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Environmentally Friendly Route for Synthesis of CuO Nanoparticles

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ABSTRACT

This study's objective is to synthesize copper (II) oxide (CuO) nanoparticles (NPs), which have numerous applications via eco-friendly route. In the study, employing curcuma herbal's ethanolic extract in the synthesis route was thought to be a good alternative for the environmentally friendly synthesis of nanoparticles because there are many benefits associated with performing so. These advantages include being affordable, conveniently accessible, easy to extract, and less susceptible to contamination. The scanning electron microscopy (SEM), energy dispersive analysis (EDX), and transmission electron microscopy (TEM) analysis were used to examine the generated particles. Additionally, UV analysis and the determination of the zeta potential of CuO NPs were performed.

CuO Nanopartiküllerinin Sentezi İçin Çevre Dostu Yol

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ÖZ

Bu çalışmanın amacı, çok sayıda uygulamaya sahip olan bakır (II) oksit (CuO) nanoparçacıklarını (NP'ler) çevre dostu bir yolla sentezlemektir. Çalışmada, zerdeçal bitkisinin etanolik ekstraktının sentez için kullanılmasının, nanopartiküllerin çevre dostu üretimi için iyi bir alternatif olduğu düşünülmüştür, çünkü bu yöntemin birçok faydası vardır. Bu avantajlar arasında uygun fiyatlı olması, kolayca erişilebilir olması, ekstaksyonun kolay olması ve kontaminasyona daha az duyarlı olması yer alır. Üretilen parçacıkları incelemek için taramalı elektron mikroskobu (SEM), enerji dağılım analizi (EDX) ve transmisyon elektron mikroskobu (TEM) analizleri kullanıldı. Ayrıca, UV analizi gerçekleştirildi ve CuO NP'lerin zeta potansiyeli belirlendi.

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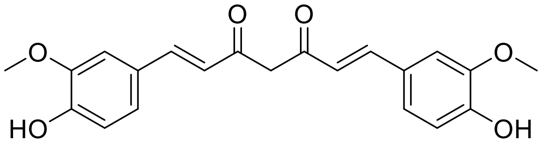
1. Introduction

