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REVIEW RESEARCH PAPER

Simple Synthesis of Copper Oxide Nanoparticles in the Presence of Extractive Rosmarinus Officinalis leaves

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ABSTRACT

In this work, copper oxide nanoparticles have been synthesized via extractive Rosmarinus Officinalis leaves by simple and economic co-precipitation method at ambient conditions which it has used $(CH_3COO)_2Cu+H_2O$ individually as Cu sources. It was found that uniform copper oxide nanoparticles have been successfully produced at various temperature, namely 400, 600, and 800 ^{0}C. Powder X-ray Diffraction analysis confirmed copper oxide nanoparticles are in monoclinic phase, which the average crystalline size estimated by using Williamson-Hall plot from the higher peak of the Powder X-ray Diffraction was about 20-30 nm for all samples. Field Emission Scanning Electron Microscopy images depict various morphologies can be successfully prepared via controlling calcination temperature and using appropriate green extractive. The study of FT-IR patterns of CuO nanoparticles confirm the formation of highly pure CuO nanoparticles. The prepared nanoparticles were subjected to the following characterizations such as, X-ray diffraction, Field Emission Scanning Electron Microscopy, and Fourier transform infrared studies.

INTRODUCTION

Nanostructured transition metal oxides (MO), a particular class of nanomaterial, are the obvious precondition for the development of various novel functional and smart materials. These transition MO nanocrystals have been attracting much attention for fundamental scientific research and various practical applications due to their unique physical and chemical properties [1] .These properties are forcefully affiliate on the sizes, shapes, compositions, and structures of the nanocrystals, which has a significant role in various applications, such as solar cell, batteries, catalytic, superconductors [2]. Various methods are available to prepare CuO nanoparticles, including microwave irradiation [3], sol–gel [4], solid-state reaction [5], precipitation-pyrolysis [6] and thermal decomposition [7]. Clearly, chemical synthesis methods lead to the presence of some toxic chemical absorbed on the surface that may have a noxious effect on the medical application. High-quality nanomaterial is a crucial challenge to material chemists for obtaining appropriate substance regard to chemical purity, phase selectivity, crystallinity, and homogeneity in particle size with controlled state of agglomeration in the feasible and low-cost process [8].

In the current essay, an attempt is made to green synthesis of CuO nanoparticles via Rosmarinus Officinalis leaves extract as a stabilizer and capping agent to control crystal growth. These green methods are low cost, fast, efûcient, and generally lead to the

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formation of crystalline nanostructures with a variety of shapes. On the other hand, calcination temperatures are investigated on the sample.

MATERIALS AND METHODS

Physical measurements

Phase identification was carried out for the as-precipitated and heat treated samples by an X-ray diffraction (XRD) method with a Rigaku D-max C III, X-ray diffractometer using Ni-filtered Cu Kα radiation. Field Emission Scanning Electron Microscope (FESEM) images were obtained on HITACHI S-4160. Fourier transform infrared (FT-IR) spectra were recorded on JASCO, 6300 spectrophotometer in in solid phase using the KBr pellets technique in the range of 3500-400 cm^{-1}.

Plant material and Extraction

Leaves of Rosmarinus Officinalis (200g) were washed in running tap water, then mixed with ethanol (50 mL) and distilled water (100mL), and last heated up to 4h on the magnetic stirrer. A small amount of the extract (100mL) is used for the synthesis.

Synthesis of CuO nanoparticles

The CuO precursor solution was prepared according to the modified co-precipitation method. All the chemical reagents used in our experiments were of analytical grade and were used as received without further purification. 50 mmol (9.982g) $(CH_3COO)_2$ $Cu+H_2O$ was individually dissolved in distilled water and then 100mL extractive Rosmarinus Officinalis leaf with 20 mL ammoniac was added into solution drop by drop under constant stirring for ~ 2h to gain homogenous mixture. The precipitates were then heated slowly up to 400 °C, 600 °C, and 800 °C in an electric furnace using alumina crucibles and maintained at the stable mentioned temperature for 2h. After calcining, the obtained products of CuO were stored in air tight container for further analysis.

