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Sustainable Synthesis of Green Cu_2O Nanoparticles using Avocado Peel Extract as Biowaste Source

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Abstract: In recent years, there has been a significant shift towards the production of advanced nanomaterials using sustainable methods, reflecting a heightened focus on reducing environmental impact and optimizing resource utilization. This growing interest stems from the necessity to address environmental concerns and embrace eco-friendly practices in material synthesis. The primary objective of this study is to explore the eco-friendly synthesis of novel metal oxide nanoparticles (NPs) by utilizing bio-waste as a sustainable precursor. The central theme revolves around employing ultrasound-assisted techniques for Cu_2O NP synthesis, with a specific emphasis on utilizing avocado peel waste as an effective phytochemical compound for capping. Through systematic process optimization, we conducted a comprehensive assessment of the resulting NPs, delving into their chemical, thermal, and surface properties. Advanced characterization techniques, including X-ray Diffraction analysis (XRD), Transmission Electron Microscopy (TEM), Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), and Fourier-transform Infrared Spectroscopy (FT-IR), were employed to gain profound insights into the attributes of the synthesized NPs. Our experimental results conclusively demonstrate the successful synthesis of spherical Cu_2O NPs, each with a diameter of 25 ± 2 nm. This was achieved by utilizing avocado peel waste (APW) and ultrasound-assisted cavitation at room temperature. The study significantly contributes to our understanding of the potential applications of green synthesis methods, paving the way for environmentally friendly and cost-effective Cu_2O NPs.

Keywords: Sustainable synthesis; Cu_2O Nanoparticles; Bio-waste utilization, Capping agent.

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1. INTRODUCTION

Nanotechnology encompasses diverse fields, including health, optics, energy, electronics, mechanics, and the environment, where advanced nanosized materials are meticulously crafted and efficiently utilized. It involves the intricate design, fabrication, and application of nanomaterials, encompassing their physical, chemical, and mechanical properties, as well as the complex relationships between these dimensions (1,2)

When managed with a sustainable approach, nanotechnology holds the potential to introduce unique innovations and solutions across various industrial sectors, such as advanced batteries (3), nanomedicines (4), and nano-adsorbents (5), alongside advancements in technology. Current studies in nanotechnology have gained significant momentum, especially with the adoption of green approaches in the production of various nanoparticles (NPs) with a diameter of less than 100 nm and a large surface area (6,7). These approaches aim to minimize environmental impacts and promote sustainable manufacturing processes. To achieve this goal, the utilization of materials obtained from natural sources, such as sustainable and environmentally sensitive biopolymers, plant extracts, bio-wastes, and recyclable materials, has taken center stage. Biowastes, being biodegradable and environmentally sustainable residues, are noteworthy for their antimicrobial, antioxidant, and other properties, making them ideal for diverse applications in NP production, providing an environmentally friendly option for industrial use.

The distinctive features of bio-based metal/metal oxide NPs and their potential in various industrial applications have sparked a rapid growth in interest in these materials (8–10). Due to their numerous surface areas and high reactivity, these NPs are well-suited for use in sensor and catalytic applications (11). Additionally, the distinct optical and electrical characteristics of metal NPs promote their utilization in various fields, such as power generation applications. Applications for these NPs are diverse and include energy storage and conversion systems, as well as medicine delivery systems. Furthermore, due to their magnetic characteristics, metal/metal oxide NPs can be applied in medical imaging applications.