The nano sized transition metal oxides have been widely investigated due to many advantages (Baladi et al., 2022; Bukhari et al., 2021; Chandrasekar et al., 2013; Janusz et al., 1999; Mohammadi et al., 2012; Yadav et al., 2022). Especially copper (II) oxide (CuO) nanoparticles have unique properties that make them useful in widely range of sectors (Bai, et al. 2022; Cuong, et al., 2022; Gupta, et al., 2022). The common usage areas of CuO nanoparticles are electronics, optoelectronics, catalysis, energy storage, biomedical applications, etc (Bai, et al., 2022; Chandrasekar, et al., 2013; Kaningini et al., 2023; Kumar, et al., 2019). CuO nanoparticles have a high surface-to-volume ratio, which means that they have a large surface area compared to their volume (Vidovix, et al., 2019). This property allows CuO nanoparticles to have excellent electronic properties, such as high electron mobility and conductivity. These properties make CuO nanoparticles useful in electronic devices where high electrical conductivity is required (Sumalatha, et al., 2023). In addition to their electrical properties, CuO nanoparticles also exhibit unique optical properties, such as high absorbance in the visible and near-infrared regions of the electromagnetic spectrum (Ghidan, et al., 2016). This property makes CuO nanoparticles useful in solar cells, where they can absorb sunlight and convert it into electrical energy. CuO nanoparticles also have a unique bandgap structure, which allows them to be used as sensors for detecting gases and other analytes. When exposed to certain gases, CuO nanoparticles undergo a change in their electrical conductivity, which can be detected and used to sense the presence of the gas (Kütük and Çetinkaya, 2022). CuO nanoparticles also exhibit a high theoretical specific capacity, which means they can store a large amount of electrical charge per unit mass. This property makes them suitable for use in energy storage devices, where high energy density is desirable. In addition to their high specific capacity, CuO nanoparticles also have a unique redox behavior, which allows them to store energy efficiently. During the charge and discharge cycle of a battery or supercapacitor, the CuO nanoparticles undergo reversible electrochemical reactions, which enables them to store and release electrical energy effectively. CuO nanoparticles also exhibit catalytic activity, which allows them to degrade pollutants through oxidation reactions(Sathiyavimal, et al., 2021). This property makes CuO nanoparticles useful for the removal of organic pollutants from water and air. Furthermore, CuO nanoparticles can be easily synthesized and functionalized with different surface groups, which allows them to be tailored for specific pollutant removal applications. Several synthesis methods of CuO nanoparticles have been used in the literature (Kaningini, et al., 2023; Zare and Moradi, 2022). For instance, the chemical precipitation approach precipitates CuO nanoparticles from a copper salt solution in the presence of a precipitant like NaOH or NH3. Sol-gel method involves the hydrolysis of a precursor solution containing a copper salt and a gel-forming agent such as ethylene glycol (Selvam, et al., 2022; Siddiqui, et al., 2021). The resulting gel is then dried and calcined to obtain CuO nanoparticles. Thermal decomposition method involves the decomposition of a precursor compound such as copper acetate or copper nitrate at high temperatures to obtain CuO nanoparticles. Hydrothermal method involves the synthesis of CuO nanoparticles under high-pressure and high-temperature conditions in an aqueous solution containing a copper salt and a hydroxide or carbonate source. The convenient and cost-effective method for producing CuO nanoparticles is the chemical precipitation method. For this purpose herbal extracts can widely use (Karuppannan, et al., 2021; Yadav, et al., 2022). Plant extract interactions with CuO nanoparticle preparations depend on a number of variables, including the type of extract used, its content, the synthesis process, and the characteristics of the resulting nanoparticles. Natural substances found in some plant extracts can function as reducing agents, stabilizing agents, or both when creating CuO nanoparticles. These substances can interact with the copper precursor to modify the final nanoparticles' size, shape, and characteristics (Kumar, et al., 2019). Some plant extracts, for instance, contain polyphenols and flavonoids that can stabilize the resulting nanoparticles and reduce copper ions. The physicochemical characteristics of the resultant nanoparticles can also be impacted by the use of plant extracts in the synthesis of CuO nanoparticles. For instance, the plant extract utilized as the reducing or stabilizing agent may have an effect on the morphology, size, and surface area of the nanoparticles. Some plant extracts have been studied for their potential application as antimicrobial agents in combination with CuO nanoparticles. The synergistic effect of the plant extract and CuO nanoparticles can enhance the antimicrobial activity of the resulting composite.

In this study we aimed environmentally friendly synthesis of CuO nano particles (NPs), for this purpose ethanolic extract of curcuma was used. Curcuma is a widely used spice in India and many other Asian nations. Curcumin, also known as "(1E,6E)-1,7-bis(4-hydroxy-3-methoxyphenyl)hepta-1,6-diene-3,5-dione," is the primary active component of curcuma (Figure 1).



(1*E*,6*E*)-1,7-bis(4-hydroxy-3-methoxyphenyl)hepta-1,6-diene-3,5-dione

Figure 1. The molecular structure of curcumin

As seen from Figure 1, the compound (curcumin) has two hydroxy groups (-OH) and two methoxy groups (-OCH_3) attached to each of the phenyl rings, which are aromatic rings with a benzene structure and two conjugated double bonds (C=C) between the heptadiene chain and the two phenyl rings, giving it a rigid and planar structure. The substance is a solid that varies in color from yellow to orange. It is only weakly soluble in water, but it is soluble in organic solvents including ethanol, methanol, and acetone. It is known to exhibit antioxidant and anti-inflammatory properties due to the presence of the phenolic groups in its structure.

The characterization was achieved via transmission electron microscope (TEM), scanning electron microscope (SEM), energy dispersive analysis (EDX), UV analysis and the zeta potential of NPs were determined.

