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# SYNTHESIS OF COPPER NANOPARTICLES USING ULTRASOUND AND MAGNETIC FIELD

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**Abstract**– In this paper, copper nanoparticles were synthesized using a chemical method. A constant magnetic field of 1.25 T and ultrasound were used to synthesize copper nanoparticles, and their effect on the properties and morphology of the nanoparticles was studied. The synthesized nanoparticles were characterized using X-ray diffraction (XRD) and SEM analysis. It has been proven that ascorbic acid has a good stabilizing effect, protecting copper nanoparticles from oxidation for a long period. The presence of polyvinylpyrrolidone (PVP) polymer effectively stabilizes particles during the synthesis process due to the dispersive effect of ultrasound. This stabilizing effect of PVP helps to achieve a constant particle size, preventing excessive agglomeration and promoting stable particle growth. The size of nanoparticles synthesized using a magnetic field is significantly smaller than without using a magnetic field.

**Key words**– synthesis, copper nanoparticles, ultrasound and magnetic field, morphology of the nanoparticles.

## I Introduction

Currently, one of the most common methods for obtaining copper nanoparticles is the chemical method of reduction of their salts from solutions. The main requirements for the chemical method for synthesizing nanoparticles are the use of an environmentally friendly method and the synthesis of pure copper nanoparticles that are stable to oxidation.

The synthesis of *Cu* nanoparticles is challenging due to its high susceptibility to oxidation. It is extremely sensitive to air, and the oxide phases are thermodynamically more stable [1]. Copper particles are easily oxidized by oxygen dissolved in water and contained in the air. The high rate of oxidation of *Cu* nanoparticles may limit their use [2].

Oxidation of copper nanoparticles can be eliminated if the synthesis is carried out in the presence of $CO$ or $H_2$. On the other hand, handling these gases is quite difficult, and their use is avoided whenever possible [3]. The production of pure copper nanoparticles is rare unless the entire procedure is carried out under an inert atmosphere [4, 5]. Khanna et al. [6] confirmed the preparation of pure copper nanoparticles by reducing the copper salt with sodium formaldehyde sulfoxylate in the presence of carboxylic acids. However, the stability of the resulting nanoparticles after exposure to air has not been studied.

*Cu* nanoparticles are usually protected with a blocking agent to minimize oxidation and control crystal growth by reducing the surface energy of the crystals [10]. However, blocking agents or stabilizers can significantly reduce oxidation but cannot completely prevent it due to their molecular movement [7, 8].

Some reports suggest that reaction medium $pH$ values between 9 and 10.5 affect the production of pure copper nanoparticles. Copper ions can be reduced to $Cu$, $Cu_2O$ or $CuO$ depending on the reducing ability of the reducing agent [9]. A mixture of $Cu$ and $Cu_2O$ nanoparticles has been achieved at $pH$ values up to 12. At low $pH$ values, the formation of $CuO$ and $Cu_2O$ is prevented (9,10,11,12). In addition, by varying the concentration of the stabilizing agent, the size and shape of nanoparticles can be controlled [10]. *Cu* nanoparticles were synthesized in aqueous media using $CuSO_4 \cdot 5H_2O$, polyvinylpyrrolidone (PVP) and sodium hypophosphite. The authors observed that at higher concentrations of PVP, the proliferation of *Cu* nanoparticles was prevented (9).

The production of *Cu* NPs consisting solely of $Cu(0)$, free of $CuO$ and $Cu_2O$, increased the number of applications, demonstrated higher antimicrobial efficacy, and was less cytotoxic compared to $CuO$ NPs (13). Copper nanoparticles are obtained by reduction from solutions of their salts with reducing agents such as hydrazine hydrate and sodium borohydride (have high reducing ability), which are toxic substances, in the presence of stabilizers of various natures (14-17). In recent years, there has been increased interest in en-

vironmentally friendly methods for the synthesis of metal nanoparticles using non-toxic and environmentally friendly substances. In works [18-23], copper nanoparticles are synthesized by reduction from an aqueous medium with ascorbic acid and glucose.

