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Efficient Single-Step Biosynthesis of Copper Nanoparticles using Essential Oil Extracted from Curry Leaves (*Murraya koenigii*) for Supercapacitor Electrodes in Energy Storage Applications

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Abstract

Nanobiotechnology is an innovative branch of modern science that has brought significantly changed in material research. The present highlights the use of essential oil collected from curry leaves (Murraya koenigii) as a simple, affordable, and efficient reducing agent for the fabrication of copper oxide (CuO) nanoparticles (NPs). These NPs were evaluated for their potential as electrodes in supercapacitors. During the synthesis, the solution changed from pale blue to green and finally to reddish-brown. This indicates the reduction of Cu^{2+} ions in copper sulfate to CuO NPs, which showed a characteristic UV-visible absorption peak at 292 nm. GCMS analysis reveals that compounds such as α-Terpinene, Linalool, Myrcene, Geranyl acetate, Elemol, and Allo-Ocimene in the essential oil played a role in NPs formation. The NPs were primarily spherical in shape with 24 nm average particle size. These particles exhibit a monoclinic crystal structure with 81.26% of elemental copper. Cyclic voltammetry results showed specific capacitances of 369, 324, 305, 265, and 210 F/g respectively at scan rates of 10, 20, 40, 60, and 100 mV/s. The maximum specific capacitance was noticed to be 369 F/g, with a retention capacity of 97.6% even after 6000 charge-discharge cycles demonstrating that these NPs have suitability for supercapacitor applications. These results suggest that biologically synthesized CuO NPs have great potential for utilization as energy storage devices, particularly as supercapacitor electrodes.

Keywords: *Green synthesis, Essential oil, GCMS analysis, CuO NPs, Supercapacitor applications.*

1. INRODUCTION

Supercapacitors also referred to as ultracapacitors or electrochemical capacitors are unique energy storage devices that bridge the gap between batteries and traditional capacitors. They electrostatically store and release energy and they offers distinct features that makes them ideal for a wide range of applications [1]. It has significant features such as efficient energy storage, high power density, longer cycle life than batteries, low internal resistance with a broad temperature range, fast charge and discharge rates. Supercapacitors are classified as electrical double-layer capacitors (EDLCs) and pseudocapacitors based on their energy storage mechanisms. Among these, pseudocapacitors stand out due to their

significantly higher energy density and specific capacitance compared to EDLCs and conventional supercapacitors [2]. As a result, considerable research efforts have been directed towards improving the performance of pseudocapacitors. Transition metal oxides were widely utilized as active materials for pseudocapacitors due to their high availability, low cost, excellent conductivity, multiple oxidation states, and good stability. Among these, CuO is particularly noteworthy for its impressive electrochemical properties, affordability, environmental friendliness, and high capacitance, making it a promising candidate for advancing energy storage technologies [3].

Green synthesis of NPs was treated as an environmentally friendly and sustainable approach for the production of NPs by eliminating harmful chemicals and minimal environmental harm. This approach typically uses natural resources like plant extracts, microorganisms, and any bio-based materials to convert metal ions into NPs. It has gained significant attention for its ability to lower the environmental footprints in the production of NPs [4]. Integrating green-synthesized NPs into pseudocapacitors is a promising and rapidly growing field in nanotechnology and energy storage. This combination emphasizes sustainability while advancing energy storage technology [5]. Current research is exploring innovative green procedures, producing novel materials, and optimizing device designs to enhance performance and enable practical applications on a larger scale [6].