RESULTS AND DISCUSSION

The correct crystallinity corresponding to those of single phase monoclinic structure (space group C2/c), with lattice parameters a= 4.6853Å, b= 3.4257Å and c= 5.1303Å, β=99.5490° (JCPDS 045-0937), was observed in the XRD pattern of powders that is represented in Fig. 1. By using the slope of the Williamson-Hall plot

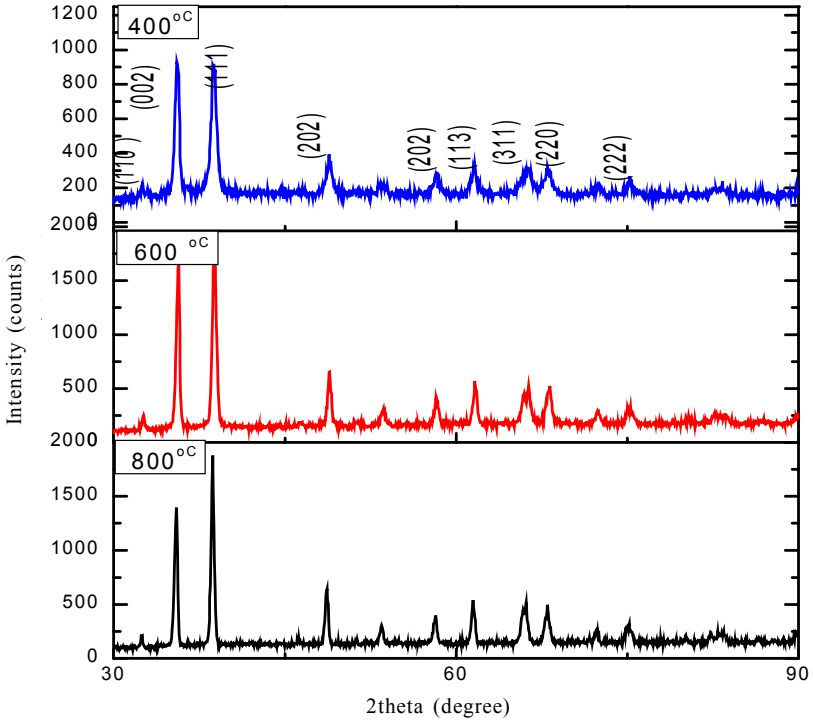

Fig. 1. XRD patterns of CuO nanoparticle at 400 ºC, 600 ºC, and 800 ºC

Table 1. Determine the size of the crystal, strain value, and dislocation density

Calcination temperature	strain value (E)	size of the crystal by Williamson-Hall (nm)	size of the crystal by Schererr (nm)	dislocation density (m^{-2})
400 ºC	0.00045	21.6	25.5	10.6×1016
600 ºC	0.00042	26.6	32.5	5.2×1016
800 ºC	0.00055	30.8	44	3.4×1016

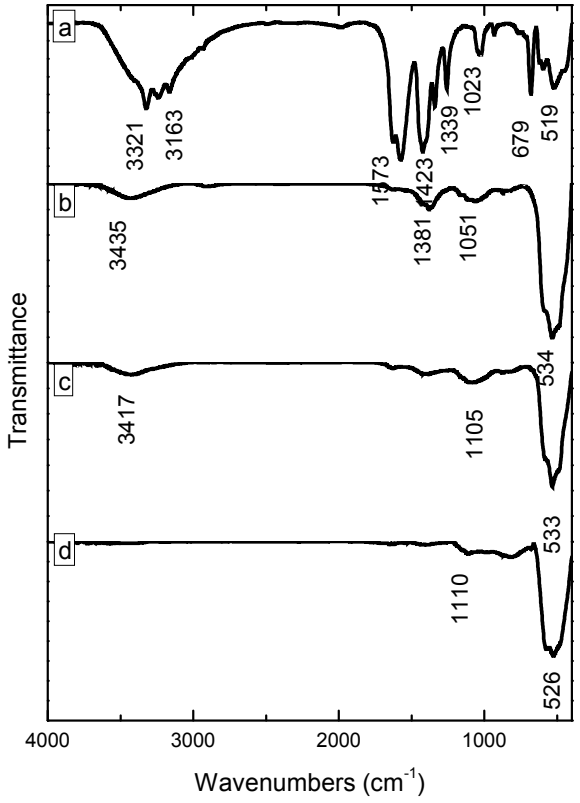

Fig. 2. FT-IR patterns of CuO nanoparticles at (a) 80 ºC, (b)400 ºC, (c) 600 ºC, and (d) 800 ºC.

