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Synthesis and Thermal Properties of Copper Nanoparticles

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Copper nanoparticles have been synthesized by simple chemical precipitation method and showed versatile thermal, mechanical, magnetic and electrical properties. Copper nanoparticles have been synthesized in following two steps, in step-1st, synthesis of polymer metal complex and in 2nd step formation of copper nanoparticles. Polymer metal complex is confirmed by IR spectroscopy and XRD confirmed by size of nanoparticles is 13.13 nm, Crystal system is orthorhombic, Bravais lattice is primitive, space group pccn(56) and $2\theta = 42.045$. Thermal stability is confirmed by TGA and the compound is very slow (rate of 3 %) loss in weight per 100 °C increase in the temperature up to 900 °C. It shows that 70 % thermal stability of the compound at 900 °C. Endothermic nature of copper nanoparticles has been confirmed by DSC.

Keywords: Copper-nanoparticle, Thermal stability, Orthorhombic, Bakelite.

INTRODUCTION

Nanomaterial is a mesmerizing material that is found in many applications in the field of basic and applied research. Copper nanoparticles with high specific surface to volume area have been widely studied. The copper nanoparticles have special physico-chemical characteristic which include catalytic activity, electronic properties and antimicrobial activity [1]. Nanoparticles have received considerable interest due to their good thermal, optical and electrical properties. Copper oxide is a semiconducting compound and its structure is a monoclinic. Copper compounds exhibit a range of potentially useful physical properties such as high temperature superconductivity and spin dynamic [2,3]. As in important p-type semiconductor copper oxide has found many diverse applications. Copper nanoparticles have been assumed cost effective as compared to noble metals like Ag, Au and Pt. They are potentially applied in the fields of catalysis [4], conductive inks [5] and fluids [6]. The synthesis of mono disperse, ultrafine metal nanoparticles have been potential application in photonics, catalysis, nanofluides, cooling fluids for electronic systems, Plasmonic photovoltaic and composite food packaging [7-14]. There are different methods of synthesis of copper nanoparticles including thermal decomposition [15], solid liquid discharge [16], quick precipitation [17] metal vapour synthesis [18], chemical reduction [19], vacuumed vapour deposition [20], radiation method [21], micro-emulsion techniques [22], polyol processes [23] and laser ablation [24]. Major limitations in the synthesis of their ease of oxidation [25]. Chemical precipitation method is one of the most convenient methods for the synthesis of metallic nanoparticles because this synthesis is simple, shape and size of nanoparticles can be controlled. Copper nanoparticles have been prepared by simple chemical precipitation method and characterized by infrared spectroscopy, scanning electron microscope and thermal gravimetric analysis.

EXPERIMENTAL

All the chemicals were used of AR grade, formaldehyde and phenolfrom [Central Drug House (P) Ltd.], glacial acetic acid and hydrochloride acid (Fisher scientific). Metal solution has been prepared by dissolving appropriate amount of copper(II) chloride in distilled water.

Synthesis of copper nanoparticles

Set-1st: Synthesis of polymer metal complex: In 250 mL beaker taken of 10 g phenol and 20 mL of formaldehyde after continuous 15 min stirring, then 25 mL of glacial acetic acid was added drop by drop with maintained temperature. Few drops of HCl was also added. After stirrer few minute, light pink colour bakelite compound was formed. In another beaker freshly prepared 1 N copper solution was taken. Added drop by drop 15 mL of 1 N metal ion solution then the reaction

mixture was continued stirrer for 30 min then heated at 45 °C for 1 h on heating mental. After heating the polymer composite was formed. The solid sample had been purified by the washing with distilled water solution. The excess metal ion and impurities on the sample purified by washing. Then it was kept into the desiccators for drying.

Set-2nd: Synthesis of copper nanoparticles: In order to get nanoparticles of polymer metal complex decomposition takes place at 850 °C for 30 min and purification done in following steps.

Purification of copper nanoparticles

Step-1st: Remove volatile impurity: At the time of decomposition many volatile impurities were separated and nanoparticles become free from these impurities.

Step-2nd: Remove metallic impurities: For removal of the metallic ions, nanoparticles were kept in 12 N HCl solution for 24 h. Then they were centrifuged and washed with distilled water till hydrochloric acid was completely removed.

RESULTS AND DISCUSSION

^{1}H NMR spectra: The analysis of NMR spectra of polymer metal complex indicate three types of proton present in sample. Peak at 3.524 ppm shows that phenolic protons present in compounds, peak at 2.471 ppm it represent that the benzylic proton is present in polymer metal complex, at 6.744 ppm it represent aromatic proton present in polymer metal complex. Multiplates at 6.744, 3.695 and 2.471 ppm, it represents that the formed complex polymerized substances have been formed.

IR spectra: The IR Spectra analysis of polymer metal complex are given in Table-1.

