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The analytical comparison on Cu_2O nanoparticles synthesized by the different applied methods

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ABSTRACT

Copper (II) Oxide (CuO) nanoparticles can be synthesized by many different methods. Among them, the chemical precipitation method and pulsed laser ablation technique were applied according to the simplicity of the methods and the effectiveness of the cost and the product. For the former method, by using copper sulfate pentahydrate is a precursor and ammonia solution as a stabilizing agent, CuO nanoparticles were produced. For the latter method, the pure copper target metal (immersed in the liquid environment of distilled water) was ablaze by using 1064 nm wavelength of Neodymium: Yttrium Alumina Garnet (Nd: YAG) laser for producing CuO nanoparticles. The synthesized CuO nanoparticles were characterized by XRD, SEM, FTIR, and Zetasizer (DLS). X-ray Diffraction (XRD) pattern confirmed that as-synthesized particles were CuO and Fourier Transform Infrared Spectroscopy (FTIR) was used to study the vibrational frequencies between the bonds of atoms for a synthesis CuO nanoparticles. The Scanning Electron Microscope (SEM) analysis indicated flake-like and spherelike structures for chemical precipitation and pulsed laser ablation techniques. The zetasizer was used to study the size distribution of the CuO nanoparticles.

Keywords*— Copper (II) Oxide (Cuo) Nanoparticles, (Nd: YAG) Laser, Size distribution, XRD, SEM, FTIR and Zetasizer (DLS)*

1. INTRODUCTION

Nanotechnology encompasses the production and application of physical, chemical, and biological systems at scales ranging from individual atoms or molecules to submicron dimensions. Nanotechnology is likely to have a profound impact on our economy and society in the early 21st century, comparable to that of semiconductor technology, information technology, or cellular and molecular biology. [1] Preparation and application of nanosized materials are significant scientific and industrial interests, due to their unique or improved properties, which are primarily determined by size, composition, and structures [2]. These properties are strongly related to the synthesis processes. The nanoparticles can be obtained with different synthesis methods such as physical techniques (pulsed laser ablation, vacuum vapor deposition, pulsed wire discharge, and mechanical milling), and chemical methods (microemulsion techniques, sonochemical method, electrochemical, microwave-assisted and hydrothermal) [2]. Nanomaterials now become available and useful in all the man daily life applications such as in medicine, solar cells, water purification, pharmaceutical and catalysts [3].

1.1 Application of CuO nanoparticles

Metal oxide nanoparticles are using in field of sensing, optoelectronics, catalysis and solar cells due to their unique physical and chemical properties. Among all metal oxide nanoparticles, copper oxide has gained the most interest because of its wide applications, such as in catalysis, gas sensors, magnetic storage media, batteries, solar energy transformer, semiconductors, and field emission [5]. Copper oxide is a semiconductor material and has a natural abundance of starting material (Cu). It is non-toxic and easily obtained by the oxidation of Cu [3]. Copper oxide is one of the most important metal oxides which has attracted recent research because of its low cost, abundant available as well as its peculiar properties [4]. Copper (I) oxide (Cu2O) and copper (II) oxide (CuO) are two important oxide compounds of copper. [6]. Copper oxide nanomaterials have the advantage of a lower surface potential barrier than that of metals, which affects electron field emission properties [7]. Copper (II) oxide or cupric oxide (CuO), is a p-type semiconductor exhibiting narrow bandgap, and its potential application is in nanodevices such as electronic, optoelectronic and sensing [8].

In this paper, copper (II) oxide (CuO) nanoparticles were synthesized by simple chemical precipitation and pulsed laser ablation technique. The synthesized nanoparticles were characterized by XRD, SEM, FTIR, and Zetasizer (DLS).

Table 1: The synthesis of CuO NPs with different methods results in different sizes

Preparation Method	Size (nm)
Electrochemical method	4
Sonochemical synthesis	20–30
Sol-gel techniques	7–9
Microemulsion system	5–25
Precipitation synthesis	4
Microwave irradiation	3–5

2. OBJECTIVES OF STUDY

- Understand the basic knowledge and concepts of nanomaterials, nanotechnology, applications, and properties.
- Apply the research methodology related to the synthesis of CuO nanoparticles by the selected suitable methods and characterization tools.

3. RESEARCH METHOD AND MATERIALS

(a) Chemical method (chemical precipitation method)
(b) Physical techniques (pulsed laser ablation)

The analytical grade of copper sulfate pentahydrate, ammonia solution, and distilled water were used for chemical precipitation method. Magnetic stirrer, centrifugal machine, and drying oven were used for preparation. The high purity of cathode copper (99.99%) target metal, ultrasonic cleaner and fiber laser marking machine were used for pulsed laser ablation.

