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Synthesis of CuO via Sol-gel method and preparation of its Urea formaldehyde Nanocomposite

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ABSTRACT

The current work mainly deals with the development of new method of synthesis of CuO nanoparticles by using Sol-gel method. The obtained CuO was encapsulated in urea-formaldehyde (UF) resin by acid catalysed polymerization process. The characterization of CuO and CuO-UF made by Infra red spectroscopy (FT-IR), X- ray diffraction (XRD) and Field emission scanning electron microscopy (FESEM). FT-IR data gives the information of nature of bonds present in metal oxide CuO and on CuO-UF. Crystalline size of CuO nanoparticle calculated using Debye-Scherer formula found to be 24 nm. SEM and EDX analysis gives the diameter of particles and its elemental composition. From the interpretation of characterization data synthesis of CuO and nanocomposite of CuO-UF is confirmed.

Keywords: Nanoparticle, Nanocomposites, CuO, CuO-UF

I. INTRODUCTION

Various metal elements form a large diverse class of compounds with oxygen called oxides/metal oxides. Metal oxides covers many branches of science and technology including physics, chemistry, material sciences and engineering[1]. Due to their unique properties, nanostructures of transition metal oxides have piqued the interest of material scientists and engineers in recent years, the corresponding bulk counterparts, which in turn provide promising applications in a variety of technological fields. Preparation of high-quality nanostructures with defined, controllable size and morphology is a critical requirement for developing nanodevices and other applications such as catalysts, sensers, and pharmaceuticals[2].

Metal oxide nanoparticles such as TiO_2, SiO_2, iron oxide, zinc oxide (ZnO), gallium oxide (Ga_2O_3), nickel oxide (NiO), copper oxide (CuO), and others have been synthesized. Metal oxide nanoparticles demonstrated exciting results in terms of physical and chemical properties due to their high density and limited size; therefore, it is highly desirable to understand their various aspects in terms of synthesis, properties, and applications[3]. Cupric oxide (CuO), with a narrow band gap of 1.2 eV and a wide range of chemophysical properties, has recently gained popularity in a number of fields, including energy conversion, optoelectronic devices, and catalysts. CuO nanostructures have advanced properties when compared to bulk materials; however, the fact that these materials cannot yet be produced on a large scale is an obstacle to their use[4]. Properties of CuO nanomaterials largely depends on crystalline sizes, orientations, aspect ratios and crystalline densities. Utilities of nanomaterials restricted due to their agglomeration properties. Agglomeration minimize by composite formation[5]. Encapsulation of metal oxides on the surface of polymer is a critical in the preparation of metal oxide-polymer nanocomposite. Nanophase separated structure makes altering in the properties of both metal oxides and polymeric materials. In order to form a homogeneous nanophase separated structure, metal oxide must be uniformly dispersed throughout the polymer matrix[6].

Copper oxide has been prepared using variety of synthesis techniques, including thermal oxidation, solid-state reaction, electro-deposition, chemical bath deposition, microwave synthesis, sol–gel, solvothermal, surfactant templating approach, hydrothermal, and so on[7]. Sol-gel method offers many advantages compared to other synthesis methods, such as high homogeneity in size and shape of the nanomaterials with low consumption of raw materials and large scale production[8].

The current work was based on the cost effective highly selective method for the synthesis of CuO. Here costing of method get lowered by using citric acid as reducing agent instead of oxalic acid[9].

II. MATERIALS AND METHODS

2.1 Chemicals and Reagents:

Copper (II) acetate monohydrate $[Cu(CO_2CH_3)]H_2O$(sigma),C_2H_5OH(Xilong chemical), Citric acid, ethylene glycol, NaOH,H_2SO_4, etc.

2.2 Synthesis of CuO Nanoparticles:

All chemicals of analytical grade were used as received without further purification. Nearly about 20 g of copper acetate monohydrate dissolved in 200 ml ethanol with constant stirring till all copper ions get dissolved. To it aqueous solution of citric acid and ethylene glycol added drop wise. After six hours at 60ºC, dark green gel was obtained. The gel was dried slowly for 24 hours in oven to vaporizes excess of solvents. The product obtained was calcined at 600ºC and milled.

2.3 Preparation of CuO-UF Resin

To make composite of CuO on urea formaldehyde, 20 ml of 40% formaldehyde and 10 g of urea in standard molar ratio 2:1 in 250 ml beaker stirred magnetically for uniform mixed at 60ºC.To this homogeneous clear mixture add 100 mg of CuO and 0.5 ml of concentrated H_2SO_4 with continuous stirring there is a formation of CuO-UF nanocomposite having white color with black spots. CuO-UF nanocomposite was dried in oven at 100°C for 12 hours.

3. Characterization:

The FT-IR spectra of CuO and CuO-UF materials were obtained by Bruker Alpha Spectrometer in the 400-4000 cm^{-1} range. Powder X-ray diffraction (xrd) pattern were measured with a Benchtop X ray Diffractometer .The copper Kα rays (λ=1.5406 A°) with a nickel filter were used to cumulate the sample spectrum (workable at 40 kV, 15 mA).The XRD data was gained from a 2θ values of 10^0 to 80^0 via a flat holder in Bragg-Brentano geometry. The SEM picture of CuO and CuO-UF JSM7610FPLUS machine.

