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**ORIGINAL ARTICLE**

# Synthesis and characterization of copper nanoparticles by reducing agent

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**Abstract** Cu nanoparticles were synthesized by solution reduction process successfully. The influence of parameters on the size of Cu nanoparticles was studied and the referential process parameters were obtained. The morphology and structure of the synthesized Cu nanoparticles were characterized by transmission electron microscopy (TEM), powder X-ray diffraction (XRD), QELS data, infrared spectroscopy (IR) and solid state UV. The average size of nanoparticles was found between  $15 \pm 2$  nm.

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**1. Introduction**

Copper and the compounds of Au, Ag, Pd and Pt are widely used these days. Copper has an excellent electrical conductivity. Due to relatively low costs, this metal plays a significant role in modern electronic circuit (Schapter et al., 2004). Because of its excellent electrical conductivity, catalytic behaviour, good compatibility and surface enhanced Raman scattering activity, Cu nanoparticles have drawn the attention of scientists to be

used as essential component in the future nano-devices (Pergolese et al., 2006). Cu nanowires are used in nanoelectronics and have application possibilities for magnetic devices, nano-sensors, electron emitters and other electronic applications. Cu nanoparticles have been explored to be used as nanoprobe in medicines and bio-analytical areas (Tomil, 2003). High temperature superconductivity materials are mostly prepared from CuO based compounds (Bhattacharya et al., 2006). Similarly semiconducting antiferromagnetic materials are also synthesized from Cu. Nanoparticles are a class of materials with properties which differ from their characteristics and find use in different areas such as electronic, magnetic, pharmaceutical, cosmetic energy, catalytic and materials applications. A large number of nanoparticles have been prepared most frequently by dispersion of performed polymers (Kompella et al., 2001; Santanu et al., 2007) solvent evaporation method (Kwon et al., 2001) and ionic gelation method (Calvo et al., 1997). In order to produce small particle size, often a high speed homogenization or ultrasonication may be employed. Conventional methods such as solvent extraction–evaporation, solvent diffusion and organic phase separation methods are hazardous to the environment as well as physiological systems. A variety of reducing systems are available to carry out reduction that

