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SYNTHESIS, SPECTROSCOPIC AND ULTRASONIC STUDY OF WATER-BASED COPPER NANOFLUIDS

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Abstract

This study reports the synthesis of well-dispersed, uniformly sized copper nanoparticles and their structural and acoustical characterization. Copper nanoparticles were prepared using a simple and economical chemical reduction technique, with copper sulfate pentahydrate serving as the precursor material. The synthesis was performed under varied experimental conditions, and the crystalline characteristics of the nanoparticles were examined using X-ray diffraction analysis. UV–visible spectroscopy revealed a distinct absorption maximum at 295 nm. Scanning Electron Microscopy confirmed the formation of predominantly spherical nanoparticles with average particle sizes ranging from 30 to 60 nm. Ultrasonic investigations were conducted at a frequency of 5 MHz using an ultrasonic interferometer to measure ultrasonic velocity, density, and viscosity for copper nanofluids of different concentrations at temperatures of 25 °C, 30 °C, 35 °C, and 40 °C. Additional acoustical parameters were derived from the measured data. Variations in ultrasonic parameters were analyzed to elucidate the interaction mechanisms between copper nanoparticles and the aqueous base fluid.

Keywords: Copper nanoparticles; water-based nanofluids; spectroscopic characterization; ultrasonic characterization

Introduction:

Nanotechnology has rapidly evolved into a cornerstone of modern science, enabling the design of materials with properties that differ fundamentally from their bulk counterparts. One of the most intriguing developments in this field is the creation of *nanofluids*—liquids containing dispersed nanoparticles—which exhibit enhanced thermal, optical, and acoustic behaviors compared to conventional fluids [1]. Since the pioneering work of Choi and Eastman in the 1990s, nanofluids have been investigated for their potential in energy systems, cooling technologies, lubrication, and biomedical applications [2]. Copper-based nanofluids occupy a special place in this research landscape. Copper is abundant, relatively inexpensive, and possesses excellent thermal conductivity, making it a strong candidate for improving the performance of water-based systems [3]. When copper nanoparticles are suspended in water, they can significantly modify the fluid's physicochemical and acoustical properties, opening pathways for applications ranging from advanced heat exchangers to ultrasonic sensors. However, the preparation of stable copper nanofluids is not straightforward. Nanoparticles tend to cluster due to their high surface energy, which can compromise both stability and functionality. Careful control of synthesis conditions, along with the use of stabilizers or surfactants, is therefore essential [4].

Among the various fabrication techniques, chemical reduction methods are widely favored for producing metallic nanoparticles. These approaches are cost-effective, scalable, and relatively simple to implement. In the case of copper, salts such as copper sulfate pentahydrate are commonly employed as precursors, with reducing agents like sodium borohydride or hydrazine facilitating the conversion to metallic copper [5]. Reaction parameters—including concentration, temperature, pH, and the presence of stabilizing agents—strongly influence the resulting particle size, morphology, and dispersion quality. Prior studies have shown that optimized reduction conditions yield spherical copper nanoparticles with diameters typically in the tens of nanometers [6].

Spectroscopic and microscopic techniques are indispensable for confirming nanoparticle formation and probing their structural features. UV–visible spectroscopy provides evidence of copper nanoparticle synthesis through the appearance of a surface plasmon resonance (SPR) band, generally observed between 280 and 320 nm [7]. The exact position of this absorption peak depends on particle size, shape, and concentration. X-ray diffraction (XRD) analysis further reveals the crystalline structure of the nanoparticles, enabling estimation of crystallite size and lattice parameters [8]. Complementary imaging techniques such as Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) allow direct visualization of particle morphology and distribution, thereby validating spectroscopic findings.

Beyond optical and structural studies, ultrasonic techniques provide a dynamic perspective on nanofluid behavior. By measuring parameters such as ultrasonic velocity, attenuation, density, and viscosity, researchers can infer how nanoparticles interact with the surrounding fluid [9]. At frequencies around 5 MHz, ultrasonic interferometry offers precise measurements that can be used to derive secondary acoustical properties, including adiabatic compressibility, acoustic impedance, and intermolecular free length [10]. These parameters are sensitive to nanoparticle concentration and temperature, making ultrasonic studies a powerful tool for assessing dispersion stability and microstructural relaxation processes.