TABLE-1
IR SPECTRA

Prominent absorption band (cm^{-1})	Functional group
3278	OH stretching (broad peak)
3007-2921	C-H stretching (aromatic)
1173	C-O stretching

X-RD characterization: Orthorhombic copper nanoparticles have been determined using XRD technique. Other use of XRD technique is to evaluate the particle size using Scherrer equation:

$$D = K\lambda/(B \cos \theta)$$

where D is the mean size of crystallites (nm), K is crystallite shape factor a good approximation is 0.9, λ is X-ray wavelength, B is full width at half the maximum (FWHM) in radians of the X-ray diffraction peak and θ is the Bragg angle. Different copper nanostructures sizes were obtained using wet chemical precipitation method. By applying Debye-Scherrer equation to the obtained XRD pattern (Fig. 1) of the copper nanoparticles, the average nanoparticles size was found to be 13.13 nm and bravais lattice is primitive and space group is pccn(56) and 2θ = 42.045.

TGA and DSC: Thermal properties of copper nanoparticles were characterized using thermogravimetric analysis (TGA). The TGA thermograph predicts the 30 % mass decomposition of composite from 29-900 °C. The TGA graph shows at a heating up to 100 °C/min. The graph shows little loss in a sample with increased temperature. The decomposition of copper nanoparticles takes place at very slower rate of 3 % loss in weight per100 °C increase in the temperature up to 900 °C. It shows that 70 % thermal stability of the compound at 900 °C (Fig. 2). The isothermal behaviour of copper nanoparticles has been investigated using DSC technique over the temperature range 25-900 °C in ambient air. DSC curve (Fig. 3) of copper nanoparticles shows the large endothermic peak at 609.61 °C.

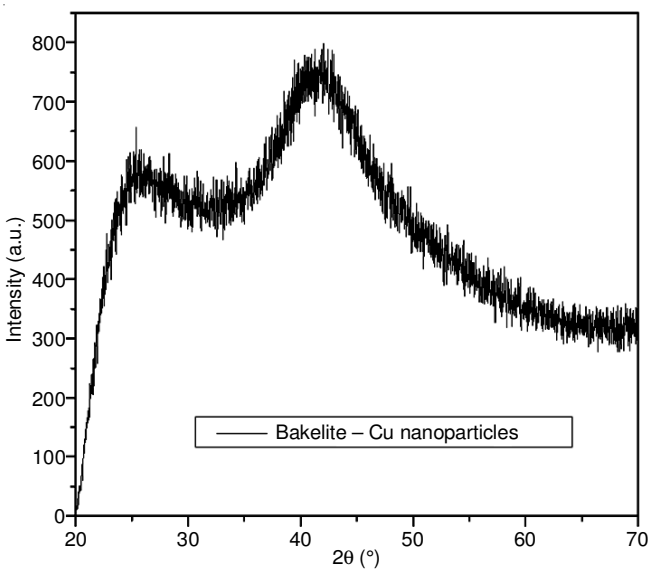

Fig. 1. XRD spectra of copper nanoparticles

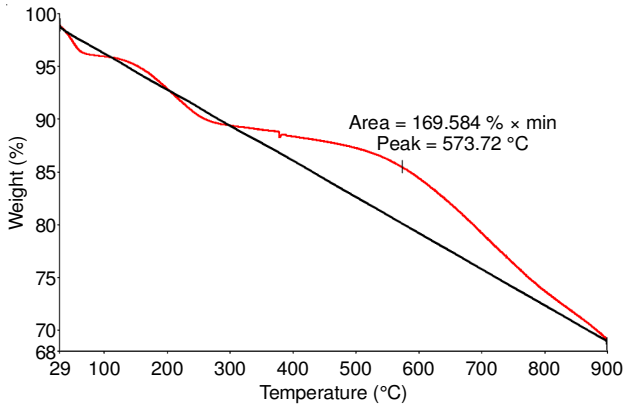

Fig. 2. TGA graph of copper nanoparticles

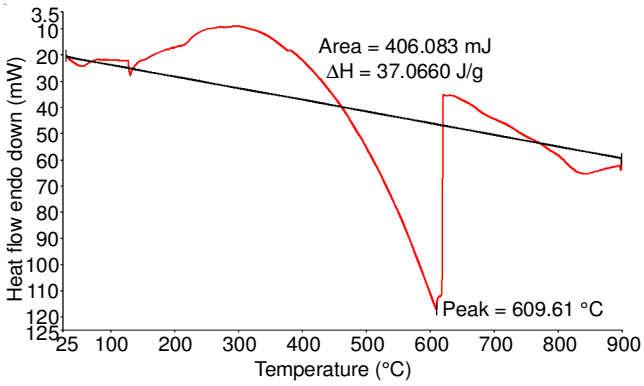

Fig. 3. DSC graph of copper nanoparticles

Conclusion

Copper nanoparticles have been synthesized by the chemical precipitation method. The complex formation is characterized by infrared spectroscopy and NMR spectroscopy. The crystallinity of the synthesized nanoparticles is confirmed by XRD and result revealed that size of copper nanoparticles were of 13.13 nm. Thermal studies confirms the high thermal stability of and endothermic nature of nanoparticles.

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