Several reports have been published on green nanomaterials composed of various nanostructures, including kiwifruit (12), *Corylus colurna* extract (13), agro-biomass wheatgrass extract (14), and MXene (15) based Cu_2O NPs. In 2021, Al-Hakkani *et al.* developed C@Cu_2O@Cu nanocomposites utilizing *Vicia faba* seeds aqueous extract as a mediator (16). In another study, Ramesh *et al.* investigated the development of novel antibacterial Cu_2O NPs utilizing *Manihot esculenta* leaf extract (17). However, there is a paucity of research focused on developing different strategies to overcome the recognized challenges associated with using avocado waste to synthesize nanostructures at low temperatures. In the pursuit of this study's objectives, our focus was directed towards the environmentally friendly synthesis of innovative metal oxide NPs derived from bio-waste. Our primary objective centered on employing ultrasound-assisted techniques for synthesizing Cu_2O NPs, with a particular emphasis on leveraging avocado peel wastes as phytochemical compounds to serve as effective capping agents. Through rigorous process optimization, we systematically assessed and characterized the chemical, thermal, and surface properties of the resulting NPs, employing techniques such as X-ray Diffraction analysis (XRD), Transmission Electron Microscopy (TEM), Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), and Fourier-transform Infrared Spectroscopy (FT-IR). This comprehensive examination provided a profound understanding of both the surface, thermal, and chemical attributes of the synthesized NPs. Experimental findings conclusively demon-strated the attainability of synthesizing Cu_2O NPs with a spherical morphology and a diameter of less than 50 nm from avocado peel waste, utilizing ultrasound-assisted cavitation at room temperature. The overarching goal of this study was to advance our comprehension of the potential applications of green synthesis methods in the fabrication of green and low-cost Cu_2O NPs.

2. EXPERIMENTAL SECTION

2.1. Materials

Avocados (*Persea americana*) were purchased from a local market in Istanbul, Türkiye. The avocado pulp (APW) exhibited a nutritional composition, with approximately 66% carbohydrates, 5% lipids, 4% proteins, and an estimated 25% of dietary fiber. High-quality copper (II) chloride (anhydrous, purity 99.995%) was purchased from Sigma Aldrich Company in Germany, while ethanol (purity ≥99.5%) and sodium hydroxide (NaOH) were purchased from Merck Company (Germany). To ensure sample purity, all specimens underwent filtration using sterile syringe filters with 0.45 and 0.22-micron retention. It is noteworthy that all chemicals and reagents were employed without the need for additional purification steps.

2.2. Sustainable fabrication of avocado peel extract-based Cu_2O nanoparticles

Utilizing an environmentally friendly and straightforward sonochemical irradiation technique, copper(I) oxide nanoparticles (Cu_2O NPs) derived from avocado peel extract (APE) were intricately synthesized. The initial phase encompassed the meticulous purification of raw avocado peel, ensuring the elimination of impurities through successive washes with distilled water and absolute ethanol, followed by gentle drying at 80 °C. To obtain APE, 40 g of avocado peel in 100 mL ethanol-water (v/v 1/9) underwent sonication at an amplitude frequency of 40% and 37 °C for 30 minutes. Simultaneously, a solution comprising 5 g of $CuCl_2$ in 250 mL distilled water, and another solution containing 0.84 g of NaOH in 50 mL distilled water, were meticulously prepared at 25 °C. The controlled addition of the $CuCl_2$ solution to the APE solution, along with the introduction of 1 mL of NaOH solution, marked the subsequent stages. The resulting mixture, i.e., the APE/$CuCl_2$ solution, underwent a 20-minute ultrasonication process at an amplitude frequency of 40%. The resultant solution underwent filtration through sterile syringe filters with retention sizes of 0.22 and 0.45 microns. The finalized APE-Cu_2O NPs were securely stored in a light-shielded environment at 25 °C for future utilization.

2.3. Characterization Part

The avocado peel extract (APE) and APE-based Cu_2O nanoparticles (NPs) underwent comprehensive characterization, employing various techniques to investigate their chemical, thermal, and surface properties. High-Resolution Transmission Electron Microscopy (HRTEM) was performed using a Hitachi HighTech HT7700 model in high vacuum mode at 100 kV. Fourier Transform Infrared (FTIR) Spectroscopy was conducted using a Jasco 6800 model in the frequency range of 4000–400 cm^{-1} wavenumber, with a resolution of 4 cm^{-1} and 64 scans. X-ray diffraction (XRD) analysis utilized Cu Kα radiation at 45 kV and 40 mA, with the Highscore Plus XRD software employed for data interpretation. Additionally, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were carried out using TA Instruments Discovery SDT 650, featuring a heating rate of 10 °C/min and a flow rate of 100 mL/min, ranging from 20 °C to 900 °C under a nitrogen atmosphere.