METHODS AND MATERIALS

2.1 Chemicals

All chemicals used in the experiment are analytic reagent grade. Copper sulfate pentahydrate ($CuSO4 \cdot 5H2O$) of 98% purity is used. Deionized water was used throughout the experiment as a solvent. Sodium borohydride ($NaBH4$) is used as a reducing agent in the reaction. Sodium hydroxide ($NaOH$) is used to adjust pH. Ascorbic acid ($C6H8O6$) is used as an antioxidant agent for colloidal copper nanoparticles.

2.2 Synthesis

The copper nanoparticles were synthesized by a chemical reduction process using copper sulfate pentahydrate ($CuSO4 \cdot 5H2O$) as a precursor. The preparation method starts with the addition of 0.01 M copper sulfate pentahydrate solution into 0.02M solution of ascorbic acid under continuous magnetic stirring for 30 min. In the second step, 1M solution of NaOH in de-ionized water was added to adjust pH. After stirring for 30 minutes at room temperature, 0.1M solution of $NaBH4$ in de-ionized water was added under continuous stirring. The stirring was continued for 15 minutes in an

ambient atmosphere to complete the reaction. Lastly, the blue color of the initial reaction mixture turned red-brown color. After the completion of the reaction, the solution was taken from the heat and allowed to settle overnight and the supernatant solution was then discarded cautiously. The precipitates were separated from the solution by filtration and washed with deionized water and ethanol three times to take out the excessive reagents bound with the nanoparticles. Finally, the precipitates obtained are dried at room temperature. After drying, nanoparticles were stored in a glass vial for further analysis.

