One pot facile synthesis of nanosized Copper oxide by direct precipitation method

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ABSTRACT *Nanosized metal oxide namely copper oxide has been synthesized by precipitation method and characterized by using XRD (X-ray diffraction), TEM (transmission electron microscopy) and Magnetic Measurements techniques. XRD studies show that copper oxide was formed as CuO and it has monoclinic structure. Magnetic measurements showed copper oxide has one unpaired electron and is paramagnetic in nature. The particle size of the synthesized copper oxide was determined by TEM. TEM images show that the size of particles of CuO varied from 12nm to 35nm which is in good agreement of the theoretically predicted size of nanomaterials. This method is convenient, easy and effective in comparison to the known methods of the synthesis of nanomaterials like thermal decomposition of precursors, co-implantation of metal and oxygen ions and ultrasonic spray pyrolysis.*

***Key words**: Nanomaterial, copper oxide, TEM, XRD analysis*

Introduction:

Transition metal oxides have many applications as catalyst [1-5], sensors [6-9], superconductors [10-11] and adsorbents [12-13]. Among transition metals oxides, copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer applications. It has been reported that 4% addition of CuO improves the thermal conductivity of water by 20% [14]. CuO is a semiconducting compound with a narrow band gap and used for photoconductive and photothermal applications [15]. Very few methods of synthesis of Copper oxide particles have been reported as compared to other oxides. CuO particles have synthesized using different methods like sonochemical method [16], sol–gel technique [17], one-step solid state reaction method at room temperature [18], electrochemical method [19], thermal decomposition of precursors [20], co-implantation of metal and oxygen ions [21] and ultrasonic spray pyrolysis [22]. A novel nano-sized copper oxide modified carbon paste electrode has been fabricated to determine the amikacin by cyclic voltammetry and amperometry. The oxidation current of the amikacin on nano-sized copper oxide modified carbon paste electrode was about 40 times higher than that on bulk CuO modified carbon paste electrode [23]. Nanosized copper ferrite spinel particles by a precursor approach with the aid of ultrasound radiation have been synthesized [24]. Influence of various preparation parameters on the formation of copper ferrite was studied. The preparation parameters included concentrations of precipitation agents and copper salt, sonochemical reaction time, calcination temperature and time. The reactions for the formation of $CuFe_2O_4$ were explored by analyzing X-ray diffraction data obtained under different processing conditions. Nano-powders of p-type transparent conductive copper aluminum oxide (CAO) by co-precipitation method by adding sodium hydroxide into the mixed solution of copper chloride and aluminum chloride have been synthesized [29].Co-precipitate precursors of CAO with particle size around 50-60 nm were produced after washing, filtering, and drying of the co-precipitates, nano-powders of copper aluminum oxide were produced when the dried co-precipitate precursors were calcined at temperature above 1100°C. In the present manuscript, we have synthesized CuO nanoparticles by simple aqueous precipitation method. This method involves a simple, cheap and one step process for synthesis of CuO nanaoparticles. The obtained particles of CuO have size from 12-35 nm. The synthesized nanoparticles were characterized by XRD, Magnetic susceptibility and TEM.

2. Methods and materials

2.1 Chemicals:

All chemicals used in the experiment are analytic reagent grade. Copper nitrate Cu $(NO_3)2$ was purchased from Merck, India. Ammonium hydroxide (liquor ammonia) was purchased from SRL. Deionized water was used throughout the experiment.

2.2 Synthesis of copper oxide:

500 ml of 0.1M solution of Cu $(NO_3)_2$ was taken and aqueous ammonia was added drop wise with constant stirring until the pH of the solution reached to 10. The precipitates thus obtained were filtered by

Buckner funnel and was washed several times with distilled water. The precipitates were dried in oven at 70°C for 24 hrs and were calcined at 400°C in a muffle furnace for 5 hrs. Obtained material was ground and sieved through 100 mesh size sieve.

2.3 Equipments:

The powder X-ray diffraction (XRD) was performed using X-ray diffractometer system Philips PW 11/90, with nickel filtered CuKα (l = 1.5405 Å) radiation. Magnetic measurements were done using vibrating sample Magnetometer Model 155. The transmission electron microscopy (TEM) was performed with Tecnai 20G2 under 200 KV.

Results and discussions:

3.1. X-ray studies:

X-ray diffraction of synthesized oxide is shown in Figure (1). X-ray diffraction pattern of pure copper oxide indicated that copper oxide in the form of CuO [Fig- 1]. In X-ray diffraction, some prominent peaks were considered and corresponding d-values (2.52028, 2.31782, 1.86566….) were compared with the standard [JCPDS File No. 89-5899] [Table-1].X-ray diffraction shows that metal oxide is pure CuO having monoclinic structure. Thickness of the crystal has been calculated using Scherer formula and in the range of 12nm-40nm [Table-1]

3.2 Magnetic measurements:

The magnetic moment for copper oxide is found to be 1.731 B.M. This value of magnetic moment supports the fact that the formed copper oxide is in the form of CuO with actual magnetic moment 1.732 B.M. This indicates that 1 unpaired electron is present in CuO. Thus the oxide formed is paramagnetic in nature.