Excess ascorbic acid may act as a stabilizer, preventing the rapid oxidation of copper nanoparticles by oxygen (24). As a reducing agent, ascorbic acid effectively promotes the reduction of copper, even at low concentrations [25-31]. Thus, in the initial minutes of the reaction, a complex of copper with ascorbic acid is formed, which undergoes redox decomposition, resulting in the formation of ultrafine copper and oxidation products of ascorbic acid. Ascorbic acid exhibits an effective stabilizing effect, protecting copper nanoparticles from oxidation for a long time.

The size, morphology, stability, and other characteristics of the resulting copper nanoparticles are influenced by a number of factors (Fig.1)(32).

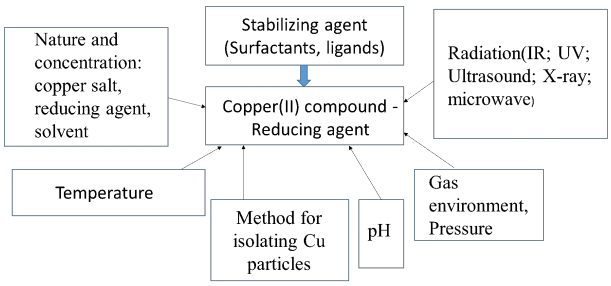



**Fig. 1**

Figure 1 reveals that the synthesis of metal nanoparticles by a chemical method is carried out in combination with external influences, such as infrared, ultraviolet, X-ray, microwave and ultrasonic radiation. The method with ultrasonic radiation is known as sonochemistry and a huge number of research works are devoted to this method (33-37). In the synthesis of nanoparticles, ultrasound is mainly used for the synthesis and dispersion of nanomaterials (38). The use of ultrasound in the synthesis of metal nanoparticles in the chemical method is explained by the following factors:

Ultrasound creates high-speed shock waves, creating cavitation in the liquid, which breaks down large particles and promotes the formation of nanoparticles.

Ultrasound promotes uniform distribution of reagents in the reaction mixture, which also contributes to the formation of homogeneous nanoparticles. Ultrasound increases the reactivity of reagents, which accelerates the synthesis process. This is due to the fact that ultrasonic waves cause ionization of reagent molecules. Ions are more reactive than neutral molecules, so they react more easily. This also helps speed up the synthesis process. As a result of the use of ultrasound in the synthesis of metal nanoparticles using the chemical method, it is possible to obtain nanoparticles with high purity and homogeneity.

Figure 1 lists all the external influences used in the chemical method, but research work devoted to the use of a magnetic field is almost never found. In work (39), the authors studied the effect of electric and magnetic fields on the synthesis of copper nanoparticles using a laser method. The result shows that the resulting nanoparticles have a mixture of $Cu$ and $Cu_2O$. Therefore, based on the analysis of literary sources, it was decided to investigate a method for producing copper nanoparticles by reducing non-toxic stabilizers in an aqueous environment with ascorbic acid, which is an environmentally friendly reducing agent, in combination with ultrasound and a constant magnetic field.

## II EXPERIMENTAL DETAILS

Three methods were used:

**Method 1.** The synthesis of copper nanoparticles was carried out without using a stabilizer.

**Method 2.** The synthesis of copper nanoparticles was carried out using the stabilizer polyvinylpyrrolidone (PVP).

**Method 3.** Synthesis of nanoparticles using a magnetic field. Materials for the experiment: copper sulfate $(CuSO_4)$, Ascorbic acid $(C_6H_8O_6)$, polyvinylpyrrolidone (PVP), distilled water.

**Method 1.** To obtain copper nanoparticles, a solution of $CuSO_4 \cdot 5H_2O$ was prepared in an aqueous solution with a salt concentration of 0.16 mol/L and a volume of 400 ml and 200 ml of ascorbic acid was added to the solution with continuous stirring on a magnetic stirrer.

In work (24), to determine the optimal ratio between the copper salt and the reducing agent ascorbic acid, solutions stabilized with polyvinylpyrrolidone were prepared with a ratio of copper sulfate: ascorbic acid = 1:50, 1:75, 1:100, 1:150 and a concentration of $CuSO_4 \cdot 5H_2O$ 0.01 mol/l. As an alkaline agent, a concentrated solution of $NaOH$ was introduced dropwise to $pH$ 10-11.