4. RESEARCH CONTENT

4.1 Synthesis of CuO nanoparticles by chemical precipitation method

All chemical reagent used in the experiment is analytical grade. The 0.1 M of copper sulfate solution is prepared by dissolving 2.5 g of copper sulfate pentahydrate in 100 mL of distilled water. The solution was stirred for 30 min at room temperature and 40 mL ammonia solution (25%, 2 M) is added to above aqueous solution. The mixed solution was kept at constant stirring rate for 30 min. The large amount of bluish-white Cu $(OH)_2$ precipitate is formed immediately. It is centrifuged and washed 4-5 times with distilled water. The obtained precipitate was dried in an oven at 60 °C for 3 hours. Finally, the Cu (OH)2 was calcined at 700 °C for 2 hours to produce CuO nanoparticles.

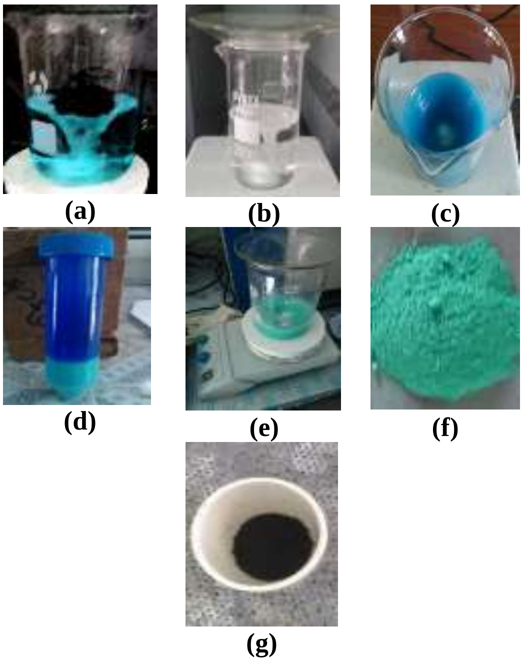


Fig. 1: Steps for Synthesis of CuO Nanoparticles by Chemical Precipitation Method (a) Preparation of Copper Sulfate Solution, (b) Preparation of Ammonia Solution, (c) Precipitation Reaction for CuO at pH 9, (d) Centrifugation, (e) Washing, (f) $Cu(OH)_2$ Powder, and (g) CuO Powder

4.2. Synthesis of CuO nanoparticles by Pulsed Laser Ablation Technique

CuO nanoparticles were synthesized by ablation of Nd: YAG laser, operating at the fundamental wavelength of 1064 nm, on the surface of a copper target. The laser machine is Fiber laser marking machine. The target metal was 1.5 mm in thickness with a purity of 99.99 % Cu and the size of (12 x 12 x 1.5) mm^3. It was rinsed to clean the surface by absolute ethanol and distilled water in an ultrasonic cleaner before ablation. The clean target was placed at the bottom of a glass vessel, filled with distilled water to the desired height of 4 mm above the target surface, ablated by laser power of 90 W (4.5 mJ) and pulsed frequency of 20 kHz, respectively.

During the laser ablation, plasma plume above the target surface occurred. The nanoparticles were observed indicating by light green color in colloids after a few minutes during the ablation.

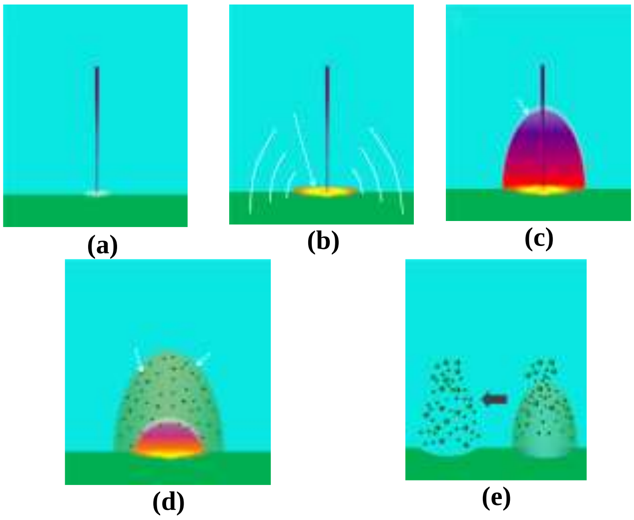


Fig. 2: Basic Process of Nanosecond Second –LAL (a) Pulse Penetration in Liquid, (b)Absorption of the Laser Pulse, (c) Expansion of the Plasma Plume, (d) Generation of the Cavitation Bubble, and (e) Collapse of the Cavitation Bubble [9]