2. Materials and methods

2.1. Synthesis of CuO NPs

The CuO NPs were produced via environmentally frienly synthesis method via curcuma extract without any reducing toxic chemicals. Fort he preparation of curcuma extract; 10 g of curcuma powder was weighted and mixed with 100 mL of ethanol (70%). It was shaken well for 5 min, and soaked for two days at room temperature approximately 25-30 °C. The produced ethanol extract was filtered using qualitative Whatman filter paper no.1 (125 mm) and stored at 4 °C.

In the synthesis of CuO NPs, copper (II) nitrate solution at the concentration of 10 mM in 50 mL ethanolic extract of curcuma was incubated at 60 °C, for 60 min. This process was achieved in a black room. Afterward, the mixture was cooled at 25 °C for 24 h; subsequently, this mixture was centrifuged 30 min at 3600 rpm. Then, the product acquired was cleaned many times with distilled water and ethanol. Finally, a darkbrown precipitate was shaped, which was desiccated for 1 h at 90 °C.

2.2. The Characterization of NPs

The synthesized CuO NPs were monitored via scanning electron microscope (SEM), it was run on JEOL JSM-5500LV. The energy dispersive X-Ray analysis (EDX) was run using an X-ray micro-analyzer (Oxford 6587, INCA) attached to SEM at 20 kV. Further, the NPs were analyzed via transmission electron microscope (TEM) samples were run on a (Thermoscientific, Talos F200i) using the carbon-coated grid (Type G 200, 3.05 μ diameter). The zeta potential of NPs were achieved via Malvern Panalytical instrument, in the analysis the dispersant was ethanol. The UV-Visible absorption spectroscopy was run on Uni cam UV-VIS spectrophotometer UV2.

3. Results and Discussion

There are various widely used approaches for morphological characterisation of NPs. TEM is a high-resolution imaging technique that allows the observation of the morphology, size, and shape of nanoparticles at high magnification (Rydz, et al., 2019). It is widely used for the characterization of NPs due to its high sensitivity and ability to produce detailed images (Huang, 2010; Luna, et al., 2016; Wang and Huang, 2016). SEM is another high-resolution imaging approach that is frequently used to evaluate the bulk and surface properties of nanoparticles and provides information on their morphology, size, and shape (Lassoued, et al., 2017; Rydz, et al., 2019; Wei, et al., 2023). EDX is a technique that is often used in conjunction with SEM to analyze the chemical composition of a sample at a microscopic level (Bibi, et al., 2019; S. Z. Mohammadi, et al., 2012). UV spectroscopy is a useful tool for the characterization of NPs because it provides information on the size, shape, concentration, surface properties, and interactions of the NPs (Luna, et al., 2016). The zeta potential is an important characteristic of NPs that refers to the electric potential at the surface of the NPs. It can provide important information about the stability and behavior of NPs in various applications (Chandransekar, et al., 2013; Meng, et al., 2016). It gives information about their surface charge, toxicity, and formulation (Ateş, 2018; Cuong, et al., 2022; Karuppannan, et al., 2021). Therefore all these techniques were used in this study.

The SEM micrograph of CuO NPs was given in Figure 2. It illustrates the presence of granular-shaped particles with a range in particle size between 0.107 and 0.190 μm. The granular shape have been widely seen in literature (Le Van, et al., 2016; Luna, et al., 2015). As can be observed in the SEM images, the agglomeration is most likely the result of electrostatic contact between layers of NPs' surface (Figure 2). Abbas Eslami et al.(Eslami, et al., 2017) discovered comparable experimental data, and they stated that a few aggregates were also observed, which could be related to aggregation during the washing process.