In our case, we used the ratio of copper sulfate: ascorbic acid = 1:10 in each method without using a stabilizer.

**Experimental setup:** The experimental setup is very simple. The ultrasonic transducer with a maximum power of 200 watts has 4 transducers. Each transducer has a power of 50 watts and is located in a bath of water to provide ultrasound transmission and to cool the solution. As shown in Figure 2, the flask with the solution is located above the ultrasonic transducer and the ultrasound is directed vertically upward on four sides (arrows numbered 1, 2, 3, 4 on the flask indicate the direction of the ultrasound) to ensure the uniformity of the ultrasound while creating a complex acoustic field. During the synthesis process, under this complex field, the solution on the flask constantly fluctuated.

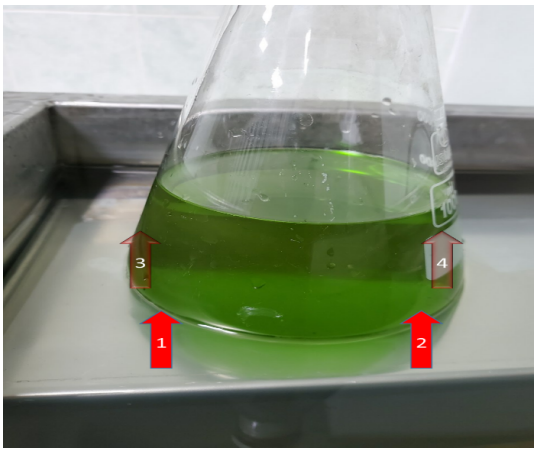



**Fig. 2:** Experimental setup

The synthesis of copper under the influence of ultrasound continued for 1 hour. Initially, the color of the solution was green; after adding the precursor, the color of the solution became yellowish. 20-30 minutes after the start of the experiment, the color turned bright brown (Figure 3). This can be explained by the fact that the reaction in the chemical reduction method took place under ultrasonic influence. It can be said that the chemical reduction method can be carried out in combination with ultrasound.

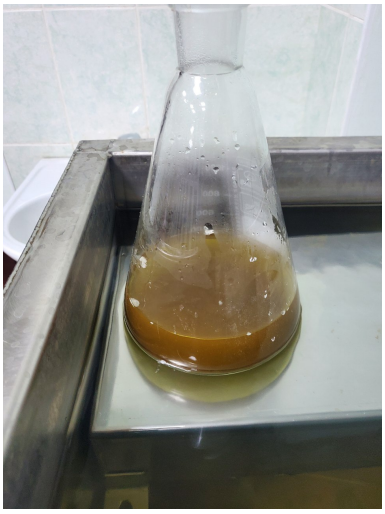


**Fig. 3:** The process of synthesizing copper nanoparticles

X-ray diffraction analysis (XRD) of the prepared sample of copper nanoparticles was carried out, $Cu-K$ X-rays with a wavelength $\lambda$ = 1.54056 A, data were taken for the $2\theta$ range from 10 to 80° with a step of 0.025°.

Surface morphology was analyzed using SEM.

## III RESULTS AND DISCUSSION

XRD analysis of the phase and elemental composition revealed that the composition of the resulting nanoparticles consists of pure copper (Figure 4).