The crystalline size of the NPs can be determined using the Debye Scherrer equation (13),

$$D = 0.89\,\lambda / B\cos\theta \qquad \text{(Eq. 1)}$$

where D is the crystalline size, K is the Scherrer constant (0.98), λ is the wavelength (1.54), and β is the full width at half maximum (FWHM).

3. RESULTS AND DISCUSSION

To investigate the chemical, thermal, and surface properties of the APE and APE-based Cu_2O NPs, a thorough characterization process utilizing a variety of techniques was conducted. Their structural characteristics were thoroughly revealed by HRTEM. Utilizing FTIR Spectroscopy, their chemical property and functional groups were examined. The APE-based Cu_2O NPs were provided information about their crystalline structure and size by XRD technique. Furthermore, their thermal behavior was examined using DSC and TGA techniques.

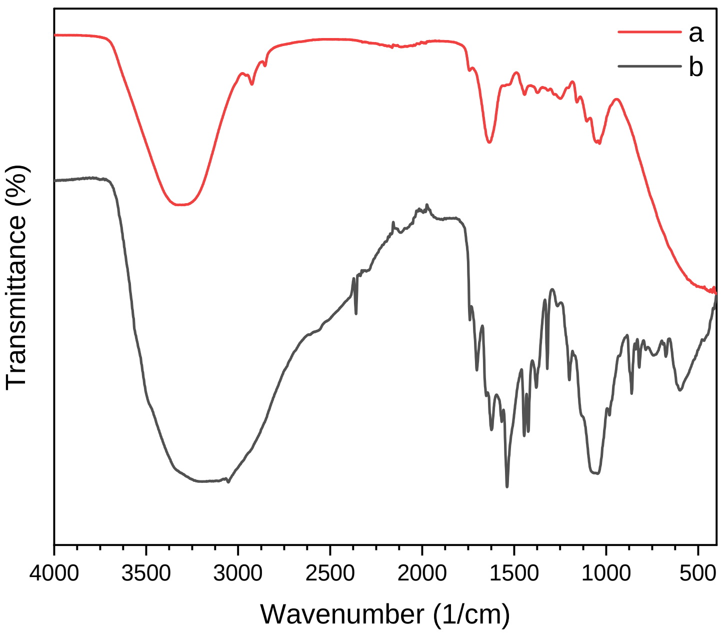

Figure 1: FTIR spectra of (a) APE, and (b) APE-based Cu_2O NPs.