Murraya koenigii (L), commonly referred to as "curry leaves," belongs to the *Rutaceae* family and thrives in tropical and subtropical regions worldwide [7]. This small evergreen tree typically reaches heights of 4 to 5 meters, boasting foliage with a delightful blend of sweet curry fragrance [8]. The leaves of *M. koenigii* impart a slightly bitter taste coupled with a pungent aroma and subtle acidity [9]. In Indian cuisine, these leaves are valued for their medicinal properties, serving as digestives, analgesics, appetizers, and antihelminthics. In traditional medicine, *M. koenigii* leaves find use in treating various ailments including inflammation, dysentery, edema, itching, piles, and bruises, while roots possess purgative properties and are utilized to alleviate common body pains [10]. Additionally, the bark is recognized for its effectiveness in snakebite treatment. Extracted essential oil from curry leaves exhibits a range of beneficial effects in animal models, including antioxidant, hepatoprotective, antimicrobial, antifungal, antiinflammatory, and nephroprotective properties [11]. These medicinal significances are attributed to a diverse array of chemical constituents present in *M. koenigii*, including flavonoids, carbohydrates, carbazole alkaloids, terpenoids, phenolics, vitamins, nicotinic acid and carotenoids found in various parts of the plant [12].

In NPs synthesis, plant-derived essential oil serves as reducing and stabilizing agents. Several studies have reported the use of various plant essential oils for synthesizing metal NPs such as silver [12-17], titanium dioxide [18], iron [19], gold [20], magnesium [21], and copper [22]. There is no author reported the green synthesis of CuO NPs using essential oil extracted from curry leaves. Although researchers have investigated the potential of green-synthesized NPs in energy storage applications [23-26], their full potential remains underexplored. Addressing this gap, the present study aims to utilize CuO NPs produced using curry leaf essential oil as electrodes for supercapacitors in energy storage devices.

2. MATERIALS AND METHODS

2.1. Reagents and Materials

Curry leaves were procured from a local market in Guntur, Andhra Pradesh, India. Analytical-grade chemicals, including copper sulfate ($CuSO_4$), sodium borohydride ($NaBH_4$), ascorbic acid, and

polyethylene glycol (PEG) 8000 were sourced from Merck Chemicals, Mumbai.

2.2. Essential Oil Extraction from Curry Leaves

Demineralized water was used as the extraction medium for isolating essential oil from curry leaf powder. Approximately 150 g of leaf powder was hydro-distilled with 1.5 L water at 80 °C for 4 h using a Clevenger apparatus (Borosil®). The collected essential oil was characterized by its yellow color and strong pungent odor which was carefully separated. The oil was preserved at 6 °C and utilized for NPs synthesis.

2.3. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

The GC-MS analysis extracted essential oil and the oil-mediated CuO NPs was performed to evaluate the chemical constituents involved in the formation of NPs. The analysis was performed on an Agilent (USA) 7890 model gas chromatograph equipped with a DB-624 capillary column (30 m × 0.25 mm; 0.25 µm). Helium served as the carrier gas at a flow rate of 1.2 mL/min with a split ratio of 1:4. The oven temperature was initially held at 50 °C for 4 minutes, then increased to 280 °C at a rate of 5 °C per minute and maintained at 280 °C for 1 minute. A 0.2 µL sample was injected into the system through an injector set at 250 °C. For compound detection, the mass spectrometer operated in electrospray ionization mode at 70 eV, with spectra recorded in the range of 40 to 350 atomic mass units. The compounds were identified by comparing their retention indices, determined using n-alkanes, and their mass fragmentation patterns with entries in the NIST (National Institute of Standards and Technology) library.

2.4. Synthesis of CuO NPs

A precisely measured 20 mL of PEG solution was taken in a 500 mL beaker having 100 mL of $CuSO_4$ (0.1 M) solution. The mixture was stirred for 1 hour at room temperature followed by the addition of 40 mL ascorbic acid (0.5 M) solution was added. The stirring was continued at 500 rpm for 30 min at 100 °C. Then, 80 mL of $NaBH_4$ solution was introduced, followed by the gradual addition of 10 mL of curry leaf essential oil with continuous stirring for another 30 minutes at 500 rpm. The resulting particles were collected by vacuum filtration using a Buchner funnel and Whatman No. 1 filter paper. Then the particles were separated, washing thoroughly with distilled water several times to remove any residual impurities and then drying at 60°C in a hot air oven [26].

