf. A: *Physicochem. Eng. Aspects*, **1996**,108, 117-126.
[12] H. H. Adler, P. F. Kerr, *Am. Mineral*., **1965**, 50, 132-147.
[13] Y. Shen, C. Li, X. Zhu, A. Xie, L. Qiu, *J. Zhu, J. Chem. Sci*., **2007**, 119, 319-324.
[14] J. Manam, S. Das, *Indian J. Pure Ap. Phy*., **2009**, 47, 435-438.
[15] J. Li, Y. Xu, D. Wu, Y. Sun, *China Particuology*, **2003**, 1, 134-136.