The distinctive absorption peaks collectively yielded valuable insights into the molecular composition of both APE and APE-based Cu_2O NPs. The comparative analysis facilitated a comprehensive understanding of the nanostructure's composition and crystalline nature, enabling the evaluation of changes in functional groups. Figure 1 presents the FTIR spectra, detailing the functional groups and crystalline characteristics of APE (a) and APE-based Cu_2O NPs (b). Upon scrutinizing the FTIR graph of APE (Fig. 1.a), distinct absorption peaks indicative of various functional groups emerged. The region spanning 3345-3268 cm^{-1} featured stretching vibrations of hydroxyl (-OH) and amino (-NH) groups. The peak at 2929 cm^{-1} corresponded to the asymmetric stretching of CH bonds, while the one at 2855 cm^{-1} was attributed to the symmetric stretching of CH bonds. Notably, the absorption band at 1743 cm^{-1} signified the ester C=O group), and at 1635 cm^{-1}, the presence of CN stretching was evident. Additional peaks at 1538 cm^{-1} (-NH), 1443 cm^{-1} (C-H bending), and 1372 cm^{-1} (C-H bending) further characterized the molecular structure. The bands at 1248 cm^{-1}, 1159 cm^{-1}, 1105 cm^{-1}, and 1050 cm^{-1} were attributed to the C–O extension of the ester group within the triglyceride molecule (18,19). The FTIR spectra of the APE-based Cu_2O NPs revealed the O–H stretching mode of alcohols and phenols at 3500–3375 cm^{-1}, along with the C–H stretching of alkyl groups at 1623 cm^{-1}. These observations provide compelling evidence that alcohols, alkyl groups, and phenols remain unbounded during the production of APE-based Cu_2O NPs. Specifically, on Cu_2O NPs, peaks at 1423, 1380, and 1320 cm^{-1} indicated a reduction in the N=O bending vibration of nitro compounds, the C-N stretch of aromatic amines, and the C–C stretch (in-ring) of aromatics. After the sonochemical method, new peaks at 785 and 743 cm^{-1} were observed, indicating the presence of C–Cl alkyl halides. Additionally, a peak at 676 cm^{-1} denoted the C–H bend of alkanes, and a peak at 599 cm^{-1} represented the C–I stretch in aliphatic iodo compounds. These bio-waste not only served as capping agents but also bonded to the Cu_2O NPs. The absence of peaks at 1543 and 2929 cm^{-1}, coupled with the emergence of new peaks at 1066 cm^{-1}, suggested that the formation of Cu_2O NPs had induced structural alterations. The C–H stretching of alkyl groups, the C–N stretch of aliphatic amines, the N–H bending vibration of nitro compounds, and the C–OH of carboxylic acids were identified as the causes of these modifications. The prepared Cu_2O NPs were formed, as indicated by the appearance of new peaks at 820 cm^{-1} and the presence of additional peaks at 468 cm^{-1} (20).