2.5. Characterization of CuO NPs

The optical properties of curry leaf essential oil and the CuO NPs synthesized using it were analyzed using a double-beam UV-visible spectrophotometer (JASCO, V-560, Japan) in the wavelength range of 200–800 nm. The essential oil chemical constituents that are involved in NPs formation were identified through GC-MS analysis. The Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) coupled with Energy-Dispersive X-ray Spectroscopy (EDX) studies were performed for the structural characterization of CuO NPs. The NPs were washed multiple times with methanol followed by distilled water. Then these cleaned NPs were fixed onto carbon-coated copper grids and analyzed using TEM (Jeol JEM 2100, Japan) at an operating voltage of 120 keV. The SEM (Jeol 6390LA, Japan) coupled with EDX (OXFORD XMX-N, UK) was utilized to evaluate the particle size and elemental composition of NPs. The instrument utilized a tungsten lamp as the energy source and was operated at an accelerating voltage of 0.5 to 30 kV. The crystal size of the CuO NPs was evaluated through an X-ray diffraction (XRD) instrument (Bruker D8 Advance, USA) with Cu Kα radiation, and the analysis was performed over a 2θ

range of 20°–100°. The size distribution and zeta potential of NPs were determined using a Brookhaven (DR-525, USA) 90Plus NPs size analyzer.

2.6. Electrochemical Characterization

The electrochemical workstation (CHI660E, USA) equipped with Ni foam as the working electrode, Platinum wire as the auxiliary electrode, and Ag/AgCl as the reference electrode was utilized to characterize the electrochemical behavior of curry leaf essential oil-mediated CuO NPs. In this study, 1 mg of synthesized CuO NPs were accurately weighed and mixed with N-N-Methyl pyrrolidone (0.5 mL) to form a slurry paste with a binder (10% polyvinylidene fluoride). The slurry was applied to the working electrode (1 × 1 cm) and dried at 25 °C for 2 hours. A 3 M potassium hydroxide (KOH) solution was used as the electrolyte. Cyclic voltammetry (CV) experiments were carried out between 0 to 0.65 V at different scan rates. Galvanostatic charge-discharge (GCD) curves were measured at an operating voltage of 0 to 0.6 V at different current densities. Electrochemical Impedance Spectroscopy (EIS) analysis was performed at a fixed amplitude (5 mV) and a frequency range of 0.001 kHz. The specific capacitance (Cs) in F/g of the electrode was determined using the formula.

$$Cs = \frac{i\Delta t}{m\Delta v}$$

In the equation, *i* is the current (A), *m* corresponds to the mass of active material mass in g, Δt corresponds to discharge time in seconds and ΔV represents a potential window in V

$$c = \frac{1}{mv(Vc - Va)\int_{Va}^{Vc} I(V)dV}$$

In the equation, *I* is applied current in A, V corresponds to the potential rate (mV/s) and m mass of NPs loaded on the electrode at 1 cm^2, Vc − Va represents a potential range.

3. RESULTS

This study aimed to synthesize CuO NPs utilizing volatile constituents in the essential oil extracted from *Murraya koenigii* leaves. The mixing of essential oil, PEG, sodium borohydride, and ascorbic acid into the copper sulfate solution triggered the formation of NPs. The synthetic mixture pH was varied between 4 and 10, to optimize the yield with reduced synthesis time. The pH of 8 was proved to be the most ideal for NPs synthesis which yields a high quantity of NPs with very less reaction time. The solution color changed from pale blue to brown and eventually dark brown, indicating the reduction of Cu^{2+} ions to CuO NPs. This color change was monitored with a UV-visible spectro-photometer in the 200-800 nm wavelength range. The formation of CuO NPs shows a broad absorption peak at 290 nm representing the interband transition of 'core electrons'. This peak was not observed in the UV-visible spectrum of pure essential oil, which is consistent with previous reports [26, 28]. As the size of the NPs decreases there will be an increase in the band gap noticed suggesting the dose-dependent band gap. Understanding and controlling the optical band gap was very crucial for tailoring the optical properties of CuO NPs for various applications, especially in electronics in which band gap impacts electrical conductivity and charge carrier behavior. Hence the following Tauc classical approach was employed for evaluating the optical band gap (E_g) of synthesized NPs