3.1 FT-IR Spectra:

3.1.1) CuO: Infrared band is used to detect the presence of particular functional group in the compound. In a figure there are three peaks. The first peak at 3347 cm^{-1} wave number was ascribed H-O-H bending and on the O-H streatching of hydroxyl group. Next peak at 1370 cm^{-1} wave number linked on the C-O asymmetric in structure of copper oxide nanoparticle. Wavenumber 419 cm^{-1} is for Cu-O bond[9].

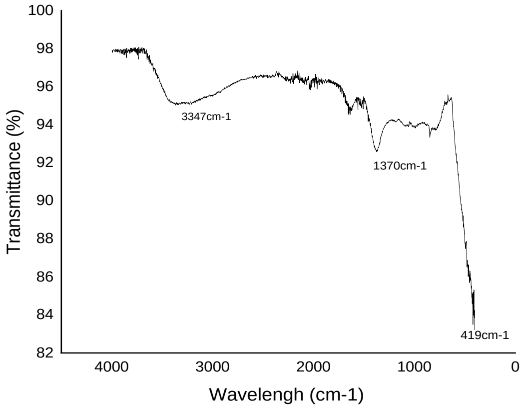

Figure.1 FT-IR of CuO

3.1.2 CuO-UF: Shifting of bands at higher wavelength gives indication of composites formation. The peaks for M-O shifts at 638 cm^{-1}.Carbonyl group peaks shifted to 1632cm^{-1}.C-N peaksappear at 2139 cm^{-1}.N-H peaks get shifted to 3735[10].

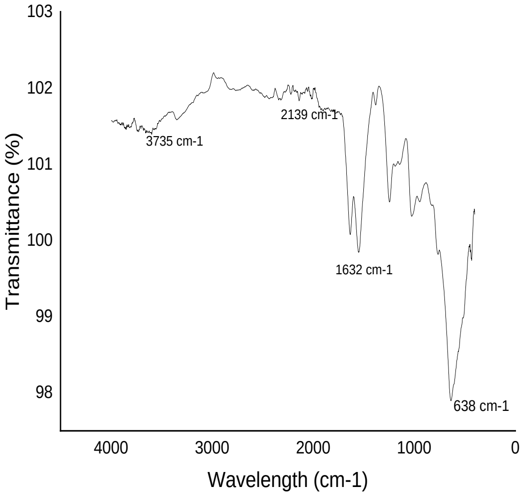

Fig.2 FTIR of CuO-UF

3.2. XRD-rays diffraction studies:

3.2.1. CuO: X-ray diffraction studies shows that product CuO is single phase with monoclinic structure. The appears of sharp peaks shows crystalline nature of CuO. The corresponding of 2θ values with hkl indicates product form is highly pure matches with jcpds file 96-901-4581[11].

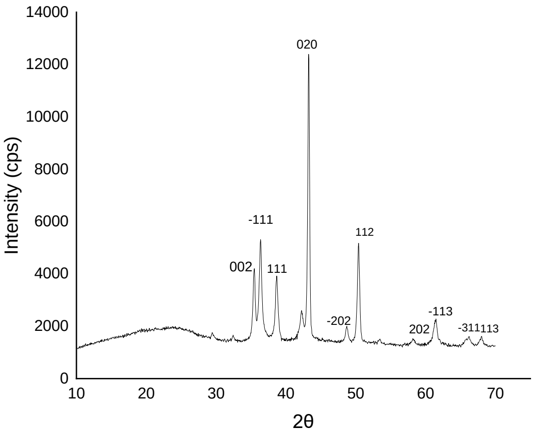

Fig.3.XRD Spectra of CuO

The crystalline size is calculated by using Debye Scherer formula as

$$D = K\lambda/\beta COS\theta$$

Where,

D=crystallites sizes(nm)

K=0.9 (Scherrer constant)

λ =0.15406nm(wavelength of X-ray source)

β= FWHM(radians)

θ= peak position in radians

The crystalline size of CuO found to be 24 nm.

3.2.2 CuO-UF: In XRD Spectra of CuO-UF shows individual nanometer layer suspended in urea formaldehyde polymer matrix exfoliated with well order encapsulation of metal oxide on urea formaldehyde. “d” spacing between the layers is significantly expanded due to composite formation[12].