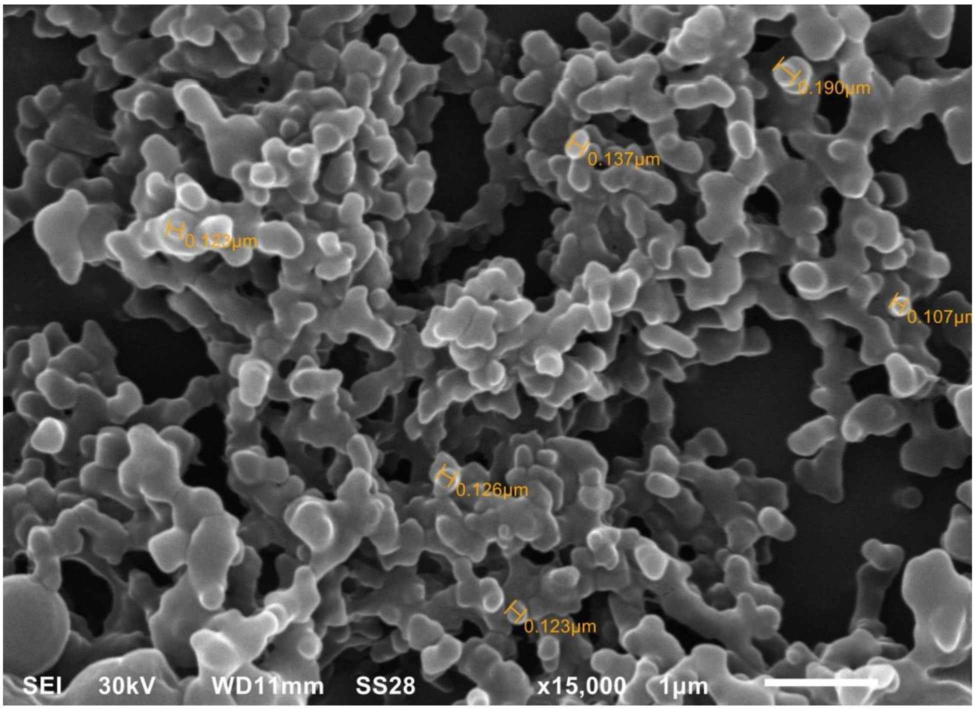


Figure 2. The SEM micrograph of CuO NPs

The EDX spectrum of CuO NPs was given in Figure 3. The oxygen (O) with copper (Cu) in the EDX spectrum shows the formation of copper oxide. The weight% of Cu and O was 79.4% and 20.6%, respectively.