$\alpha E_p = K(E_p - E_g)^{1/2}$

In the equation, Ep corresponds to discrete photo energy; K is a constant

The results proved that the CuO NPs exhibit a 3.1 eV band gap. This relatively high band gap enables the tuning of their properties and makes NPs ideal for a wide variety of applications, including sensing, electronics, catalysis, and biomedical fields. The ability to control the band gap is essential for optimizing the NPs' performance in these diverse applications.

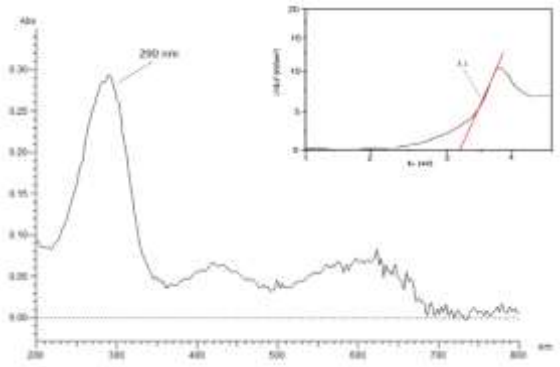


Figure 1. *UV visible absorption spectrum of CuO NPs synthesized in the study; Inset shows a graph related to the band gap of NPs by plotting $(\alpha E_p)^2$ against E_p.*

The GC-MS analysis provides detailed insights into the chemical composition of both essential oil and the synthesized CuO NPs. Figure 2 presents the GC-MS chromatogram of the CuO NPs which proves the presence of key compounds such as geranyl acetate, α-terpinene, myrcene, allo-ocimene, linalool, and elemol. These compounds play dual roles as both capping and reducing agents during the NPs formation. Table 1 provides a detailed breakdown of the phytochemical constituents identified in the essential oil and the synthesized CuO NPs by offering a comprehensive overview of the chemical composition of the NPs. These findings highlight the involvement of specific compounds in the NPs synthesis process, further elucidating their role in shaping the properties of the CuO NPs.

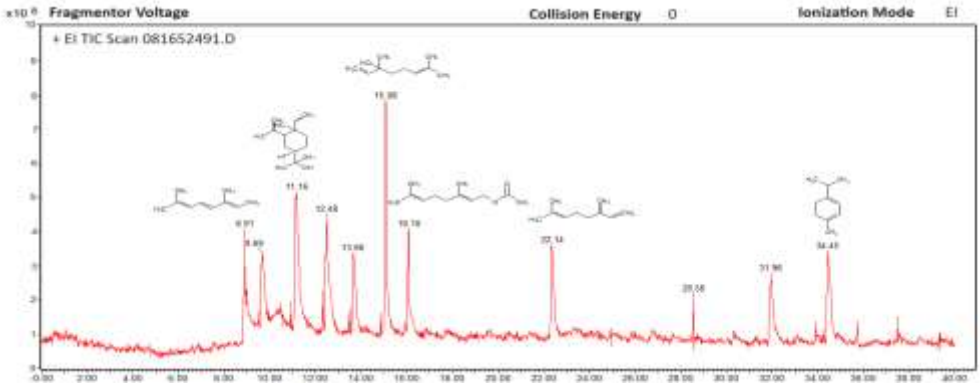
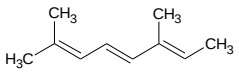
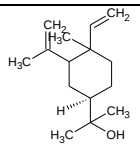
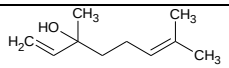
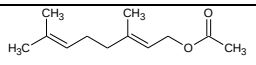
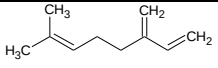
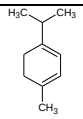