5. RESULTS AND DISCUSSION

5.1 Characterization of CuO nanoparticles

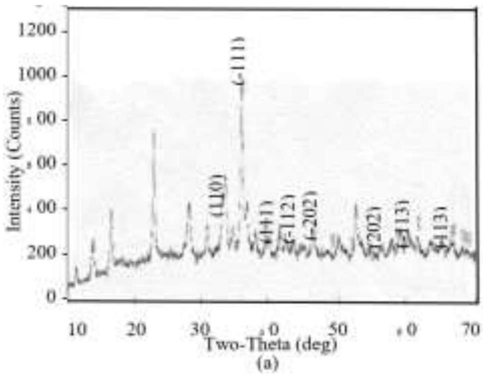
The synthesized CuO particles were analyzed by using Multiflex X-Ray Diffractometer (XRD) for confirmation of the formation of CuO nanoparticles. FTIR spectrum of CuO sample was recorded using a Perkin Elmer Spectrum Version 10.03.08 for the bonds of atoms in CuO. The morphological characterization of CuO nanoparticles was performed by Scanning Electron Microscopy (SEM). The zetasizer DLS (Nano ZS, Malvern Instruments Ltd) was used to study the size distribution of the nanoparticles. Measurement and analytical processes were carried out in Nanotechnology Research Department, Department of Research and Innovation (DRI), Yangon, Myanmar.

5.2 Characterization by X-ray Diffraction (XRD)

The X-ray diffraction pattern of nanosized CuO is shown in Fig. 3. All the XRD peaks are indexed to the monoclinic crystal system of CuO. Different peaks were observed at (2θ) = 32.3(110), 35.302(-111), 38.501(111), 46.022(-112), 48.543(-202), 51.29(112), 53.373(020), 56.630(021), 58.040(202), 61.376(-113), 65.699(022), 66.112(-311), 66.192(310), 67.746(113), 67.953(220), and 68.721(-221). It is clear that the major peaks located at 2θ = 35.302 and 38.501 are the characteristics peaks for the pure monoclinic phase of CuO nanoparticles. The sharp and narrow diffraction peaks indicate that the material has good crystallinity and no other impurities were detected at 700 °C than 400 °C as shown in figure 3(a) and (b). The crystalline size is calculated by using the Debye Scherrer formula:

$$D = 0.89\, \lambda / \beta \cos\theta \qquad (1)$$

Where D is the crystallite size, λ is the wavelength (1.54056 Å for Cu Kα), β is the full-width at half maximum of main intensity peak in radian and θ is the diffraction angle. The average grain size was found to be around 17 nm and 54 nm.



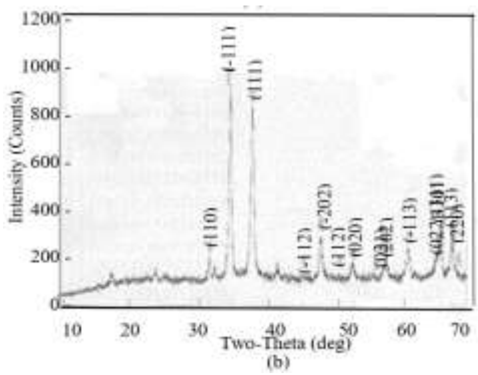
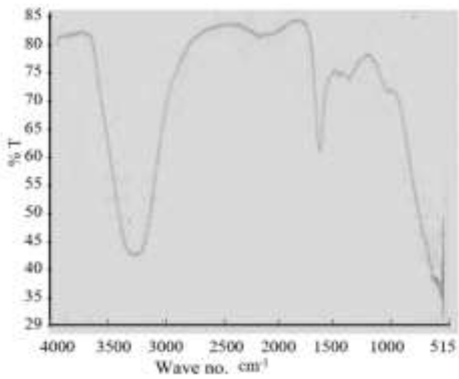


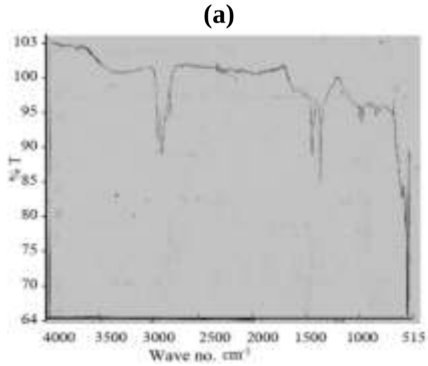
Fig. 3: XRD patterns of CuO nanoparticles by chemical precipitation method (a) at 400 °C and (b) at 700 °C

5.3 Characterization by Fourier Transform Infracted Spectroscopy (FTIR)

FTIR spectra were recorded in the solid phase using the KBr pellet technique in the range of 400-4000 cm^{-1}. FTIR spectrum (Fig.4) showed bonds at 641, 570 and 535 cm^{-1} for the sample, which can be attributed to the vibration of CuO, confirming the formation of CuO nanoparticles.



