Research article

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STRUCTURAL ANALYSIS OF CuO NANOMATERIALS PREPARED BY NOVEL MICROWAVE ASSISTED METHOD

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ABSTRACT:

Nanotechnology research is increasing in the present days due to their unique properties, such as high electrical conductivity, high thermal conductivity, chemical stability, strong oxidizing power, high photo catalytic activity, toughness and ductility, high hardness and strength of materials, and good optical properties. The present work deals with CuO metal oxide nanomaterials synthesis using simple and novel microwave-assisted method. It is rapid heating technique. Copper acetate, room temperature ionic liquids (RTIL), and NaOH were used as precursor materials in the synthesis process. The structural properties were studied by XRD (average crystallite size), PSA (average particle size), SEM (morphology and size), and TEM (morphology and d-spacing).

KEY WORDS: Microwave-assisted synthesis, Room temperature ionic liquids, CuO nanomaterials, XRD, TEM

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INTRODUCTION:

Metal oxides were playing an important role in various applications in the field of nanotechnology. The properties of metal oxides in the nano range increasing and used as photo catalysis, solar cells, sensors, electronics and potential applications. Copper oxide material is having the band gap range 1.2 eV to 2.0 eV [1]. Generally CuO nanomaterials having different structures depend on their sizes, such as nanowires, nanofilms, nanoflakes, nanoribbons, and nanobelts. There are so many methods were available for the preparation of CuO nanomaterials. Some of the methods as mentioned in previous journals named as solution combustion synthesis, hydrothermal process, sol-gel technique, chemical

precipitation synthesis, thermal decomposition, electrochemical method, chemical oxidation and reduction processes, green synthesis and so on [2,3]. Copper oxide is a p-type semiconductor material, depending on their excellent physical, optical, electrical, electronics and magnetic properties it is used as a passive and energetic component in various optoelectronics applications [4,5].

The present paper deals with the CuO nanostructured materials synthesis using a novel technique named as Microwave assisted method. This is very simple, easy, and rapid heating technique. The obtained CuO nanostructured materials have been characterized by XRD, DLS, SEM and TEM for their structural properties.

MATERIALS AND METHODS:

Copper acetate and 1-ethyl, 3-methyl-imidazolium ethyl sulphate, and NaOH used as precursor materials for the preparation of CuO nanostructures. Prepare copper acetate solution initially, and add 1-ethyl-3-methyl-imidazolium ethyl sulphate drop by drop with vagarious stirring. After 5 minutes of stirring NaOH solution is added to the above solution. This solution was stirred by 10 minutes and keeps it into microwave oven. This Microwave oven was operated under atmospheric pressure at 180°C for 5 minutes, the CuO nanostructured materials was formed.

Characterization Techniques:

The obtained CuO nanostructured materials have been characterized by Brooker D8 X-ray diffractometer for average crystallite size, Horiba SZ-100 Dynamic Light Scattering is used for measuring of average particle size, morphology by HITACHI S 3400N Scanning Electron Microscope, and measuring of shape, size and d-spacing by JEM-100CXII Transmission Electron Microscope.

RESULTS AND DISCUSSION:

XRD

The average crystallite size was measured by Debye-Scherer's equation with the help of XRD patterns as shown in Figure 1. The sharp and strong peak observed at 35° position with (1 1 1) indicates that the sample is having high crystalline quality, and it is in the structure of monoclinic with lattice parameters a=0.4685 nm, b=0.3425 nm, and c=0.5130 nm, which is good agreement with JCPDS card number 45-0937.

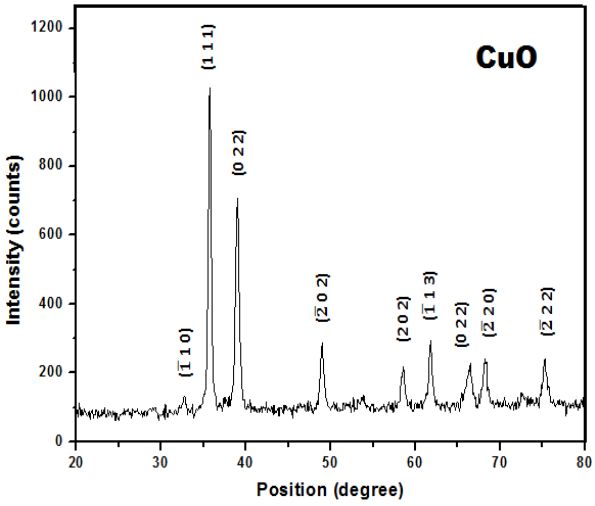

Figure 1. XRD pattern of CuO nanomaterials

The peaks position and (h k l) values mentioned in the figure 1, some of the (h k l) values shows bar on the top, it means that the negative direction of the corresponding (h k l). All the peaks are showing very sharp; it observed that there is no crystallinity impurities means the sample is having high purity. The average crystalline size was obtained 18 nm from Debye-Scherer's equation.