Figure 2. *GCMS chromatogram of CuO NPs synthesized using curry leaves essential oil.*

Table 1. *The chemical constituents identified in GC-MS analysis of CuO NPs synthesized using curry leaf essential oil.*

S. No	Rt (min)	Molecular formula	Molecular mass	Compound Name	Structure
1	8.91	$C_{10}H_{16}$	136.23 g/mol	Allo-Ocimene	
2	11.16	$C_{15}H_{26}O$	222.36 g/mol	Elemol	
3	15.08	$C_{10}H_{18}O$	136.23 g/mol	Linalool	
4	16.10	$C_{12}H_{20}O_2$	196.28 g/mol	Geranyl acetate	
5	22.14	$C_{10}H_{16}$	136.23 g/mol	Myrcene	
6	34.45	$C_{10}H_{16}$	136.23 g/mol	α-Terpinene	

The surface activity, shape, and size of synthesized NPs were examined through TEM analysis and were performed at 1 µm and 100 nm scales. The NPs shape and size were observed at 1 µm scale whereas particles were distinctly visible at 100 nm scale. The TEM result confirms that the NPs were spherical with the most abundant particle size being approximately 25 nm (Figure 3A). The spherical shape of NPs provides a high surface area with more active sites for electrochemical reactions, which enhances the capacitance of the supercapacitor. Additionally, the small particle size enables high charge and discharge rates which improve the overall conductivity of the supercapacitor. The SAED (Selected Area Electron Diffraction) pattern as shown in Figure 3B further supports the crystallinity of the NPs, as evidenced by the identification of intermediate points in concentration circles. This confirms the crystalline nature of the synthesized CuO NPs.

The zeta potential of CuO NPs was measured to assess the repulsion between particles in the dispersion solution, which helps prevent aggregation. A sufficiently high zeta potential indicates stable NPs. The CuO NPs exhibit a zeta potential response of -25.6 mV (Figure 3C) confirming the stability of the NPs at ambient temperature and these results align with previous studies reported [26,28,29].

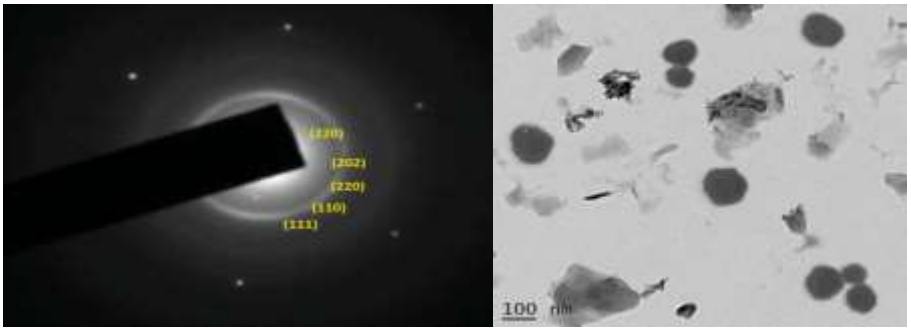
The crystallinity and structural information of CuO NPs were further analyzed using XRD and the obtained data were utilized to calculate the interplanar spacing (d) and crystal size (D) of the NPs by using Bragg's law and Scherrer's formula respectively.