(a)



(b)

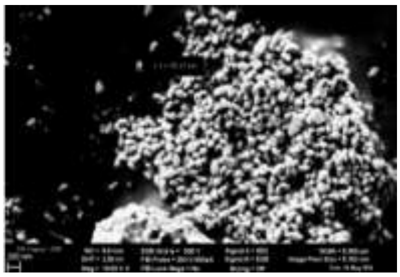
Fig. 4: FTIR spectrum of CuO NPs by (a) pulsed laser ablation in distilled water and (b) chemical precipitation

5.4 Characterization by Scanning Electron Microscope (SEM)

The morphology of CuO nanoparticles synthesized by chemical precipitation method is in flake-like structure with average thickness of 90.8 nm at 400°C as shown in figure 5(a) and in flake-like structure with average thickness of 91 nm at 700°C as shown in figure 5(b). In pulsed laser ablation technique, copper (II) oxide (CuO) nanoparticles are sphere-like structure with average the diameter of 55 nm as indicated in figure 5 (c).

(a)

(b)

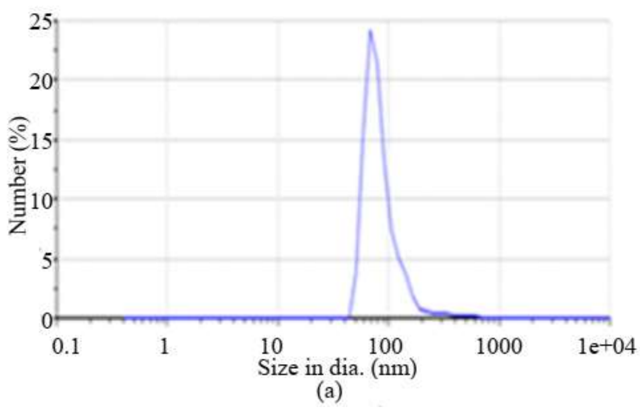


(c)

Fig. 5: SEM images of CuO Nanoparticles by (a) chemical precipitation method at 400 °C, (b) at 700 °C and (c) pulsed laser ablation technique

5.4 Particle Size Analysis

The zetasizer DLS (Nano ZS, Malvern Instruments Ltd) was applied for the size distribution of CuO nanoparticles. From this results, CuO nanoparticles synthesized by chemical precipitation method is with the range from 50 nm to 100 nm as shown in Fig. 6 (a) and pulsed laser ablation technique is with the range from 35 nm to 75 nm as shown in figure 6 (b).



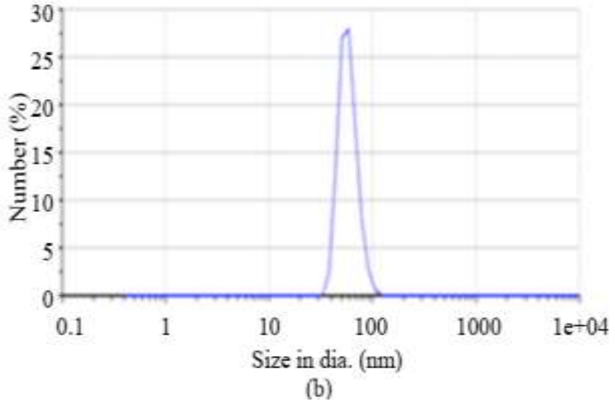


Fig. 6: Particle size distribution (PSD) of CuO Nanoparticles (a) Chemical precipitation method and (b) Laser ablation technique

6. CONCLUSION

In this paper, copper (II) oxide (CuO) nanoparticles were successfully synthesized by chemical precipitation method and pulsed laser ablation technique. Flake-like structure and sphere-like structure of (CuO) nanoparticles were confirmed by SEM results, and pulsed laser ablation technique could produce sphere-like structure. XRD results revealed the confirmation of the formation of CuO nanoparticles. FTIR spectra showed the bonds of CuO nanoparticles and confirming the formation of CuO nanoparticles. Particles with good dispersion and narrow size distribution for without agglomeration was observed in distilled water. The zeta sizer DLS results showed the average size distribution of CuO nanoparticles was 24 % for the range of 50 nm to 100 nm by chemical precipitation method and 27 % for the range of 35 nm to 75 nm by pulsed laser ablation technique. So, pulsed laser ablation technique can synthesize smaller particle size with good distribution as compared to the chemical precipitation method.

7. ACKNOWLEDGEMENT

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