PSA

The average particle size was obtained by particle size analyser with the operated phenomenon of dynamic light scattering (with laser input energy of 532 nm). The mean

value of the dispersed solution has taken as average particle size as shown in Figure 2.

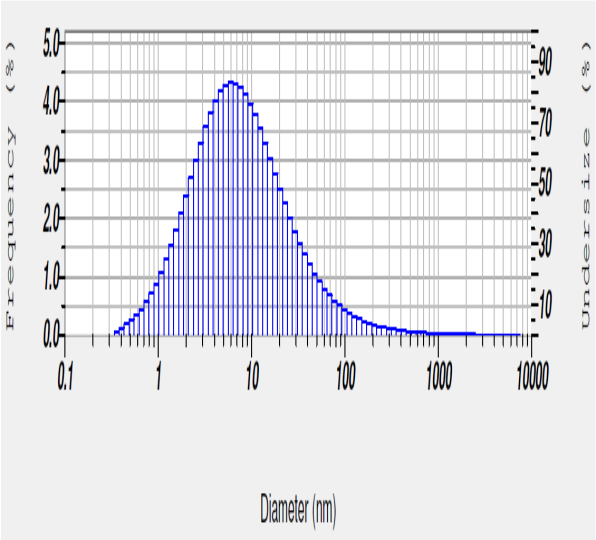

Figure 2. Particles distribution in the Particle Size Analyser

CuO nanoparticles dispersed in ethanol solution and to take the particle size distribution of the sample. The bell shape curve was given 22 nm average particle sizes. As above mentioned average crystallite size was less than the average particle sizes.

SEM

Scanning electron microscope is used for to investigate the morphology and particles sizes. The SEM image of obtained CuO nanoparticles was displayed in Figure 3.

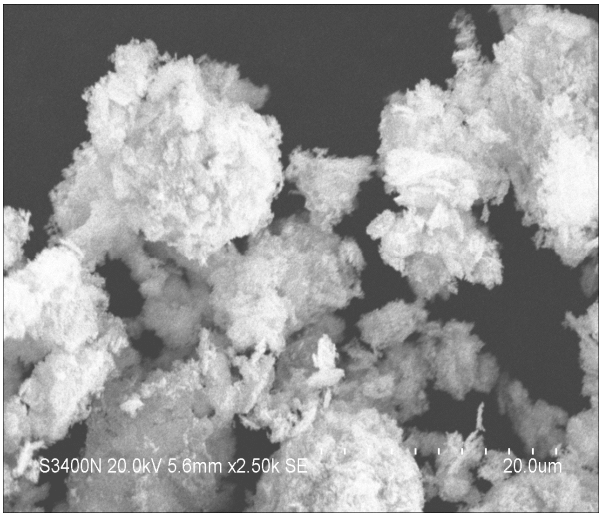

Figure 3. SEM image of CuO nanomaterials

The image shows that the CuO nanoparticles were agglomerated and showing flowers like structure. The CuO nanoparticles range is 80 nm to 100 nm. This agglomeration occurs because of the rapid heating of solution at the time of microwave synthesis.

TEM

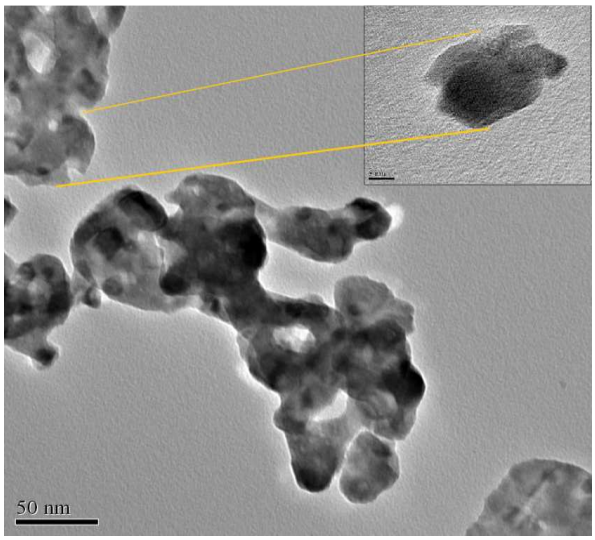

Figure 4. TEM image of CuO nanomaterials

TEM images of CuO nanoparticles as shown in figure 4. This images shows that the agglomerated CuO nanoparticles.

The d-spacing value was matched with XRD monoclinic structure data. This proves that the as prepared CuO nanoparticles were having monoclinic structure.

CONCLUSIONS:

CuO nanoparticles were prepared by novel synthesis method that is microwave-assisted method with the help of ionic liquids. The structural properties were studied by XRD, Particle size analyser, SEM, and TEM. From the XRD data the structure of the CuO nanoparticles were monoclinic and the average crystallite size was measured as 18 nm. The sharp peaks indicate that there is no impurity in as prepared sample. From the particle size analyser the distribution of particles gave the average particle size as 22

nm. SEM images shows that the obtained particles are agglomerated and flowers like structures. TEM images conforms that the structure was monoclinic and d-spacing values were nearly equal.

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