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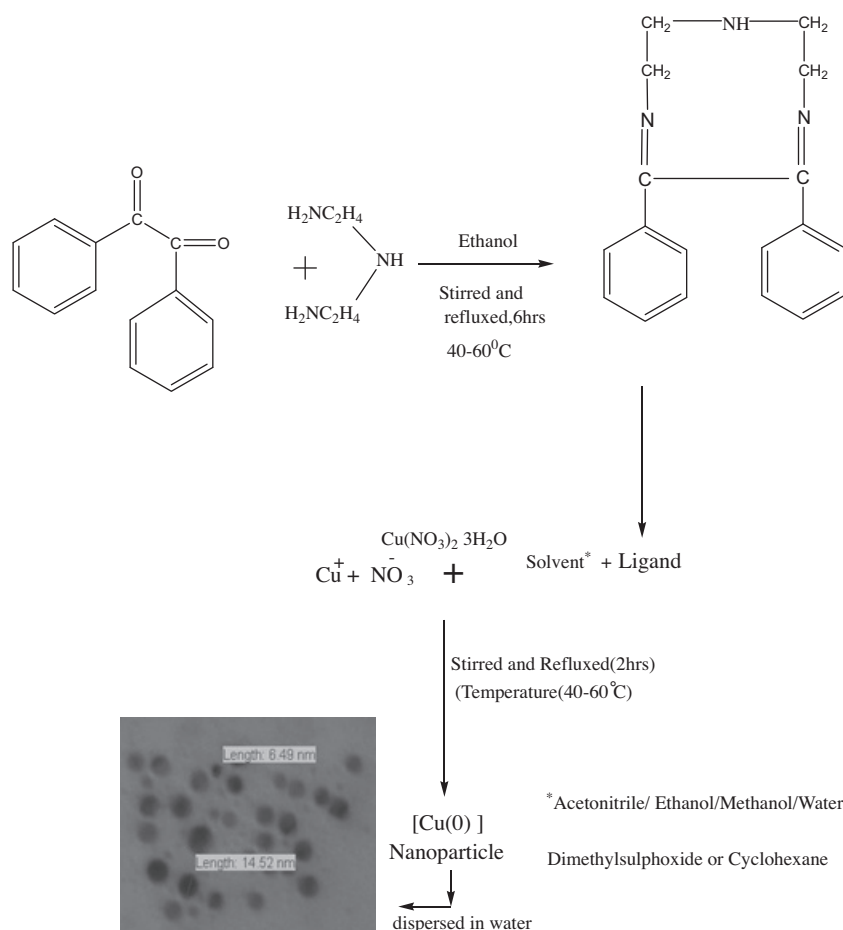
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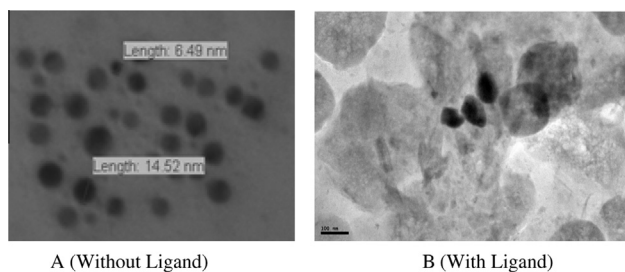
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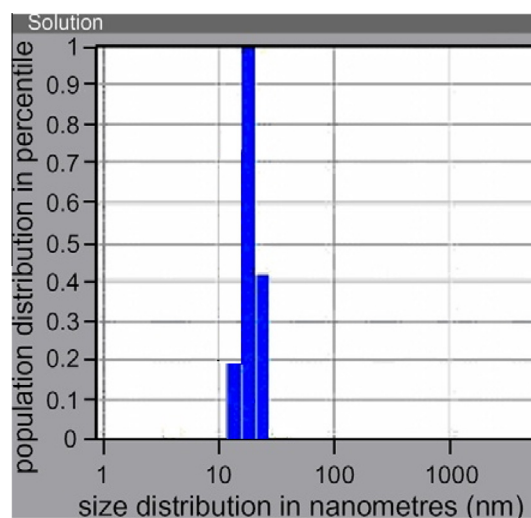


**Scheme 1** The schematic formation of Cu(0) nanoparticles by reduction method from  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ .



**Figure 1** (A and B) The TEM images of Cu(0) nanoparticles.

includes  $\text{NaBH}_4$  (Ohkuma et al., 1995), Cu, Ni, Co complexes (Chandra and Pundir, 2008) and macrocyclic ligands (Chandra et al., 2009). This method avoids the use of hazardous polymers and solvents which are required in excess amounts. The most popular Cu(I) (Shin-ichi et al., 2007) and Cu(0) (Mitamura and Akiya, 2009) nanoparticles emerged as useful and unique green catalysts whose efficiency is attributed to their characteristic high surface to volume ratio that translates into more number of active sites per unit area compared to dominate the properties of matter as size is reduced to nanoscale. As a part of continuing efforts in our laboratory towards the synthesis of nanoparticles in new synthetic methodology (Chandra and Kumar, 2011a,b), we report the synthesis and characterization of copper nanoparticles by reducing agent.



**Figure 2** The quasi elastic light scattering data (QELS) data.

## 2. Experimental

### 2.1. Reagents and measurements

Reagent grade  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  was obtained from Acros organics. Benzil and diethylenetriamine were purchased from