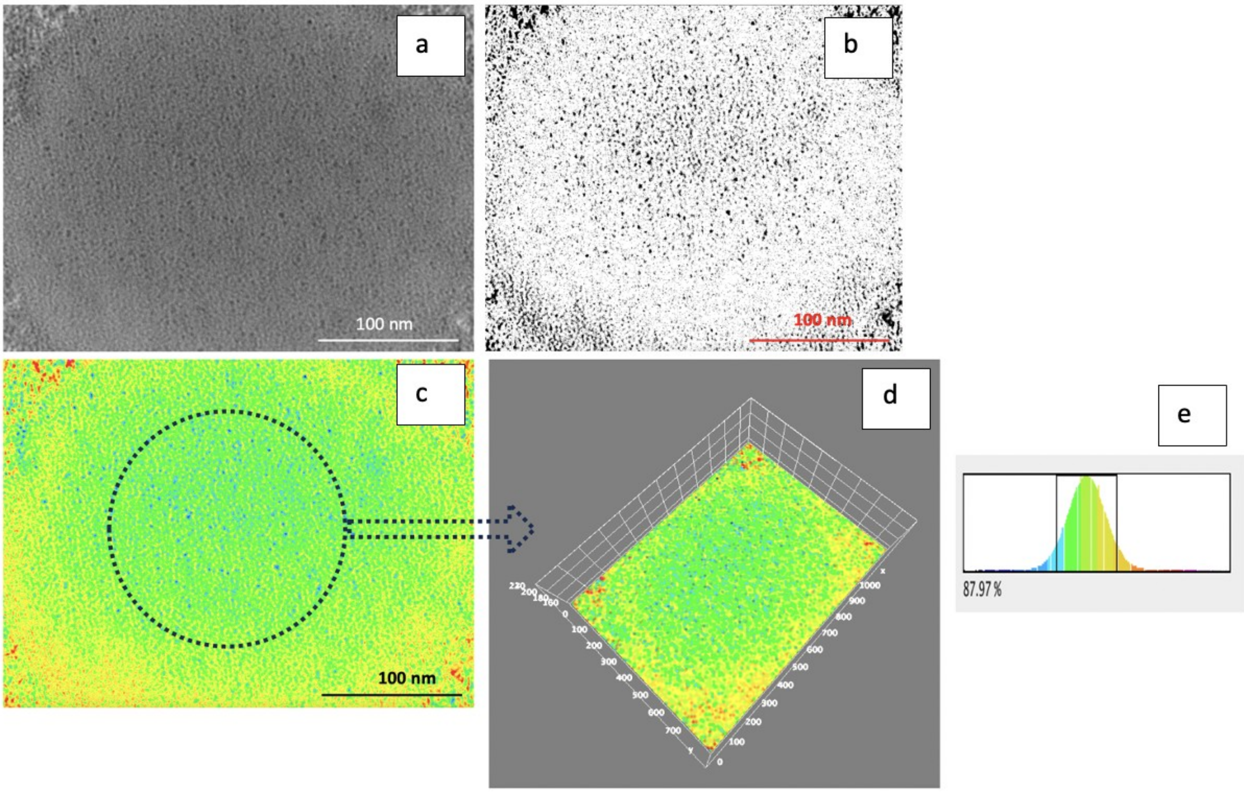

Figure 2: (a) HRTEM, (b) AI-assisted HRTEM image using threshold mode, (c) AI-assisted HRTEM image of the prepared APE-based Cu_2O NPs using RGB color mode, (d) 3D surface plot of AI-assisted HRTEM image of the prepared APE-based Cu_2O NPs, and (e) color distribution graph with the range of white and black.

Figure 2 illustrates various analyses of the prepared APE-based Cu_2O NPs, including (a) HRTEM, (b) AI-assisted HRTEM image using threshold mode, (c) AI-assisted HRTEM image using RGB color mode, (d) 3D surface plot of AI-assisted HRTEM image, and (e) color distribution graph. The TEM image in Figure 2a provided insight into the spherical morphology of APE-based Cu_2O NPs, exhibiting sizes within the range of 25 ± 2 nm. This depiction offered a detailed view of the well-dispersed particles, showcasing their uniform distribution. The integration of artificial intelligence (AI) into image processing has transformed tasks like recognition, localization, segmentation, classification, representation, interpretation, and restoration within the TEM field, as widely acknowledged. In a previous study, detailed in a study by Zheng *et al.* study (21), the synergy of AI and TEM analysis was explored, focusing on color imaging of nanoparticles with nano-sized diameters, particularly employing red-green-blue (RGB) color analysis. This approach has proven to be an efficient and cost-effective means to elucidate the morphological properties of nanostructures and enhance surface analysis.

In the investigation, a comprehensive surface characterization of the APE-based Cu_2O NPs (depicted in black color) below 20 nm was delved into, drawing insights from AI-supported TEM images (Figure 2b). The color analysis, emphasizing tones and intensities, served as a powerful tool for elucidating nanostructure properties by accentuating particle distributions. Our study employed AI-assisted RGB color analysis to unveil the spectrum of color tones (green for matrix-blue for APE-based Cu_2O NPs) within the depicted nanostructure, seamlessly integrating with TEM results (Figure 2c). This was three-dimensionally visualized in Figure 2d-e, where each pixel was represented through the combination of red, green, and blue in the RGB color model. The AI-assisted distribution analysis not only shed light on the image's overall brightness, contrast, and tonal range but also revealed a uniform distribution, particularly emphasizing, blue-based colors. Significantly, the distribution of the APE-based Cu_2O NPs was ascertained by evaluating the combined intensities of the three primary colors, prominently featuring 87.97% blue color. This finding underscored the homogeneous distribution of nanoparticles within the biomatrix, vividly illustrated by the green color representation. The application of a 3D distribution to the APE-based Cu_2O NPs, conducted in 8-bit/RGB mode using the combined intensities of the three primary colors, distinctly highlighted the prevailing blue color.