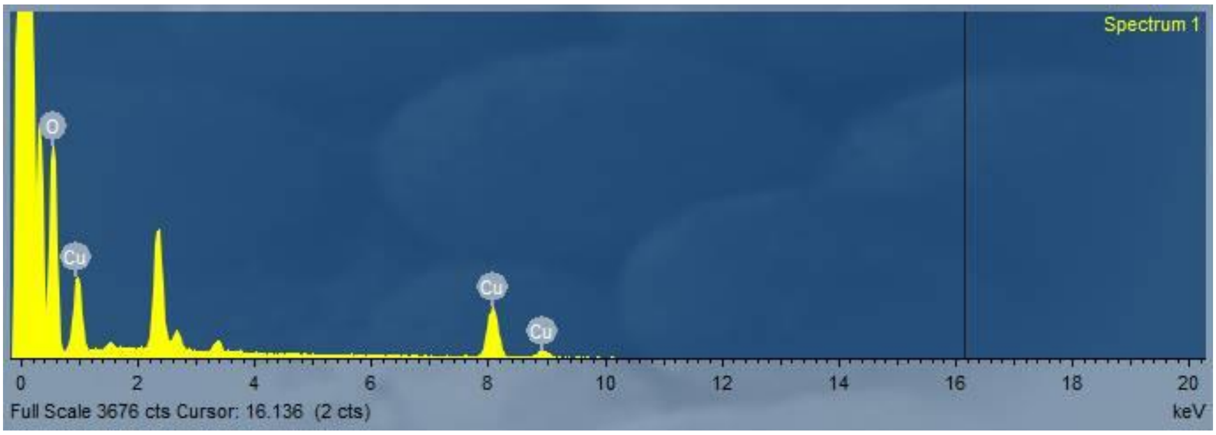


Figure 3. The EDX spectrum of CuO NPs

TEM investigation was achieved to reveal actual particle size. The largest and smallest particle sizes, as shown by the TEM result in Figure 4, were respectively close to 8.144 nm and 4.926 nm. The small-sized nanoparticle NPs have several advantages over larger-sized NPs. Small-sized NPs have a higher surface area per unit volume compared to larger-sized NPs (Siddiqui, et al., 2021; Sumalatha, et al., 2023). This increased surface area provides more opportunities for interactions with other molecules or surfaces, which can be advantageous in applications such as drug delivery, catalysis, and sensing (Bukhari, et al., 2021). Small-sized NPs exhibit enhanced reactivity due to their higher surface energy and surface area (Chandrasekar, et al., 2013). This can be advantageous in applications such as catalysis, where small-sized NPs can catalyze reactions more efficiently compared to larger-sized NPs (Yadav, et al., 2022). Smaller NPs can exhibit increased solubility in solvents or biological fluids due to their larger surface area (Kaningini, et al., 2023). They also can exhibit improved distribution due to their ability to penetrate through small pores and capillaries (Ghidan, et al., 2016).

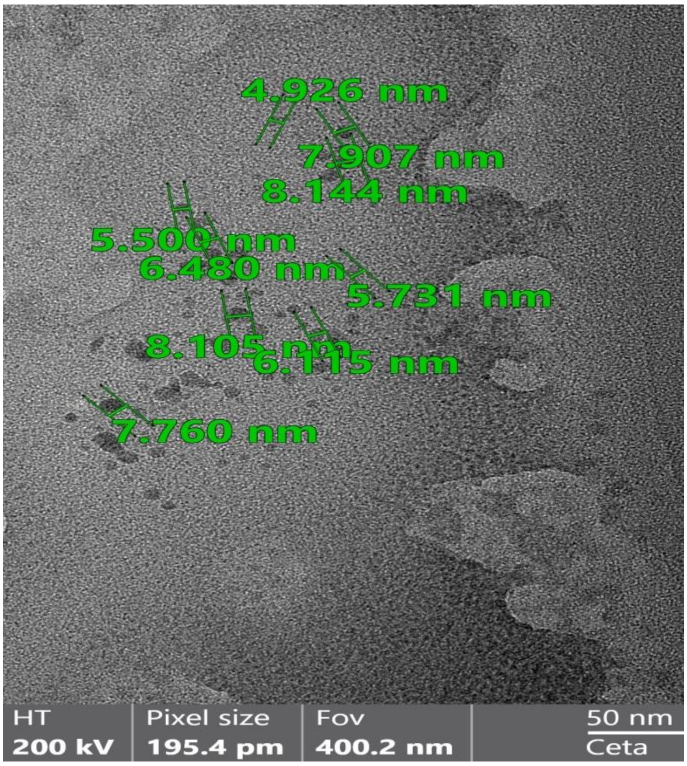
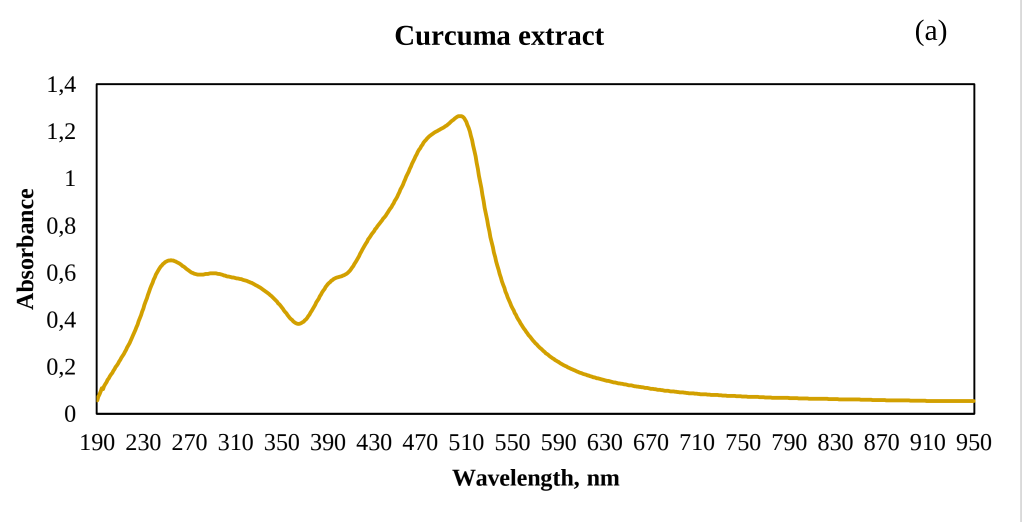


