Synthesis and Characterization of CuO Nano Particles by Novel Sol-Gel Method

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Abstract. In the development of nano size materials of metal and metal oxides particles are intensively pursued because of their prominence in different fields of applications in science and technology. All the transition metal oxides, CuO is a potential candidate for magnetic storage devices, solar energy transfer, sensors, and super capacitors and especially it acts as a good catalyst in some of the chemical reactions. CuO Nano particles are prepared by novel sol gel technique. In this technique CuCl₂.6H₂O is added with acetic acid and heated to 100 °C with continuous stirring. Control the pH of solution, NaOH is added to the solution till pH reaches desired value. The color of the solution changed from blue to black with precipitation. The black precipitation was washed 3-4 times with distill water. Finally it was centrifuged and dried in air for one day. The CuO Nano particles were characterized for the studying of their structure and composition from X-ray diffraction, Energy Dispersive X-ray analysis and size with Particle Size Analyzer. The thermal analysis was carried by TG-DTA. Catalytic nature parameter zeta potential was also measured, For the morphology test SEM and TEM carried out.

Keywords: CuO Nano-particles, Sol-gel method, Novel applications.

1. Introduction

In the universe there is number of metal oxides are available in nature but some of the metal oxides are most useful in accordance with their applications of day to day life in science and technology. In the periodic table transition metals are large in number and have number of applications in different fields applications. Some transition metal oxides like ZnO, TiO₂, and Fe₃O₄ etc. proved as potential candidates for so many applications. In the same way CuO is also one of the useful metal oxide and which has so many applications in different fields. The unique property of CuO is it acts as a semiconductor. Semiconductor materials have been particularly interesting because of their great practical importance in electronic and optoelectronic devices, such as electro chemical cell[1] gas sensors[2], magnetic storage devices[3], field emitters[4], high-Tc super conductors[5], Nano fluid[6], and catalysts[7] etc. Due to the potentiality of CuO, it acts as a catalyst; whereas all metal oxides are not useful for the catalytic activity. As like Fenton’s reagent CuO combined with another metal oxide like CeO₂, CuO is used in waste water treatment. Electrical applications like super capacitors CuO is very useful and in Nano range it has the wide band gap nearly equal to ZnO. The favorable band gap of CuO makes it useful for solar energy conversion and it can be used for solar cell window preparation. It is very well established fact that We know the application of Nano fluids acts as a coolant in refrigerators, these Nano fluids mixed with carrier liquid then it enhance the energy transfer comparatively carrier liquid alone. CuO can be used as coolant material and it can control effectively the temperature like other coolants like TiO₂, alumina and silver particles etc. The properties of Nano materials will depends mostly on the Nano powder material size, morphology and specific surface area of the prepared materials. Some aspects are strongly depends on the preparation methods. Nano materials are separated from the bulk materials are on the basis of surface to volume ratio. In Nano scale preparation we observed that the size quantization effects will have lot of influence on the material properties. Our present work on CuO Nano crystals shows distinct structural, chemical bonding and electronic characteristics. Lattice strain and
structural disorder can change the properties of materials in the applications of different fields in science and technology. There is no of methods among those we select one that is more preferable and widely used in the synthesis of Nano particles. There are wide variety of preparation methods are available for the synthesis of CuO nanoparticles. Among these methods our preparation method for the synthesis of CuO nanoparticles belongs to chemical method, i.e. Sol-Gel method. The range of the particles from the preparation methods as follows In case of MOCVD [Metal Organic Chemical Vapor Deposition] CuO Nano particles is 0.05 to 8µm[8], sonochemical method is around 10 nm to several microns[9]. Some results shows in some references which is in the range of 1-10 nm CuO Nano particles in Sol-Gel method[10]. The average diameter of the CuO Nano particle in solid state reaction is given by 15-20nm[11]. Smallest CuO Nano particle was prepared by electro chemical method and the size is around 4nm [12]. Decomposition of copper acetate is under controllable diameters of 3-9 nm CuO Nano particle [6].

2. Experimental Details

CuO Nano powders were prepared by Sol-Gel method. The aqueous solution of CuCl₂.6H₂O (0.2 M) is prepared in cleaned round bottom flask. 1 ml of glacial acetic acid is added to above aqueous solution and heated to 100°C with constant stirring. 8 M NaOH is added to above heated solution till pH reaches to 7. The color of the solution turned from blue to black immediately and the large amount of black precipitate is formed immediately. It is centrifuged and washed 3-4 times with deionized water. The obtained precipitate was dried in air for 24 h. This powder is further used for the characterization of CuO nanoparticles.