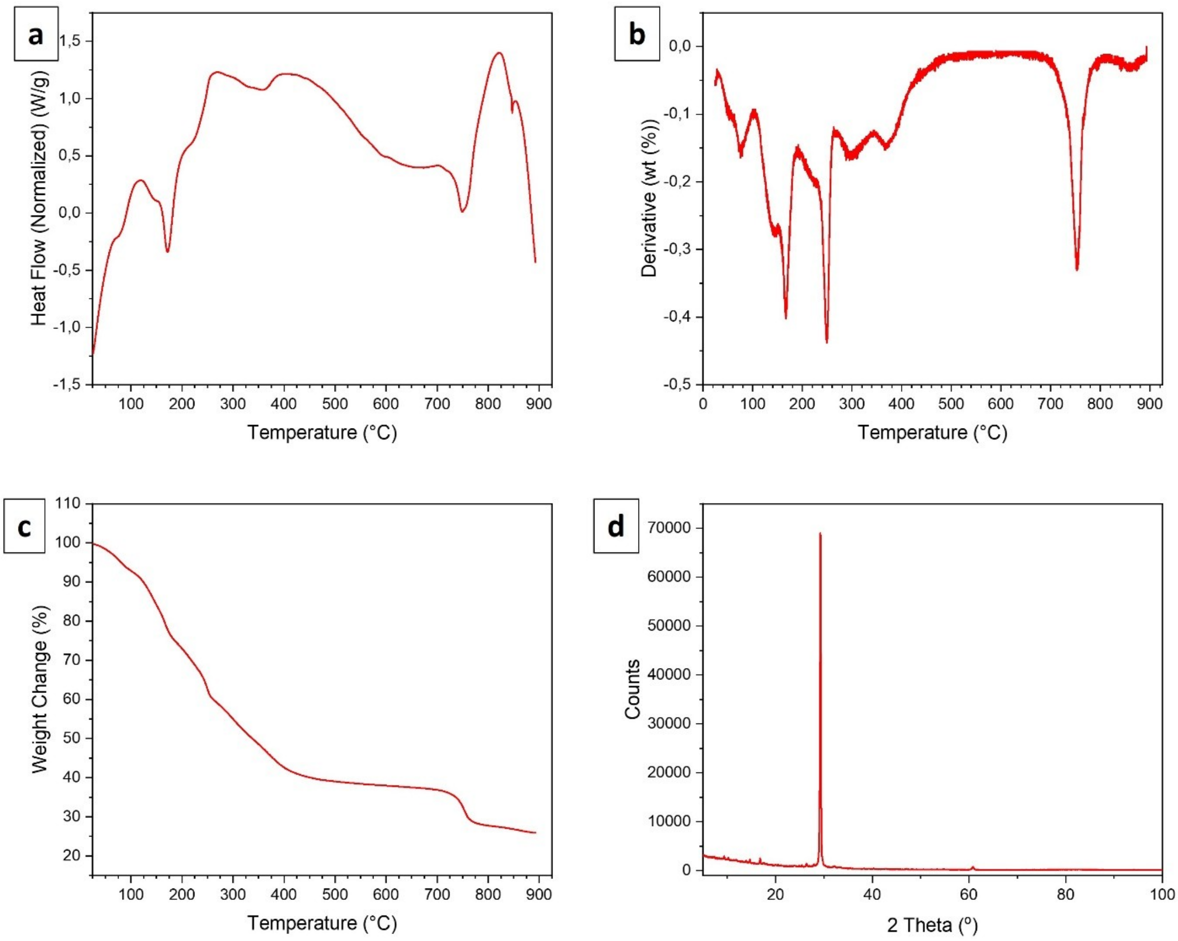

Figure 3: (a) DSC, (b) DTA (c) TGA, and (d) XRD of the prepared APE-based Cu_2O NPs.

Thermal characterization techniques, including DSC, DTA, and TGA, were used to characterize the thermal properties of the APE-based Cu_2O NPs. Figure 3a-c presents all the experimental findings. The exothermic peak of the prepared APE-based Cu_2O NPs was given in Figure 3a at 726.62 °C. A calculation of the heat produced 289.81 J/g. Using the TGA technique, the thermal stability of the prepared APE-based Cu_2O NPs was examined. TGA and DTA curves for the APE-based Cu_2O NPs were shown in Figure 3b-c, and they covered a temperature range of 20 °C to 900 °C. Furthermore, the TGA result indicated three-stage decompositions, which is corroborated by the DTA diagram that was produced, which also has three decomposition phases. With losses of roughly 27.22%, 35.97%, and 10.94%, respectively, the TGA graph of the APE-based Cu_2O NPs showed three distinct stages of decomposition: 20 °C-201 °C (evaporation of water molecules and volatile components), 201 °C-702 °C (breaking down of crosslinking and chains), and 702 °C-897 °C (phase transformation of Cu_2O NPs) (13).