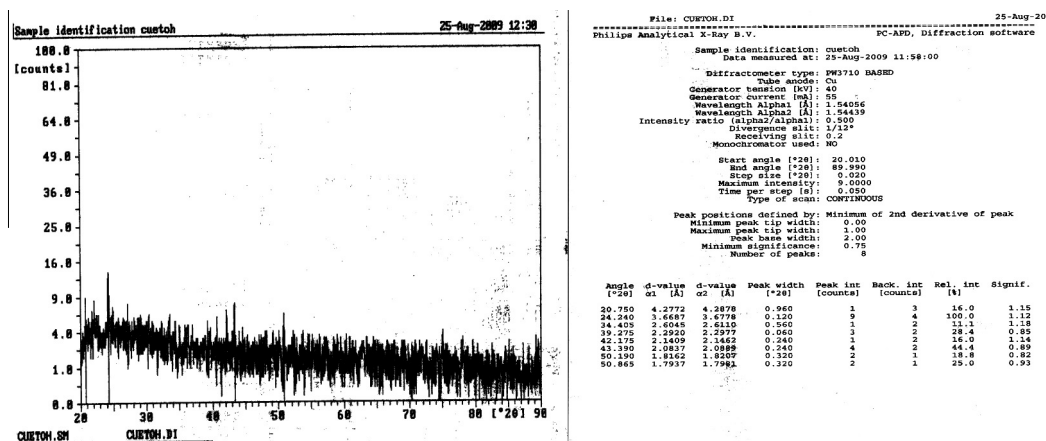


Figure 3 The powder XRD graph of Cu(0) nanoparticles.

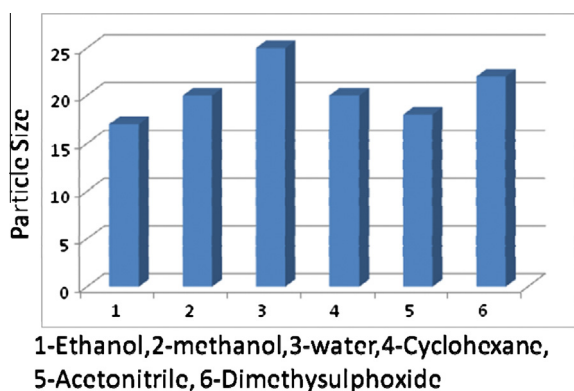


Figure 4 The particle size distribution of Cu(0) nanoparticles synthesized in different solvents.

Loba chemie and Tedia Company and used as received without further purification. Solvents were used of Analytical grade. IR spectra were recorded on Perkin-Elmer FTIR-1710 spectrophotometer using KBr. <sup>1</sup>H NMR spectra were recorded on FT NMR Hitachi R-600 (60 MHz). Elemental data was obtained by using a Perkin-Elmer 240C CHN elemental analyzer. The size of nanoparticles was confirmed as (5–20 nm) through quasi elastic light scattering data (QELS) and transmission electron microscopy (TEM). The transmission electron microscopy (TEM) images of nanoparticles were obtained with a JEOL JEM-1200EX transmission electron microscope operating at 120 kV with an energy dispersive spectrometer (EDS). The mass spectra were recorded on JEOL NMS DX 303 at 70 eV or on a KC455 Waters TOF MS spectrometer. Powder XRD was recorded on Philips X-Ray PW3710.

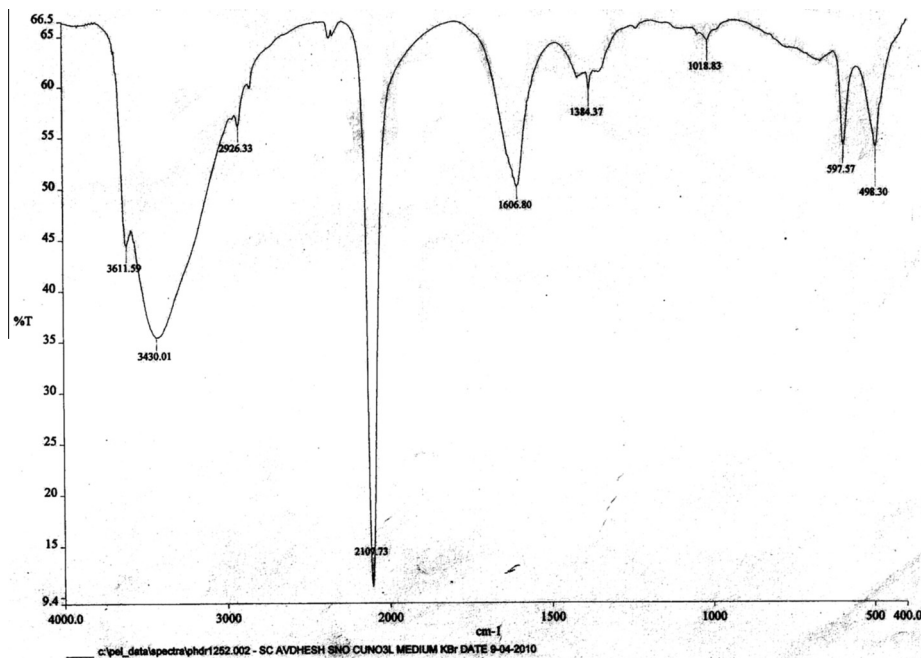


Figure 5 The IR spectrum of Cu(0) nanoparticles.