$n\lambda = 2d\sin\theta$

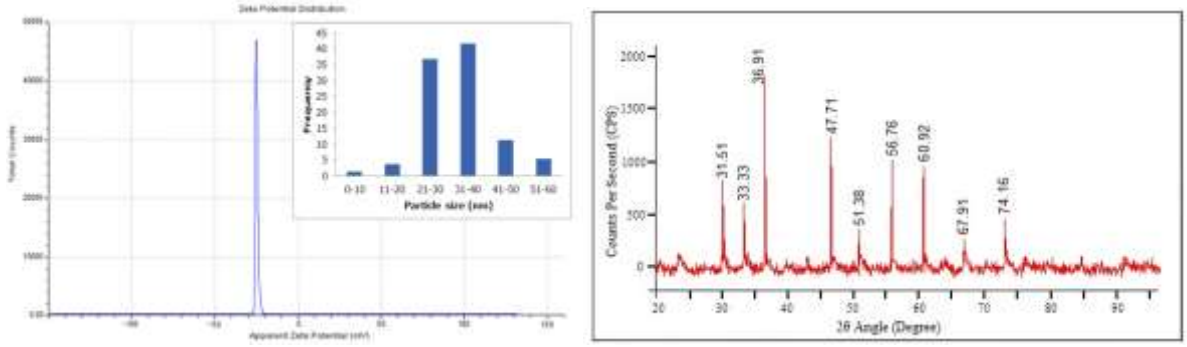
$$D = \frac{K\lambda}{\beta\cos\theta} \times 100$$

where k is shape factor (constant), λ is the X-ray wavelength studied, β was treated as full-width half maximum and θ represents Bragg's angle.

The diffraction peaks (Figure 3D) at 31.51° (110), 33.33° (002), 36.91° (111), 47.71° (200), 51.38° (202), 57.76° (002), 60.92° (113), 67.91° (220) and 74.16° (004) correlated with monoclinic phase crystal structure of synthesized CuO NPs. The crystal structure was by the standard JCPDS card No. 01-080-1268 and results were well correlated with findings published by various authors [26,28,29]. The most probable size range of NPs was noticed in the 21 to 40 nm range with approximately 24.9 nm average particle size. No unassigned diffraction peaks were detected in the XRD spectrum, indicating that no impurities were present in the synthesized NPs. This confirms that the NPs were highly pure.



A) SEM B) SAED



C) zeta potential D) XRD

Figure 3. *Characterization of CuO* NPs *synthesized via the bio-synthesis method utilizing curry leaf essential oil.*

The EDX analysis was conducted to confirm the elemental composition, as well as the atomic and weight percentage of the synthesized CuO NPs. The EDX spectrum (Figure 4) displayed a prominent peak corresponding to copper at approximately 8.05 keV. Additionally, peaks corresponding to phytochemical constituents, such as carbon (~5.83%) and oxygen (~12.91%), were observed at approximately 0.3 keV and 0.5 keV, respectively. No other peaks detected in the spectrum suggest that the synthesized NPs possess significantly high purity.

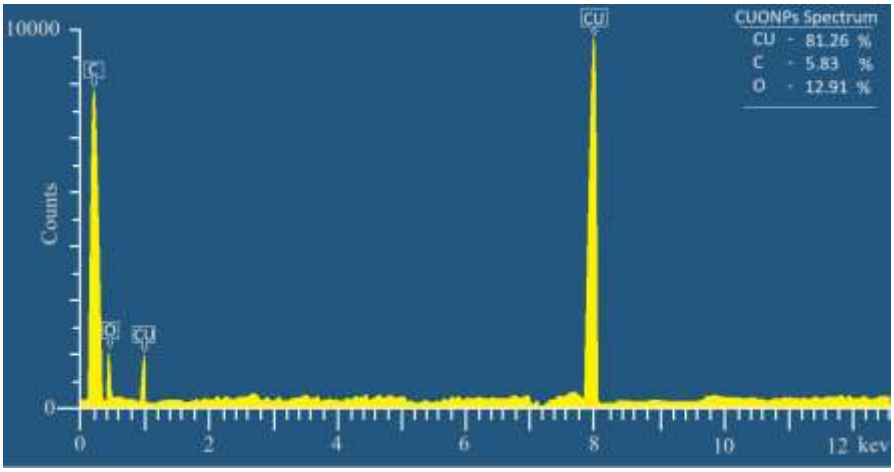


Figure 4. *EDX spectrum noticed for NPs synthesized using curry leaf essential oil.*