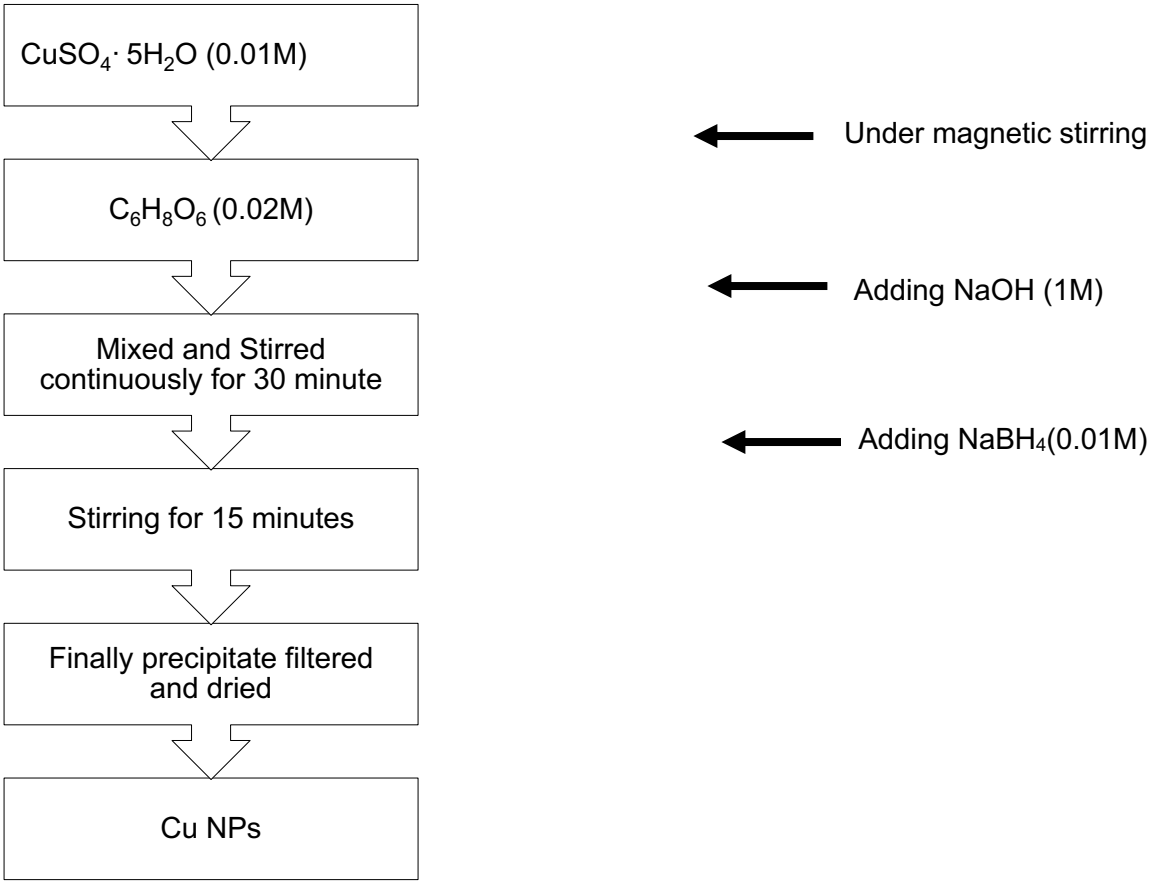

Figure1. Flow chart of synthesis of Cu Nanoparticles

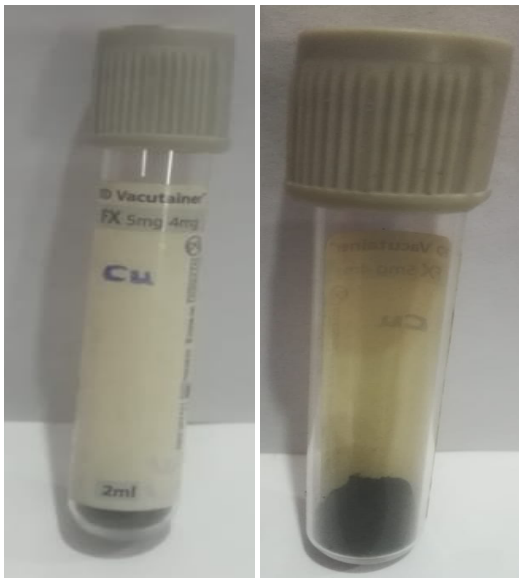

Figure 2. Prapared sample of Cu nanoparticles

2.3 Characterization

The powder X-ray diffraction (XRD) was performed using Philips Holland, XRD system PW 1710 with nickel filtered CuKα (λ = 1.5405 Å) radiation. The average crystallite size (t) has been calculated from the line broadening using Scherrer's relation: $t = 0.9\lambda / B\cos\theta$, where λ is the wavelength of X-ray and B is the full width of half maximum (FWHM). The morphology of Cu nanoparticles was studied using a scanning electron microscope (JEOL JSM 5600). The transmission electron microscopy (TEM) was performed with Tecnai 20 G 2 under 200 KV. Samples are prepared by dispersing drops of colloid on a copper grid, covered with the carbon film and the solvent is evaporated.

X-Ray Diffraction: X-ray Diffraction (XRD) is one of the most important and powerful primary techniques of characterization used by mineralogists and solid-state chemists to examine the physicochemical makeup of unknown materials. XRD is an easy tool to determine the size and shape of the unit cell for any compound. Powder Diffraction Methods are useful for Qualitative analysis (Phase Identification), Quantitative analysis (Lattice parameter determination and phase fraction analysis), etc. Diffraction pattern gives information on translational symmetry - size and shape of the unit cell from Peak Positions and information on electron density inside the unit cell, namely where the atoms are located from Peak Intensities. It also gives information on deviations from a perfect particle, if the size is less than roughly 100 – 200nm, extended defects, and microstrain from Peak Shapes and widths Peak Indexing Indexing is the process of determining the unit cell dimensions from the peak positions. It is the first step in diffraction pattern analysis. To index a powder diffraction pattern it is necessary to assign Miller Indices (h k l) to each peak. Unfortunately, it is not just the simple reverse of calculating peak positions from the unit cell dimensions and wavelength [10].