As seen in Figure 3d, the XRD pattern of the prepared APE-based Cu_2O NPs showed discrete diffraction peaks at 29.28°, 35.45°, 40.25°, and 60.82°, which corresponded to the (110), (111), (200), and (220) planes of cuprite, respectively (22). These results, in line with the JCPDS Card number 05-0667, clearly show that the prepared APE-based Cu_2O NPs were successfully formed. Utilizing Debye-Scherrer's formula (Equation 1) to derive the FWHM of the most intense peak at 29.28°, we calculated the average crystalline size of the APE-based Cu_2O NPs. The calculated average crystalline size was determined to be 17 nm. Impressively, the crystal size determined by the XRD models exhibited remarkable consistency and compatibility, aligning well with the data extracted from the TEM images of the prepared APE-based Cu_2O Nps.

In Figure 4 the schematic diagram of the experimental procedure of the prepared APE-based Cu_2O NPs was given. As a result, the synthesis of nanoparticles by the sonochemical method involves an effective and innovative approach. This method is based on the principle of creating high-intensity ultrasonic vibrations in the solution. The characterization results of the obtained nanoparticles show that the rapid synthesis process facilitates the creation of nanoparticles with nanosize and homogeneous distribution. This study represents an environmentally friendly and sustainable sono-synthesis method in the production of Cu_2O nanostructures, without the need for high temperatures or harmful chemicals. This approach plays an important role in the field of nanotechnology by making nanoparticle synthesis processes more effective, faster, and environmentally friendly.