Figure 4. The TEM image of CuO NPs

Figure 5 shows the results of the UV analysis of the produced CuO NPs and curcuma extract. The interactions of curcuma or a curcumin derivative with Cu have been examined spectrophotometrically in the literature (Chittigori, et al., 2014; Khan, et al., 2020). Khan et al. investigated the role of curcumin as a reducing agent in the production of metal nanoparticles. In brief, Khan et al. dissolved a particular amount of curcumin in alkaline solution. Curcuminoxide is generated when hydroxyl ions (basis) are added to curcumin, according to the authors. As a result, the first step in the mechanism is the formation of a curcumin-metal complex ion. This combination interacted with curcumin oxide to form metalparticles with zero oxidation state (Khan, et al., 2020). In fact, during the filtration process conducted under atmospheric conditions, CuO formation was observed, despite the fact that copper ions were reduced in the solution. In Figure 5, the absorption peaks of CuO NPs are around 250 and 510 nm, corresponds to the electronic transitions between the valence and conduction bands of CuO (Sumalatha, et al., 2023). According to literature (Kumar, et al., 2019) the peak that is located around 250 nm, is due to inter-band transition of core electrons of copper metal, while that of peak around 510 nm, and corresponds band edge transition of CuO. In the study of Joshna Chittigori curcumin and copper ion interactions were seen as an increase in absorbance at 500 nm (Chittigori, et al., 2014). The absorbance shape and position are sensitive to various characteristics such as particle morphology, size, agglomeration state, and particle nature in the solution, as well as dielectric functions of the metal and the surrounding medium. As a result, the peak location is blueshifted as metal particle size decreases, whereas aggregation causes a significant intensity rise in the red/infrared area of the spectrum (Eltarahony, et al., 2018).

The zeta potential measurement of CuO NPs was given in Figure 6. Typically, the zeta potential of CuO NPs with a size around 5 nm can range from -20 mV to +20 mV. In general, CuO NPs with a negative zeta potential (below -10 mV) are more stable in aqueous solutions due to the electrostatic repulsion between particles (Ateş, 2018). On the other hand, CuO NPs with a positive zeta potential (above +10 mV) tend to aggregate and form larger particles due to the attractive forces between particles. In Figure 6, the obtained value is 8.67 eV. According to the relationship between the zeta potential value and stability of nanoparticles in the literature, particles tend to agglomerate between 0-5 mV (Ateş, 2018).