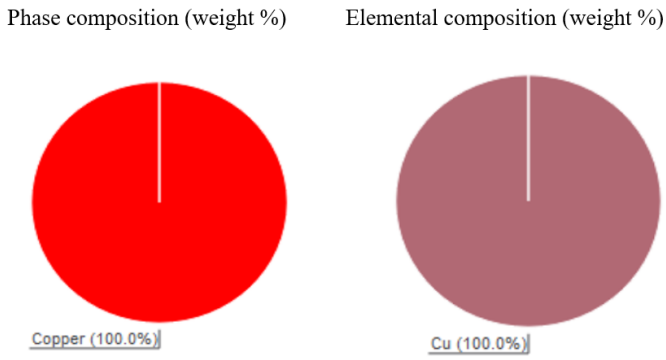



**Fig. 4:** Phase and elemental XRD analysis

The synthesis of pure copper nanoparticles is explained by the presence of ascorbic acid. The work (24) states that ascorbic acid has a good stabilizing effect, protecting copper nanoparticles from oxidation for a long period. And also in work (24) it is said that excess ascorbic acid can act as a stabilizer, preventing the rapid oxidation of copper nanoparticles with oxygen, which justifies our choice of the ratio copper sulfate: ascorbic acid = 1:10.

The diffraction pattern is shown in Figure 5. Higher peaks indicate more crystals diffracting the X-rays at that angle. The X-ray diffraction pattern has two distinct peaks, one at approximately 43 degrees and the other at approximately 50.5 degrees $2\theta$, corresponding to the (111) and (200) crystal planes of face-centered cubic (fcc) copper. These peaks are sharp, indicating clear crystallinity within the sample.

XRD analysis confirmed the presence of 81.07% amorphous structures and 18.93% crystalline structures in the synthesized samples. The high percentage of amorphous structures is due to the process in cavitation bubbles. In work (35) it is assumed that the nature of the formation of amorphous nanoparticles under ultrasonic influence is explained by the fact that the rapid process of explosion of a cavitation bubble does not allow the growth of crystallization centers, and several such centers are formed in each bubble. The growth of these centers is limited by explosion (40).

Under these extreme conditions, mixing of the constituent particles at the atomic level is achieved in the amorphous phase, so that the crystalline phase can be obtained by annealing at relatively low temperatures (35). The peaks in Figure 4 are due to the crystalline structures of the nanoparticles (18.93%).

Morphology analysis (SEM) (Figure 6) shows that the

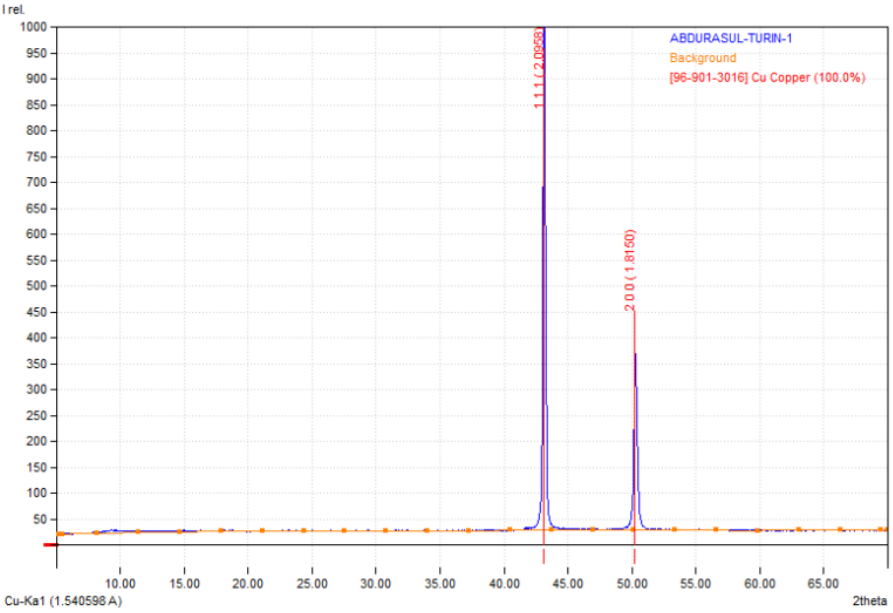



**Fig. 5:** XRD pattern of Cu nanoparticles

nanoparticles are mainly spherical in shape with some degree of faceting. Particle sizes are not uniform, but aggregation is minimal. The surfaces of the particles are not smooth, they have roughness and unevenness. The particle size may be large due to agglomeration or special synthesis conditions.