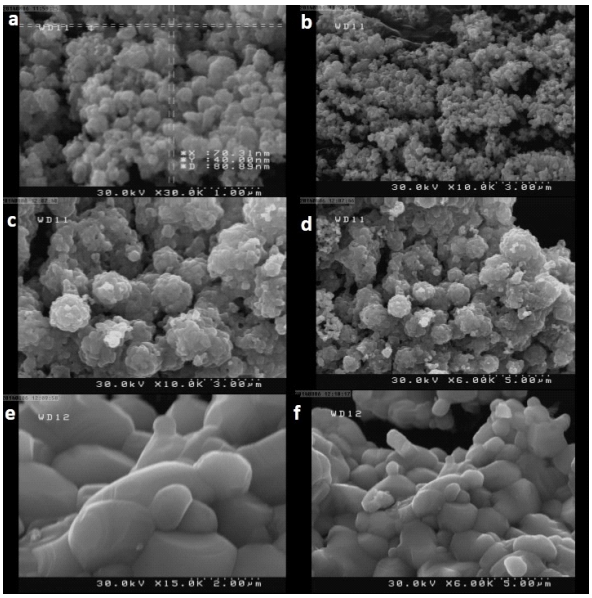

Fig. 3. SEM image of CuO nanoparticle calcinated at (a) and (b) 400 ºC; (c) and (d) 600 ºC; (e) and (f) 800ºC.

[9] based on the powder diffraction peak broadening, the strain value (ε) of CuO nanoparticles was evaluated on Table 1. Putting the crystalline size (according to Debye-Scherrer formula), α lattice constant and width of a diffraction peak at half maximum intensity â into following equation [10]:

$$\delta = \frac{15\ \beta\ cos\theta}{4\ a\ D}$$

Yields that the dislocation density (δ) in the samples have shown in Table. 1. Increasing grain boundary in the sample caused by a decrease in the grain size led to increase the dislocation density as well as the hardness of materials owing to the preventing the movement of a dislocation by the others at grain boundaries [11]. On the other hand, the crystals have grown completely and have enhanced their size by increasing the calcination temperature.

Fig. 2 shows the FT-IR spectra for the CuO nanoparticles before and after calcination at different temperatures, including 400 ºC, 600 ºC, and 800 ºC. Investigation of Fig. 2 shows that the thermal treatment resulted in the essential vanishment of the IR absorption peaks related to the organic matter, hydroxyl groups of adsorbed water and $Cu(OH)_2$. At the same time, a broad absorption peak below 600 cm-1 appeared which can be attributed to the vibrations of Cu-O, which confirms the formation of highly pure CuO nanoparticles [12].
It can be seen from SEM image (Fig. 3) that the CuO nanoparticles were synthesized in the presence extractive Rosmarinus Officinalis leaf. Images, Fig. 3 a and b calcined at 400 °C, show that the small sphere with a minimum diameter of 70-80 nm, grow radially from the nucleation points on the surface of the aggregated

nanoparticles which distribute uniformly. In Fig. 3 c and d, as the temperature increases to 600°C,agglomerated nanoparticles were obtained, self-assembly of sheet-like shapes let to the production of ûower-like microstructures. By increasing the temperature to 800 °C, various cube-shaped particles were crystallized. Clearly, the agglomeration process took place between the CuO nanoparticles capped by anthocyanin molecules in extractive Rosmarinus Officinalis leaf thanks to the presence of hydrogen bonding.

CONCLUSION

In summary, CuO nanostructures with various morphologies have been successfully prepared via a co-precipitation method with copper acetate and extractive Rosmarinus Officinalis, as surfactant is the novelty of this work which the kind of surfactant has an important effect on the size, morphology products. The results showed the nanometer scale were formed by feasible and low-cost if green synthesis. The XRD results showed that pure CuO powders were formed with the aid of this method.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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