## 2.2. Synthesis of ligand

To an ethanolic solution (20 cm<sup>3</sup>) of diethylenetriamine (0.002 mol), an ethanolic solution of Benzil (0.002 mol) was added. The solution was cooled to ca. 5 °C and then conc. 1–2 drops of dil. HCl were added. The mixture was stirred and refluxed for 6 h at ~60 °C. The resulting white coloured solid was washed with ethanol and dried under vacuum over P<sub>4</sub>O<sub>10</sub>. Yield: 70% (Scheme 1). Elemental analysis: (C<sub>18</sub>H<sub>19</sub>N<sub>3</sub>; Found: C 77.8; H 6.9; N 15.18; Calculated: C 77.97; H 6.8; N 15.3; HRMS: M<sup>+</sup> 271.1, M.P. 145 °C, IR

bands (KBr, cm<sup>-1</sup>): 32919(s), 3064(s), 2903(m), 1628(s), 1446(m), 1285(m), 1040(m), 973(m), 760(m), 697(m). <sup>1</sup>H NMR (CDCl<sub>3</sub>, δ): 7.1–7.7 for Ar-H, 3.9–4.1 for –NH<sub>2</sub>, 3.1–3.29 for =C–C–CH<sub>2</sub>– and 6.02 for NH (Chandra, 2011a,b).

## 2.3. Synthesis of Cu nanoparticles

Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (0.02 mol) was dissolved in 10 cm<sup>3</sup> ionized water. To this solution, a hot ethanolic solution of 10 cm<sup>3</sup> of ligand benzildiethylenetriamine (0.004 mol) was added. After