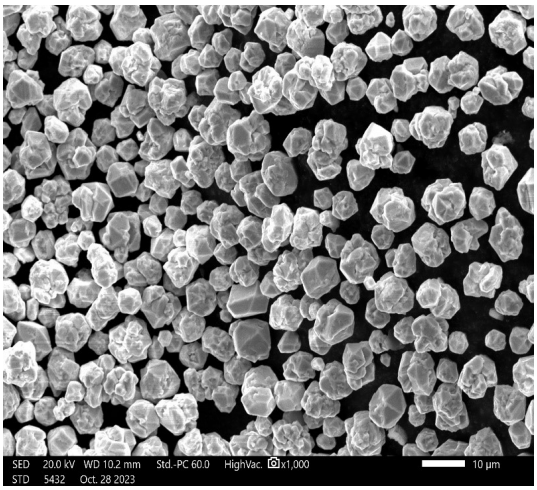



**Fig. 6:** Morphology analysis (SEM) of *Cu* nanoparticles obtained using method 1

The production of such large particle size can be prevented as follows:

**First.** To prevent the agglomeration of nanoparticles during synthesis, it is necessary to use stabilizers such as polyvinylpyrrolidone (PVP), polyacrylamide, etc., which prevent the agglomeration of particles.

**Second.** The duration of exposure to ultrasound is inversely proportional to particle size. The work (40) states that copper nanoparticles obtained as a result of a reaction of less than 20 minutes form particles with a size of about 80 nm, while if the reaction is extended to 30 minutes, the size of the resulting particles will be 45 nm. This result is believed to be due to the sufficient amount of energy supplied to the system by ultrasound after a certain time and can cause nucleation breakdown. The results will be inversely proportional in the sense that the size will increase after increasing the sonication time to 40 minutes. This is thought to be due to changes in the crystal structure caused by abundant ultrasound energy after a critical time has elapsed (41).

In our method, the synthesis of copper nanoparticles was carried out under the influence of ultrasound for 1.5 hours. Therefore, copper particles with a large size were obtained.

**Method 2.**To obtain copper nanoparticles, a solution of $CuSO_4 \cdot 5H_2O$ was prepared in a 0.1% aqueous solution of a stabilizer with a salt concentration of 0.16 mol/L and a volume of 400 ml and 200 ml of ascorbic acid was added to the solution with continuous stirring on a magnetic stirrer. The stabilizer polyvinnipyrrolidone (PVP) was added to the solution.

As in Method 1, Method 2 the ratio of copper sulfate: ascorbic acid = 1:10 was used. But the duration of the reaction under the influence of ultrasound lasted 30 minutes.

SEM analysis is shown in Figure 7.