RESULTS AND DISCUSSION

3.1 XRD study

The XRD pattern of as prepared CuO nanoparticles is shown in Figure 1. It gives a single phase with

a monoclinic structure. Lattice parameters are a = 4.84 Å, b = 3.47 Å, c = 5.33 Å. The intensities and positions of peaks are in good agreement with the reported values (JCPDS file No. 05-661). No peaks of impurities are found in the XRD pattern. The peaks are broad due to the nano-size Effect. The average crystallite size of Cu nanoparticles is found to be 30-60 nm using the Scherrer formula.

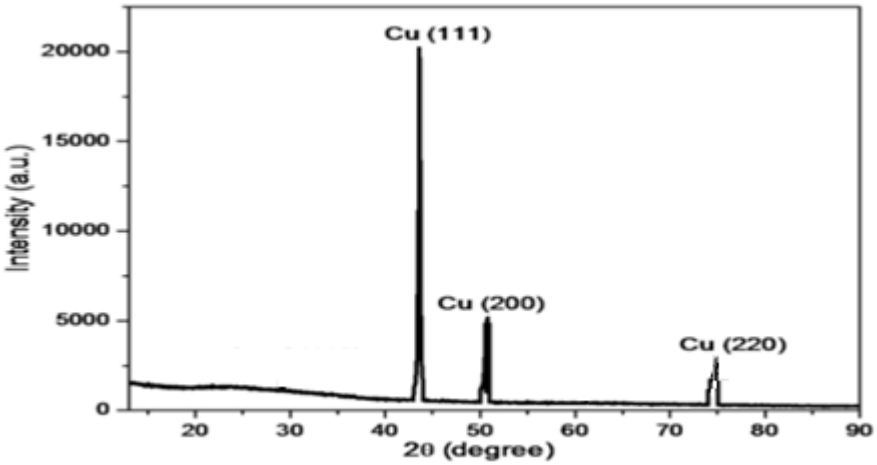

Figure3. XRD pattern of Cu Nanoparticles

3.2 Scanning Electron Microscopy:

Figure 4 shows the SEM image of as prepared Cu nanoparticles. It shows that the Cu nanoparticles are spherical. The size of the particle observed in SEM image is in the range of 30-60 nm which is in good agreement with calculated by Scherrer formula using XRD. It shows that the particles are well crystallized.

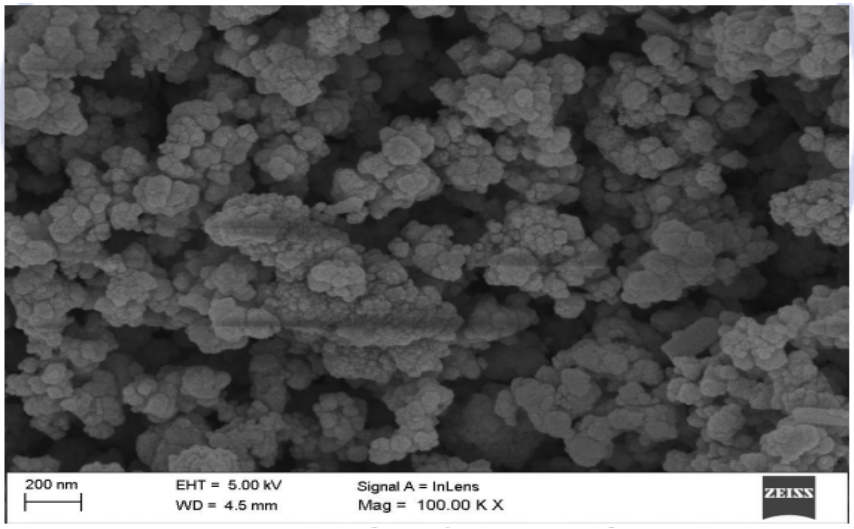

Figure4. Scanning Electron Microscopy of Cu Nanoparticles

3.3 UV-visible spectra of copper nanoparticles :

The absorption of Cu NPs was measured by UV-visible spectroscopy. The absorption band of copper

nanoparticles has been reported. UV-visible absorption spectra of Cu NPs by chemical reduction method are shown in Figure 5. The figure shows the absorption peaks at 293 nm respectively, which proves the formation of the copper nanoparticles in the solution. The initial blue-green color turned red-brown, the shifting in color is due to the surface plasmon resonance (SPR). Metals possess SPR in the visible region due to free electrons, which give such intense colors. These properties observed in Cu, Ag, and Au due to the presence of free electrons.