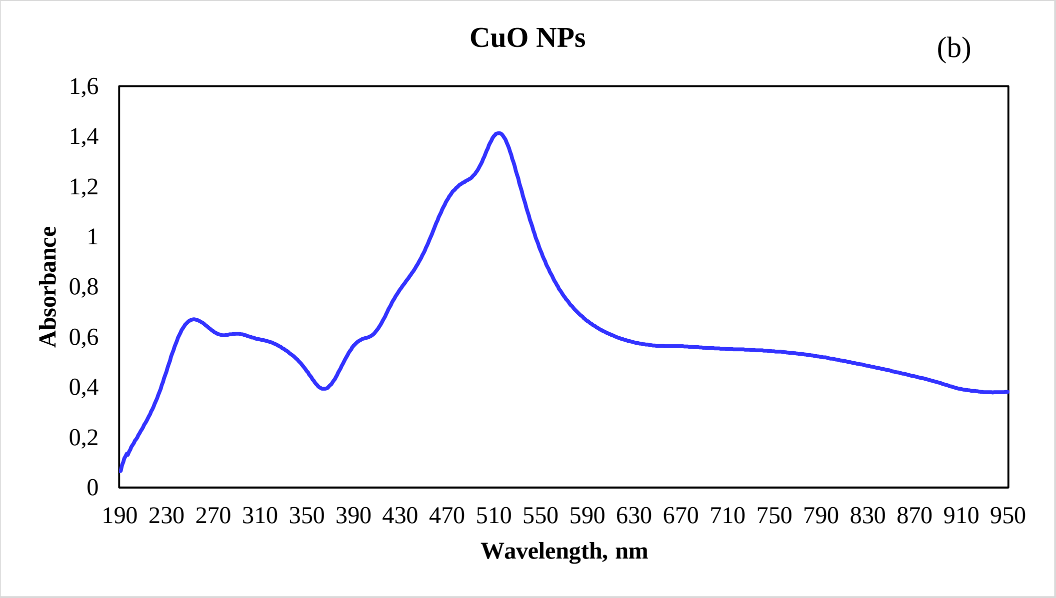


Figure 5. The UV analysis of curcuma extract (a) and synthesized CuO NPs (b)

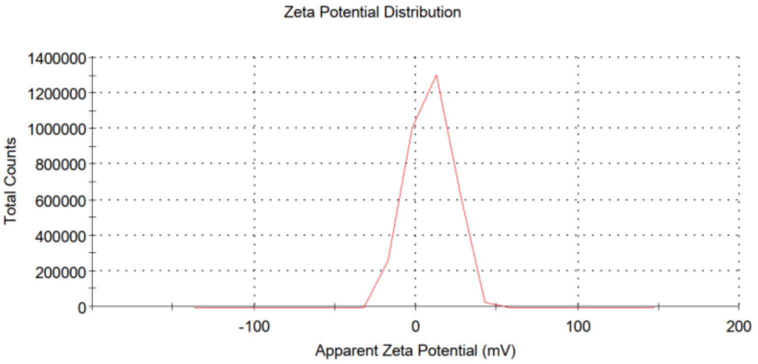


Figure 6. The zeta potential of synthesized CuO NPs

4. Conclusion

This work aimed to environmentally friendly synthesis method to generate copper II oxide nanoparticles (CuO NPs) using a herbal extract (curcuma). The powder of the widely accessible and reasonably priced curcuma plant was employed for this purpose as an ethanolic extract. SEM and TEM were used for the morphological examination, and EDX was used for the elemental composition study. The SEM micrograph of clearly demonstrates the presence of granular-shaped particles, the particles exhibit a range in particle size, measuring between 0.107 and 0.190 µm. Analyzing the SEM images in detail, it becomes evident that the observed agglomeration is likely a consequence of electrostatic contact between layers on the surface of the nanoparticles. The aggregation might occur during the washing process. TEM investigation was conducted to obtain precise information about the actual particle size of the CuO nanoparticles. TEM analysis, indicated that the largest and smallest particle sizes measured approximately 8 nm and 4 nm, respectively. It is worth noting that small-sized nanoparticles offer several advantages over their larger-sized counterparts. The average particle size, as determined by the results, was close to 5 nm, and the spherical and homogeneous forms were seen. The small-sized nanoparticles possess a higher surface area per unit volume compared to larger-sized nanoparticles. This increased surface area enables more opportunities for interactions with other molecules or surfaces, which can be particularly beneficial in applications. According to EDX results, the composition was 79.4% "Cu" and 20.6% was "O". NPs had a zeta potential of nearly 9 eV, which was supported by the literature, and UV absorption spectra showed that they had been successfully produced. The interactions between curcuma or a curcumin derivative and copper (Cu) have been further investigated by UV analysis. The role of curcumin as a reducing agent in the synthesis of metal nanoparticles have been known. The absorption peaks of CuO nanoparticles were observed around 250 nm and 510 nm, corresponding to electronic transitions between the valence and conduction bands of CuO. The peak around 250 nm was attributed to the inter-band transition of core electrons of copper metal, while the peak around 510 nm corresponded to the band edge transition of CuO. In fact, during the filtration process conducted under atmospheric conditions, CuO formation was observed, despite the fact that copper ions were reduced in the solution.

In later studies, application explorations of these particles are planned.

Conflict of Interest

There is no conflict of interest between the authors.

Contribution of authors: All authors contributed equally to the experiments and writing of the text.

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