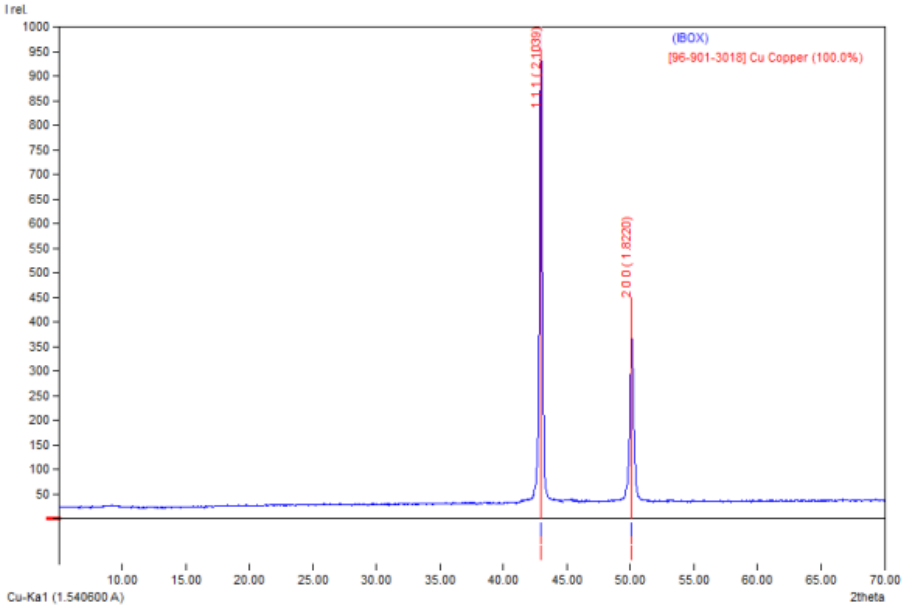



**Fig. 7:** XRD analysis of the resulting *Cu* nanoparticles by method 3

Copper nanoparticles obtained by the reduction of copper sulfate with ascorbic acid in the presence of PVP and ultrasound have a spherical shape, a uniform size distribution and a granular surface. The agglomeration of particles is insignificant, which indicates the stabilizing effect of PVP and ultrasound. The presence of PVP, which acts as a stabilizer, appears to have helped achieve a relatively constant particle size, preventing excessive agglomeration and growth. And ultrasound, in turn, did provide some dispersion, preventing particles from forming larger agglomerates.

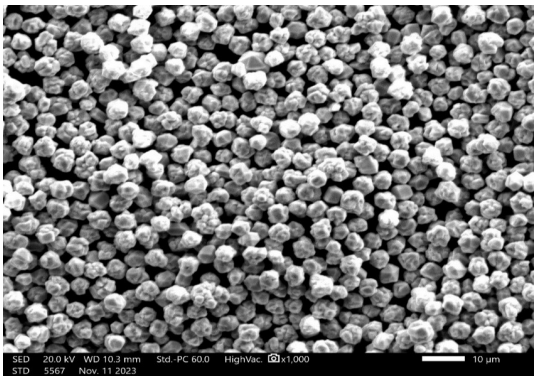


**Fig. 8:** Morphology (SEM) analysis of *Cu* nanoparticles obtained by Method 2

Comparing Figure 6 and Figure 7, it can be seen that the size of copper nanoparticles when using the stabilizer polyvinylpyrrolidone (PVP) is smaller than in the absence of PVP when synthesizing copper nanoparticles.

**Method 3.** To obtain copper nanoparticles, a solution of $CuSO_4 \cdot 5H_2O$ was prepared in an aqueous solution of a stabilizer with a salt concentration of 0.01 mol/l and a volume of 400 ml and 200 ml of ascorbic acid was added to the solution with continuous stirring on a magnetic stirrer. The stabilizer polyvinnipyrrolidone (PVP) was added to the solution. The experiment was carried out under the influence of ultrasound and a constant magnetic field for 60 minutes.

As in Method 2, Method 3 it was used the ratio of copper sulfate: ascorbic acid = 1:10.

XRD analysis of the resulting nanoparticles is shown in Figure 8.

Elemental analysis showed that the powder consisted of pure copper. XRD analysis confirmed the presence of 81.56% amorphous structures and 18.44% crystalline structures in the synthesized samples.

SEM (10 $\mu m$ resolution) analysis of the synthesized copper nanoparticles is shown in Figure 9.

Copper nanoparticles synthesized by the reduction of copper sulfate with ascorbic acid in the presence of PVP have a spherical shape, a uniform size distribution, and a somewhat textured surface. The use of a magnetic field during synthesis leads to a decrease in the agglomeration of particles and an improvement in their shape and surface. The previous SEM image (Figure 7) showed particles that were slightly more agglomerated compared to this image (Figure 9), where the application of a magnetic field appeared to result in better particle dispersion. The nanoparticles in both images (Fig-

ure 7 and 9) are spherical in shape, but in the current image (Figure 9) they have a smoother texture, which may be due to the magnetic field ordering effect during particle formation.

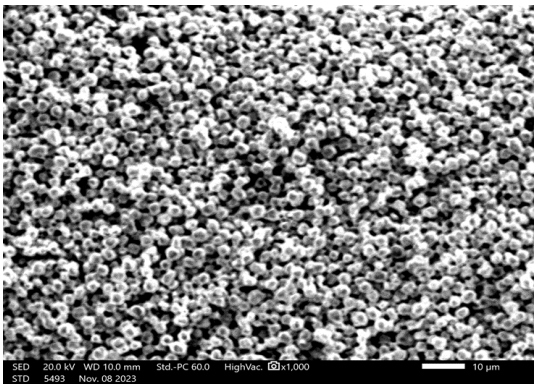



**Fig. 9:** Morphology analysis (SEM) of *Cu* nanoparticles obtained using Method 3.

From Figure 10 (SEM at 5 $\mu m$ resolution), it can be seen that several nanoparticles with the smallest size were attracted to the larger nanoparticles. The SEM image shows that the copper nanoparticles have different shapes and sizes.