A traditional 3-electrode system with potassium hydroxide as the electrolyte solution was utilized to perform electrochemical tests. The cyclic voltammetry was employed to evaluate the current response of the electrochemical system. The study utilized a nickel foam electrode coated with 1 mg of CuO NPs and was tested over 25 to 100 mV/s scan range and 0 - 0.65 V potential range. The results as shown in figure Figure 5A demonstrate that the current value increases with the increase of the current value. The Faradic redox current responses of NPs loaded nickel foam exhibit a significantly enhanced response than uncoated nickel foam. The cyclic voltammetry curve as shown in Figure 5B suggests that the NPs coated electrode shows the largest integral area and high current density at both cathodic and anodic peaks than normal electrodes.The GCD curves (Figure 5C) were measured at current densities ranging from 1 to 5 A/g. The charge-discharge time stabilized as the current density decreased. Specific conductance was evaluated using the cyclic voltammetry response and found to be 369,

324, 305, 265, and 210 F/g at scan rates of 10, 20, 40, 60, and 100 mV/s, respectively. For the GCD curves, the specific conductance was observed to be 629, 508, 421, 250, and 128 F/g at current densities of 1 to 5 A/g (Figure 5D). A comparison of CuO NPs and nano-composites reported in recent literature (Table 2) proved that the NPs synthesized in this study exhibit the most efficient capacitance performance compared to those reported.

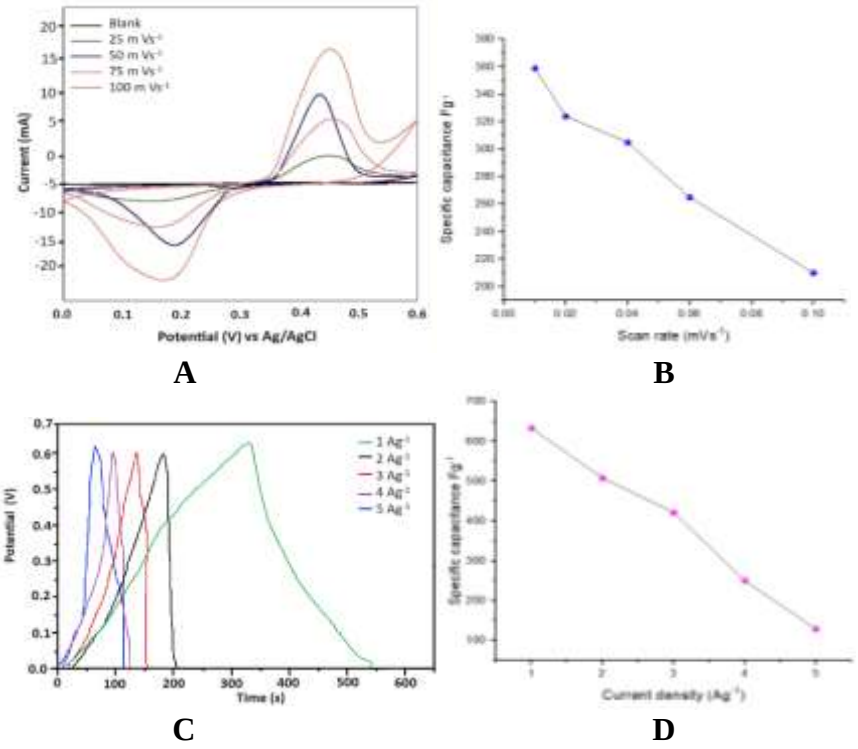
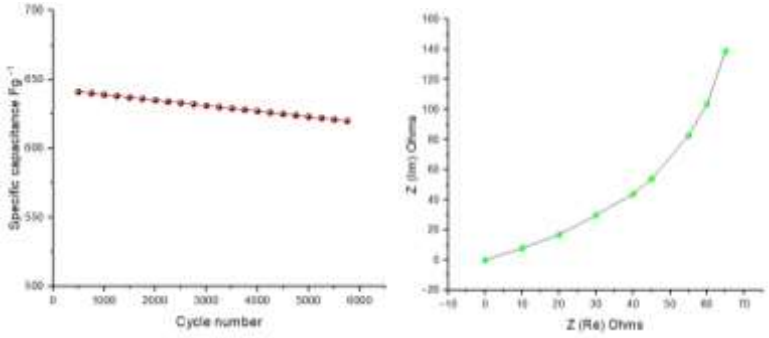