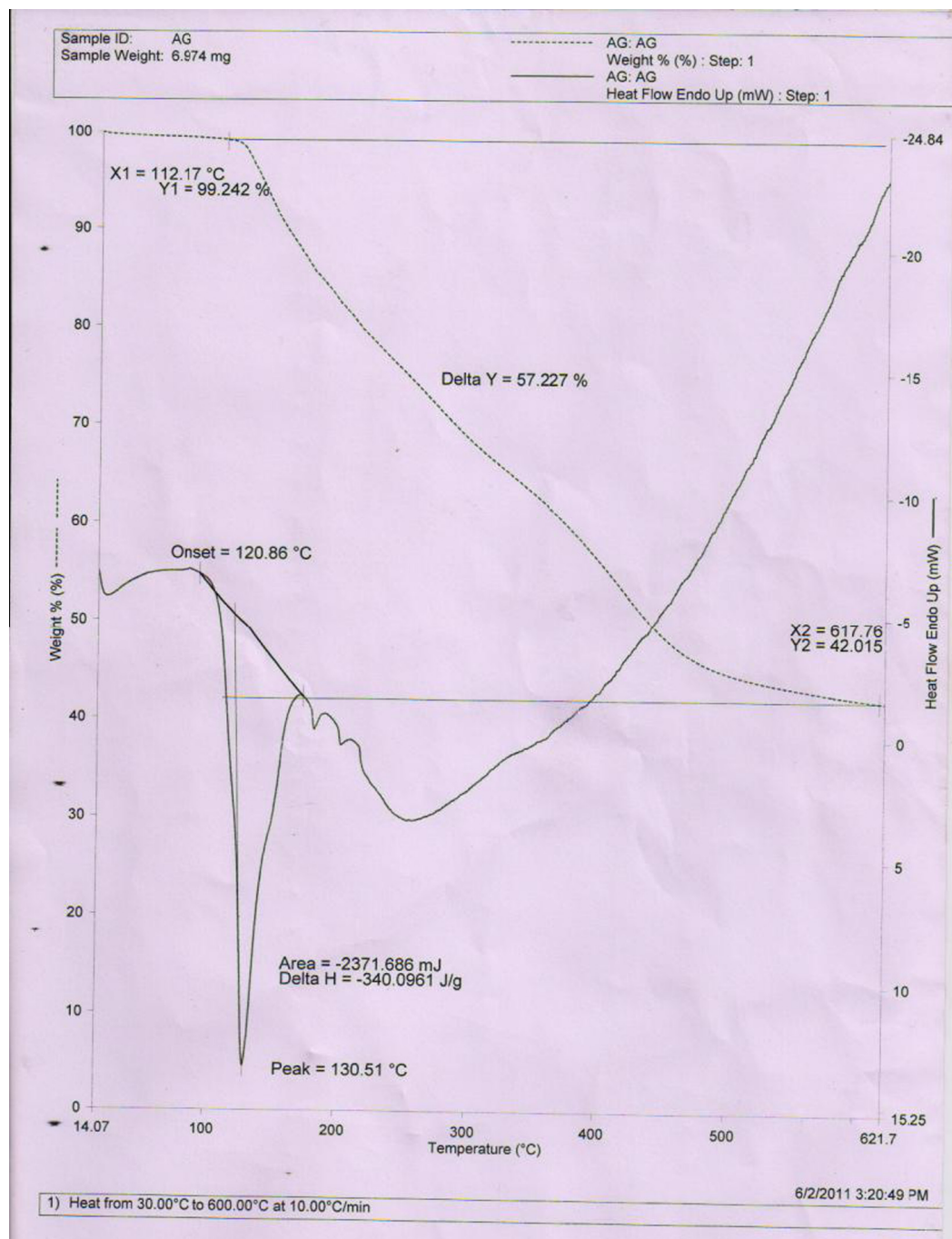


Figure 6 TGA of Cu(0) nanoparticles.

**Table 1** The effect of digestion on particles in aqueous dispersion of reducing agent.

Trial	Con. of Cu ion (M)	Con. of added reducing agent (M)	Size of Cu nanoparticles (nm)
1	0.02	0.004	15
2	0.02	0.003	28
3	0.02	0.002	40
4	0.02	0.001	63

being stirred and refluxed for (1.5–2.0 h) at moderate temperature, the colour of the solution became pale blue to black. On keeping the solution overnight, the copper nanoparticles were formed and separated out from the solution by centrifugation (6000 rpm, 10 min) to remove ligand. The particles were cleaned with acetone and dried at room temperature for 24 h.

### 3. Results and discussion

TEM images confirmed that metal nanoparticles are in nanorange and they are approximately spherical in shape. TEM images are shown in Fig. 1A and B. Average particle size was as 15–20 nm through quasi elastic light scattering data (QELS) data (Fig. 2). The average size of nanoparticles was  $15 \pm 2$  nm. The powder XRD shows that crystalline nature with peaks corresponding to Cu nanoparticles (Fig. 3). The metallic nature of Cu nanoparticles, confirmed by characteristic peak of Cu nanoparticles was on  $2\theta = 42.5$ . Crystalline size 'D' was obtained by measurement of the broadening of diffraction lines and applying the Debye-Scherrer formula (Rani et al., 2008)

$$D = \frac{0.94\lambda}{\beta \cos \theta}$$

where  $\lambda$  is the wavelength of XRD radiation,  $\beta$  is the full width at half maximum of the peak corresponding to the plane, and  $\theta$  angle is obtained from  $2\theta$  value corresponding to XRD pattern. Particle size distributions of Cu-nanoparticles in methanol, ethanol, water, acetonitrile, cyclohexane and dimethylsulphoxide are shown in Fig. 4. This shows that the ethanol is good solvent for the synthesis of Cu nanoparticles. The IR and TGA of Cu-nanoparticles are shown in Figs. 5 and 6. The effect of digestion of particles in aqueous dispersion of reducing agent on particle size is described in Table 1 which shows that more concentration of reducing agent reduces the size of the Cu nanoparticles.

### 4. Conclusion

Cu(0) nanoparticles can be prepared from reduction method which is greener and environmentally suitable, cheap and best as compared to conventional methods. The surface area to volume ratio of solid-supported metal nanoparticles (1–100) nm in size is mainly responsible for their catalytic properties. In further study we will see the catalytic and biological properties of these nanoparticles.

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