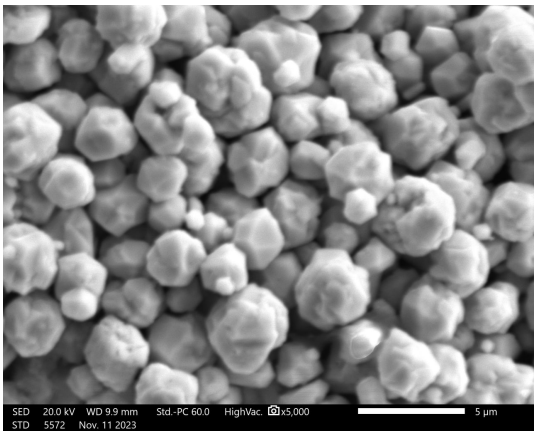



**Fig. 10:** Morphology analysis (SEM) of *Cu* nanoparticles synthesized using Method 3.

Some nanoparticles are spherical in shape, while others are irregular in shape.

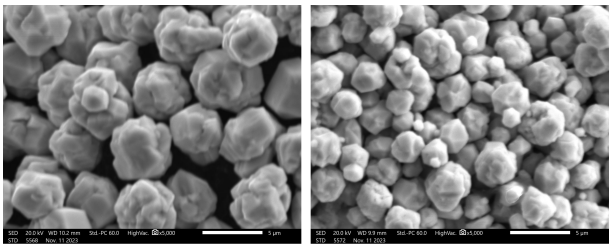
The most likely reason that some of the smallest nanoparticles are attracted to larger nanoparticles is that they have an electrical charge. Copper nanoparticles can have a positive or negative charge depending on the synthesis conditions. If nanoparticles have opposite charges, they can be attracted to each other.

The electrical charge of nanoparticles can be caused by various factors, including:

- Type of material from which the nanoparticles are made;
- Nanoparticle size;
- Concentration of the solution in which the nanoparticles are located;
- Presence of impurities in the solution.

In this case, since copper synthesis was carried out under the influence of ultrasound and a permanent magnet, it is possible that these processes led to the formation of nanoparticles with opposite charges.

From Figures 11 it is clear that the size of nanoparticles synthesized using a magnetic field is significantly smaller than without using a magnetic field. It can be assumed that the magnetic field affects the particle size.



**a)** **b)**

**Fig. 11:** SEM analysis of *Cu* nanoparticles synthesized without using a magnetic field

Figure 11(a) shows a significant degree of agglomeration. In Figure 11(b), the particles are also close together but show a higher degree of separation, which can be explained by the ordering effect of the magnetic field during the particle formation process.

Size and Uniformity: Both sets of nanoparticles have a uniform size distribution. However, Figure 11(b) shows more uniformly shaped nanoparticles, suggesting that the magnetic field may promote more uniform crystal growth.

## IV CONCLUSION

Using a ratio of copper sulfate to ascorbic acid of 1:10 makes it possible to synthesize pure Cu nanoparticles. Ascorbic acid has a good stabilizing effect, protecting copper nanoparticles from oxidation for a long period.

The presence of polyvinylpyrrolidone (PVP) polymer effectively stabilizes particles during the synthesis process due to the dispersive effect of ultrasound. This stabilizing effect of PVP helps to achieve a constant particle size, preventing excessive agglomeration and promoting stable particle growth. The size of nanoparticles synthesized using a magnetic field is significantly smaller than without using a magnetic field. Copper nanoparticles with an amorphous

structure were synthesized using ultrasound. We can conclude that our proposed method is simple and makes it possible to obtain pure copper nanoparticles with an amorphous structure.

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