Figure 5. *The electrochemical tests results noticed for synthesized CuO NPs in which 'A' corresponds to CV responses were recorded at different scan rates of 25, 50, 75, and 100 mV/s; the specific capacitance results presented in 'B'; the GDC curves at different current densities are presented in 'C' and specific capacitance results at various current densities was shown in 'D'.*

Table 2. *The correlation between supercapacitor application of various NPs reported in the literature with the findings noticed in this study.*

S No	Electrode type	Electrolyte	Specific conductance (F/g)	Retention of capacitance (%)	Reference
1	CuO NPs	KOH	369	98.9	This study
2	CuO	$NaHCO_3$	2.14	Not reported	30
3	Cu_2O/ graphene oxide composite	KOH	195	79	31
4	carbon nanotubes @TFcP/Cu	H_2SO_4	280	80	32
5	$CuFeS_2$	Na_2SO_4	501	82	33
6	Activated carbon/ CuONPs	Na_2SO_4	245	99.5	34
7	Graphene oxide- CuO composite	82.1	Not reported	98	35
8	reduced grapheme oxide-CuO composite	137	Not reported	Not reported	39

The cycle stability of the electrode was evaluated to evaluate the electrode performance and last over time when used in energy storage devices like supercapacitors and batteries. The electrodes were subjected to 6000 charge and discharge cycles to simulate real-life use over an extended period. After these cycles, the electrodes maintained 97.6% of their initial capacity (Figure 6A). To further assess their performance, electrochemical impedance spectroscopy (EIS) was carried out to examine changes in the electrode's resistance during these cycles. The study used a 1 M KOH electrolyte with a frequency range from 0.01 Hz to 100 kHz and a potential of 20 mV. The EIS results (Figure 6B) showed a solution resistance (Rs) of 3.02 Ω and a charge transfer resistance (Rct) of 15.23 Ω. The low Rs values suggest that the electrodes have improved electrical conductivity and better attachment to the current collector, which enhances their overall efficiency.



A B

Figure 6. *The results of the cycle stability study (A) and EIS analysis (B) of CuO NPs synthesized utilizing curry leaf essential oil are presented.*

4. CONCLUSION

This research employs a green chemistry method to synthesize CuO NPs using essential oil extracted from curry leaves. The NPs have a 17.6 nm average size and are composed of key compounds from the essential oil, including α-terpinene, elemol, linalool, geranyl acetate, myrcene, allo-ocimene and result in 81.26% copper content. The study explores the potential of these NPs as electrodes for supercapacitors in energy storage systems. Remarkably, the NPs exhibit a high specific capacitance of 369 F/g and retain 98.9% of their capacity at a current density of 1 A/g. They also demonstrate excellent stability, maintaining 97.6% of their capacity after 6000 charge/discharge cycles. The superior electrochemical performance of these NPs is attributed to the presence of abundant active sites, which facilitate efficient charge transfer. This work highlights the potential of green-synthesized CuO NPs as high-performance and cost-effective materials for energy storage applications. The study emphasizes the role of green chemistry in providing sustainable and environmentally friendly solutions for advancing energy storage technologies.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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