The crystalline phases of CuO Nano particles were determined by X-Ray diffractometer (XRD, BrukerD8 Advance, Germany) using CuKα as radiation source (40 kv, Step Size 0.02°, scan rate 0.5° min⁻¹, 2θ=70°) and composition analysis was done by Energy Dispersive X-ray analysis (EDAX). The Nano particle size and zeta potential is estimated by particle size analyzer (SZ-100 Nanoparticle, Horiba,Germany) data. Thermal analysis was measured by thermo gravimetric analysis (TG-DTA, A6300R). The particle size and morphology of the prepared nanoparticles particle were calculated by scanning electron microscope (SEM, HitachiVP-SEM S-3400N,Germany).

3. Results and Discussion

3.1. Crystal Structure of Product and Compositional Analysis

Fig. 1 shows the XRD pattern of prepared CuO Nano particles. All the peaks in diffraction pattern shows monoclinic structure of CuO, and the peaks compared with JCPDF card no. [89-5895] and miller indices are identified. The lattice parameters were calculated from XRD data is as follows. The average grain size calculated by using Debay-Scherrer formula is approximately 18.09nm. Lattice parameters from XRD calculated, a=0.46nm, b=0.34nm, c=0.50nm. Formula for the calculation of grain size

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \]

Where \( \beta \) is full width half maxima of the peak in xrd pattern

\( \theta \) is peak obtained angle

\( \lambda \) is X-ray wavelength

Elastic strain calculated from XRD results it shows that below 20nm smaller particles have high value of strain and greater the particles size have less value of strain. It clearly shows that smaller particles have high strain and bigger particles have less strain. Morphology index was calculated From XRD full width half maxima and it says that size of the particle increase, morphology index is also increase.

Fig. 2 shows Energy dispersive X-Ray Analysis of CuO Nano particles and the data indicates the Nano powders are nearly stoichiometric. The wt. % of copper and oxide calculated from EDAX is O 9.03wt. % and Cu 90.97wt. %. There are no traces of other impurities like carbon etc. in the EDAX spectra. The EDAX result confirms the formation of pure CuO Nano particles.

Fig. 3 shows TEM micrograph of CuO nanoparticles. The actual size of nano particles is estimated from TEM micrograph. Most of the nanoparticles have size around less than 50 nm and which is in correlation
with the particle size analyser data. The TEM graph is also showed that the copper oxide Nanoparticles are spherical in shape and nearly uniform in size.

Fig. 1: XRD pattern of CuO nano particles.

Fig. 2: EDAX of copper oxide nano particles.

Fig. 3: TEM photograph of CuO nano particles.
3.2. Thermal Properties

![TG/DTA of CuO nanoparticles](image)

Fig. 4: TG/DTA of CuO nanoparticles.

Fig. 4 shows the thermal gravimetric analysis (TGA) data of the given CuO nanoparticle. The weight loss of the material is observed between the temperatures 250-1200°C was about 0.613%. This weight loss is mainly due to vaporization of water content in the sample. From TGA analysis the total weight loss is calculated and that is 3.530%. The TGA results suggested that this compound is thermally highly stable and it has negligible weight loss when compared to bulk sample of CuO. Above 400°C-600°C it has the weight loss of 1.2% that is due to carbon group compounds. Above the 600°C weight loss is 1.2%, it may be due to oxygen loss[13].

3.3. Morphology Studies

![SEM photograph of CuO nanoparticles](image)

Fig. 5: SEM photograph of CuO nanoparticles.

Fig. 5 represents the SEM images of Nano CuO particles prepared by sol-gel method are as shown in figure5. It shows that higher tendency of agglomeration. We collect the SEM images in different scales most of the images given particle size around 0.53 µm to 0.14 µm less present of agglomeration observed in ≤0.14µm. The smallest particle size of the particle is around 0.12 µm.

4. Conclusions

- XRD pattern revealed copper oxide Nano particles have monoclinic structure.
- From EDAX analysis the copper oxide Nano particles are pure and free from impurities.
- TEM photo graph shows all particles size good agreement with the size of XRD calculations.
• TGA analysis shows that thermal weight loss is very low.
• SEM photograph shows good agglomeration of CuO nano particles.

5. References