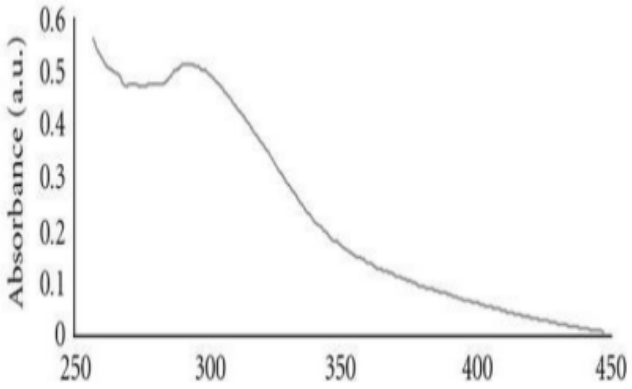

Figure 5. UV-visible spectroscopy

3.4 Ultrasonic characterization:

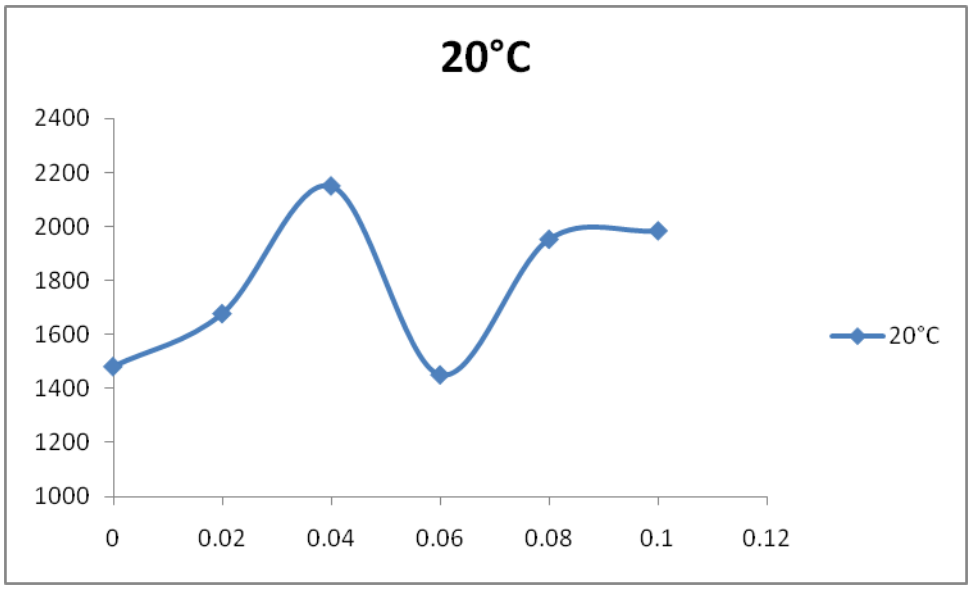

Figure 6. Ultrasonic velocity variation in water based copper Nanofluids

The figure 6. shows the ultrasonic velocity (m/s) as a function of concentration (wt%) at a fixed temperature of 20°C, showing non-monotonic behavior typical in nanoparticles suspensions. It is observed that ultrasonic velocity rises at a concentration of 0.04 wt% of copper NPs in distilled water

due to particles-fluids interaction and formation of more compact structure which enhanced ultrasonic velocity. But at higher concentration of Cu NPs in dispersion medium, there is more particles-particles interaction and dominates particles–fluids interaction [11]. Hence ultrasonic velocity decreases at higher concentration.

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