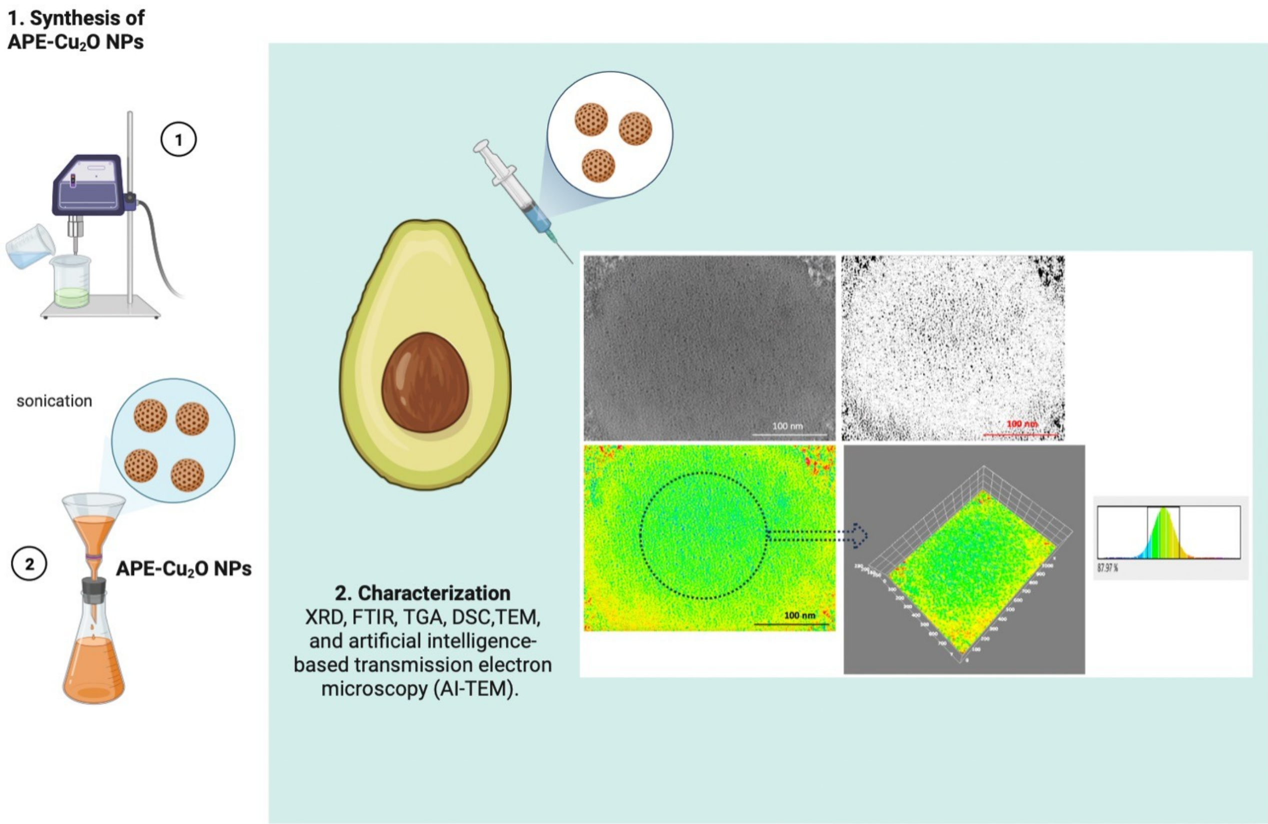

Figure 4: The schematic diagram of the experimental procedure of the prepared APE-based Cu_2O NPs.

Table 1 provides a comprehensive overview of recent progress in a variety of environmentally friendly Cu-based nanostructures, emphasizing advancements in their synthesis and applications. The literature consistently supports the notion that these green nanosystems, comprising green nanostructures, exhibit superior biological and surface efficacies compared to conventional materials, particularly in biomedical applications.

Table 1: Current developments in various green Cu-based nanostructures.

Sample	Characterization	References
Banana pulp extract-based Cu_2O NPs	cubic and octahedral shapes	(23)
Gum-mediated CuO NPs	average crystallite size about 12.91 nm, agglomerated and spherical.	(24)
Poly(butylene adipate-co-terephthalate (PBAT) containing CuO NPs	30 – 50 nm and spherical.	(25)
Tree gum extract-CuO NPs	needle-like structures	(26)
Xanthan gum-CuO NPs	100 to 750 nm and spherical.	(27)
Sunflower petal extract-CuO NPs	46 nm.	(28)
APE-based Cu_2O NPs	Particle size: a diameter of 25 ± 2 nm shape: spherical	This study

4. CONCLUSION

In recent years, a notable paradigm shift towards the sustainable production of advanced nanomaterials has emerged, reflecting a heightened emphasis on reducing environmental impact and optimizing resource utilization. This evolving interest is grounded in the imperative to address environmental concerns and adopt eco-friendly practices in material synthesis. The primary aim of this study is to explore the eco-friendly synthesis of novel APE-based Cu_2O NPs by harnessing bio-waste as a sustainable precursor. Our research primarily centers on employing ultrasound-assisted techniques for synthesizing Cu_2O NPs, with a particular emphasis on leveraging APW as a highly effective phytochemical compound for capping. Through systematic process optimization, we conducted a thorough assessment of the resulting NPs, scrutinizing their chemical, thermal, and surface properties. Advanced characterization techniques, including XRD, TEM, AI-supported TEM, TGA, DSC, and FT-IR, were employed to gain profound insights into the attributes of the synthesized NPs. Our experimental results conclusively demonstrate the successful synthesis of spherical APE-based Cu_2O NPs, each with a diameter of 25 ± 2 nm, achieved through the utilization of APW and ultrasound-assisted cavitation at room temperature. This study significantly contributes to our understanding of the potential applications of green synthesis methods, paving the way for environmentally friendly and cost-effective Cu_2O Nps.

5. CONFLICT OF INTEREST

The authors have no conflicts of interest regarding this investigation.

6. ACKNOWLEDGMENTS

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