3.3 TEM studies

TEM studies were performed to find out exact particle size of synthesized CuO. Figure 2 shows the TEM image of the synthesized CuO nanoparticles. It shows that the size of the obtained nanaoparticles is in the range of 12-35 nm. This is in good agreement of thickness calculated using Scherrer" s formula (12nm – 40 nm)

4. Conclusion:

CuO nanoparticles with monoclinic structure are synthesized successfully by aqueous precipitation method. From TEM study, it is found that particles are having average size of 12-35 nm. Magnetic measurements show that CuO has one unpaired electron and hence paramagnetic in nature. This method is advantageous over the existing methods of synthesis of nanoparticles because other methods require specialized instrumentation, highly skilled labour, expensive materials and methods. Therefore, the proposed precipitation method is very promising and may have extensive applications.

Table-1 X-RAY DIFFRACTION DATA FOR COPPER OXIDE

S. No.	d=λ / 2sinθ (Observed)	d=λ / 2sinθ (Reported)	I/I_0X100% (Observed)	I/I_0X100% (Reported)	Thickness of crystal size (t nm)
1.	2.52028	2.519	100	100	40
2.	2.31782	2.314	80.46	70	31
3.	1.86566	1.851	17.24	15	32
4.	1.50415	1.492	12.76	11	17
5.	1.40758	1.320	11.27	10	20
6.	1.37337	1.311	10.43	8	20
7.	2.74600	2.7485	9.60	10.21	30
8.	1.71104	1.7093	4.64	9	13
9.	1.57843	1.5799	7.70	12.1	30
10.	1.30206	1.30337	4.37	5.5	21
11.	1.26380	1.2641	6.51	4.3	12

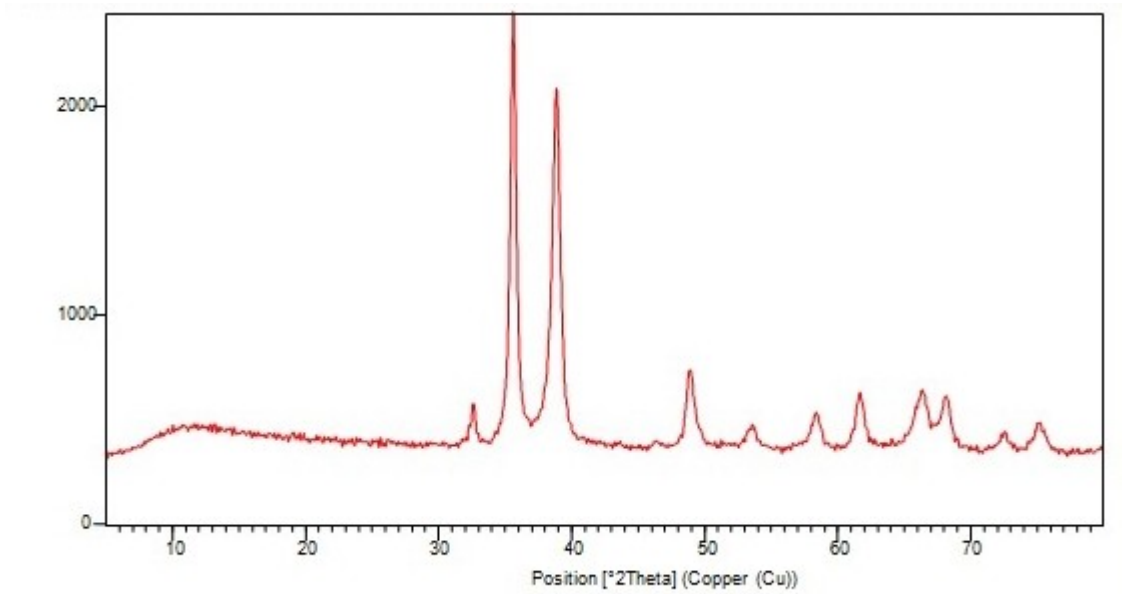
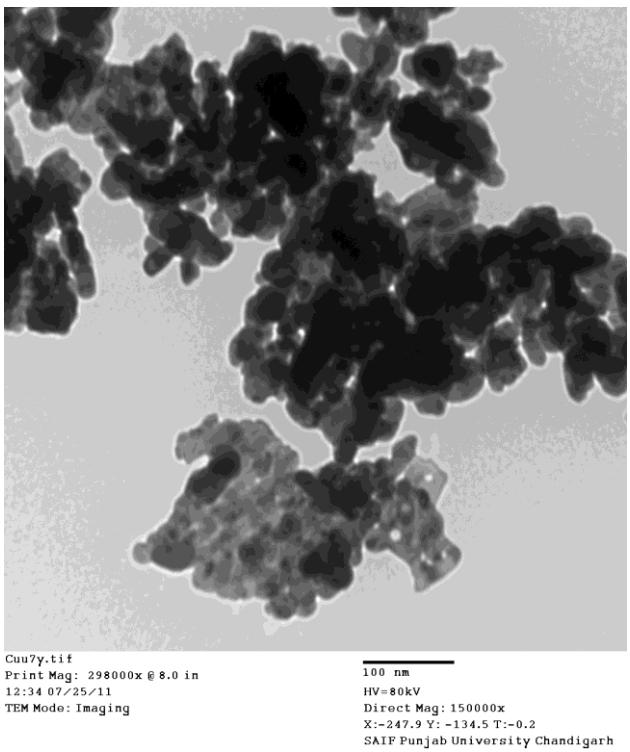
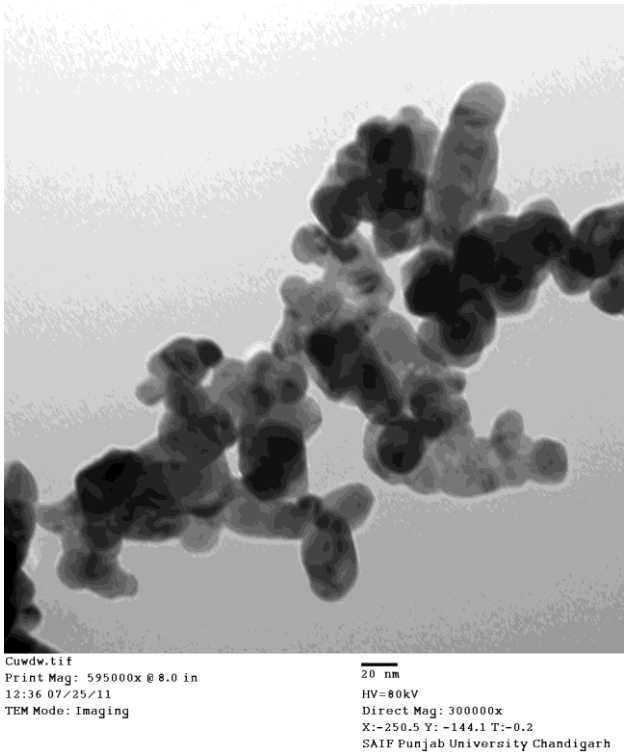