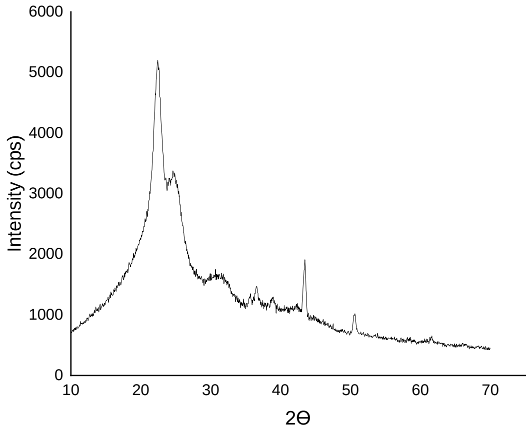

Fig.4. XRD Spectra of CuO-UF

4. SEM AND EDX: In SEM analysis of CuO shows that the particles are in nano sizes with diameter ranges from 5 to 35 nm from Gaussian graph. Fig.5.b) shows that development of pores in CuO-UF nanocomposites due to encapsulation of CuO[13]. SEM is joined with an EDX detector; where X-rays used as a signal to produce chemical information. The elemental quantification and stoichiometry ratio of CuO-NPs were confirmed by EDX analysis.

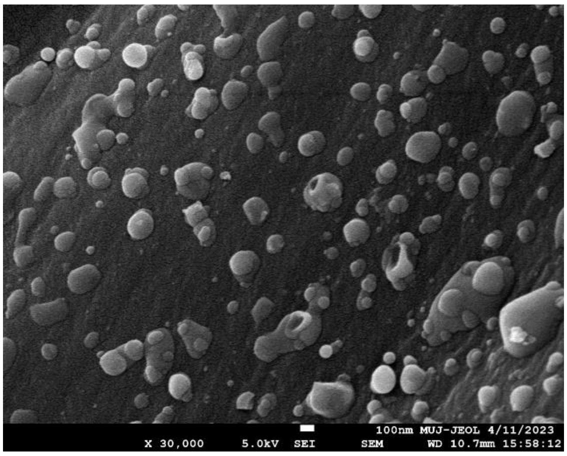

Fig.5.a) SEM of CuO

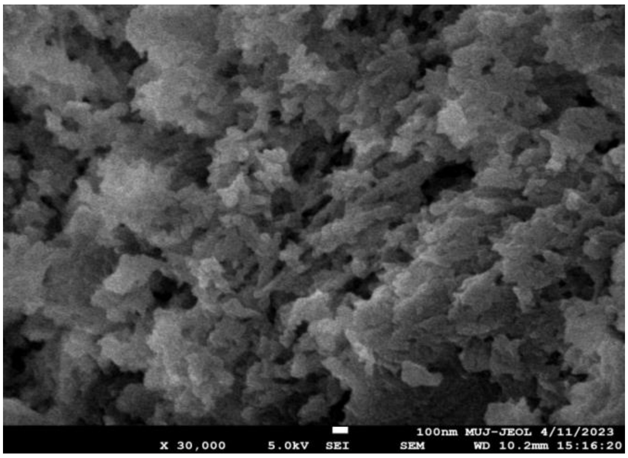

Fig.5.b) SEM of CuO-UF

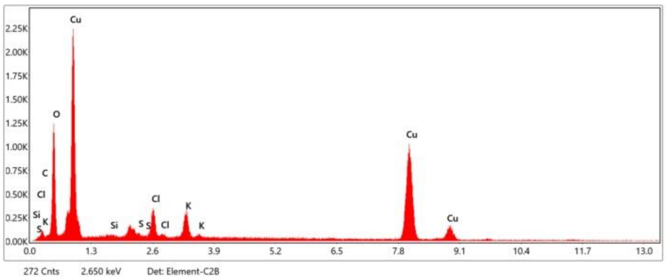

Fig.6.a) EDX of CuO

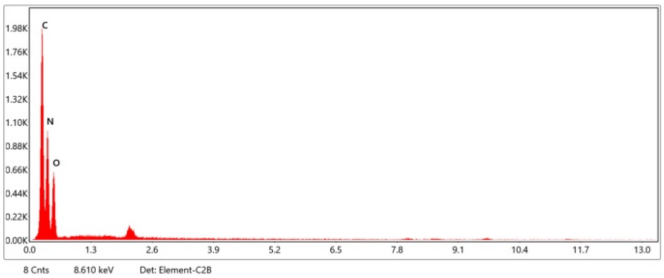

Fig.6.b) EDX of CuO-UF

EDX performed with SEM from the measurement of percentage composition of elements synthesis of CuO and CuO-UF was confirmed.

Element	Weight %	Atomic %
C	1.7	4.7
O	24.5	51.5
Cu	62.4	33

Element	Weight %	Atomic %
C	30.3	34.7
O	42.1	42.7
Cu	27.6	23.8

III. CONCLUSION AND FUTURE SCOPE

Nano sized CuO has been prepared by Sol-gel method. Here product CuO and its nanocomposite CuO-UF formed matches with the data obtained from FTIR, XRD, SEM and EDX. Sizes of product metal oxide CuO matches exactly in both characterization XRD and SEM. Accumulation of all the characterizations results confirmed the synthesis of CuO NPs and CuO-UF nanocomposites. Novelty of this work the reducing agent used in this method is citric acid which is economical and easily available. Instead of citric acid natural sources of citric acid like leman, orange extract can used for synthesis purpose which again reduces the cost of synthesis and may found to be excellent approach towards green chemistry. This study also proves the importance of UF and further open a new scenario for the application of UF as good stabilizer to develop the nanocomposites.

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