Fig.1- XRD spectra of synthesized copper oxide





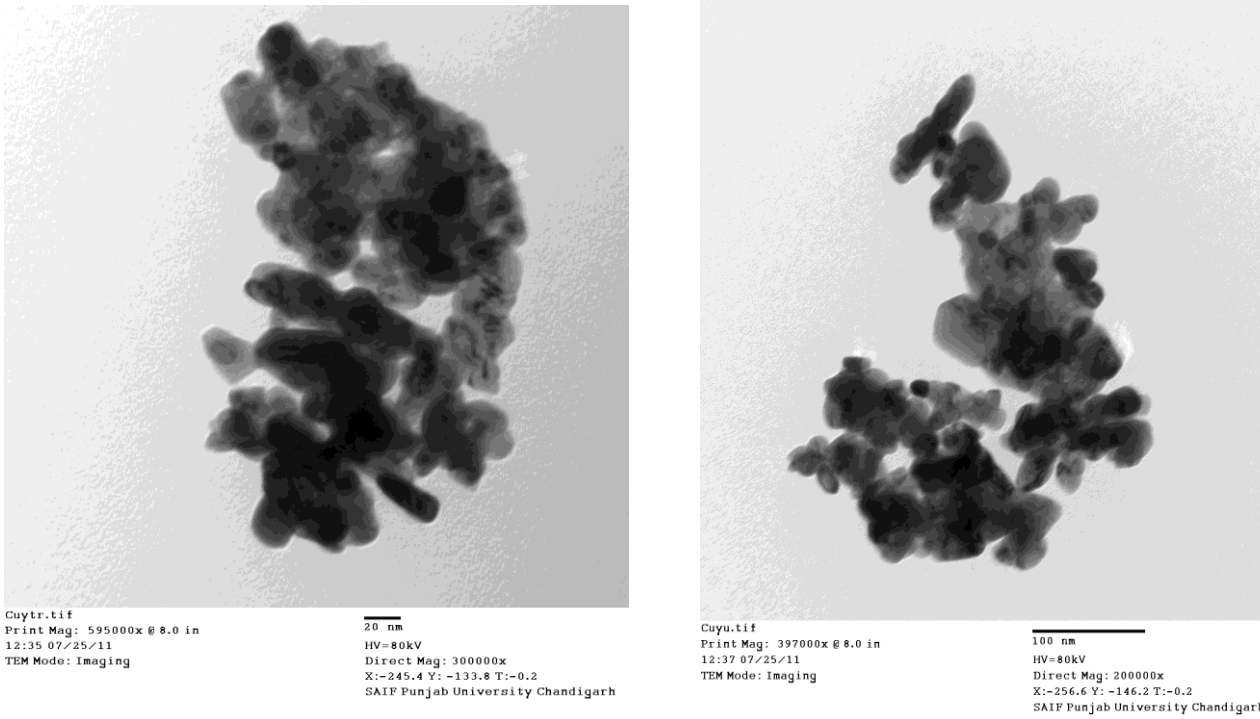


Figure 2: TEM images of copper oxide particles

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