

Soyabean Husk-Assisted Green Synthesis of rGO/CuO Nanocomposite for Supercapacitor Applications

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ABSTRACT

The rising global demand for green energy storage systems has led to the development of studies aimed at finding environmentally friendly electrode materials to use in supercapacitors. The conventional approach to producing graphene-based nanocomposites usually requires the use of toxic chemicals, a high amount of energy as well as non-renewable materials and this is of concern to the environment and poses a question of scalability. Another variant that has come up as a feasible alternative in this direction and integrates material efficacy with sustainability is biomass-assisted green synthesis.

This paper provides a detailed analysis of green synthesis of reduced graphene oxide/copper oxide (rGO/CuO) nanocomposites using soybean husks and their application as high-performance electrodes to supercapacitors. Our reducing and stabilizing agent, which was a natural by-product (soybean husk), was used to reduce graphene oxide and simultaneously produce CuO nanoparticles due to the sufficient amount of polyphenols and reducing sugars in soybean husk. The existence of the structural, morphological, and electrochemical characteristics of the successful formation of the composite demonstrated the presence of increased surface area, uniform distribution of the nanoparticle, and electrical conductivity.

Electrochemical determinations reflected high-quality specific capacitance, great rate performance and great cycling stability as compared to pristine rGO and CuO electrodes. The rGO-CuO interaction was synergetic and was able to stimulate effective transfer of charges and enhance the behavior of pseudocapacitance. The study has shown how agricultural waste could be used to produce high-performing functional materials and has set a sustainable process for producing next generation supercapacitor electrodes.

Keywords- Green synthesis, reduced graphene oxide, copper oxide nanocomposites, soybean husk, supercapacitors, biomass-derived materials, sustainable electrode fabrication.

I. INTRODUCTION

1.1 Challenges in energy storage in the modern world:

The transformation of the energy systems, which are reliant on fossil fuels, to renewable sources is one of the major technological challenges in the twenty-first century. Solar and wind technologies are examples of renewable energy resources, which tend to be intermittent, and as such, require a reliable energy storage mechanism to offer power stability and impedance to the grid. Also, the rapid advancement of portable electronic gadgets, electric cars, and wearable devices has increased the need for lightweight, fast chargeable, reliable and

green energy storage systems. In the current scenario lithium-ion batteries are leading the energy storage market on account of their high energy density. These have their drawbacks in the form of relatively low charge-discharge rates, limited life cycle, safety risks associated with thermal runaway, and dependence on easily available raw materials. Such concerns have sparked the search for other energy storage technologies and one of the areas of concern is that of supercapacitors.

1.2 Supercapacitors: Principles and Benefits:

Also referred to as electrochemical capacitors, supercapacitors are used to bridge the performance gap that exists between conventional capacitors and batteries.

They are high power density, rapid charge-discharge, long cycle life and safer operation. Energy is stored using two broadly heard methods with electrical double layered capacitance (EDLC) and pseudocapacitance, in supercapacitors.

EDLC is due to the charge accumulation at the electrode-electrolyte interface as a result of electrostatic actions, which is usually seen in carbon-based materials like activated carbon, carbon nanotubes and graphene. Pseudocapacitance on the other hand is caused by relaxation redox reactions at the electrode surface occurring rapidly and reversibly, usually exhibited by transition metal oxides and conducting polymers.

Physical In combination Hybrid electrode materials involving EDLC and ES scaffolds are being developed to achieve high overall capacitance and higher energy density. Graphene-based metal oxide nanocomposites are one of the potential categories of materials.

1.3 Graphene and Reduced Graphene Oxide in Power Storage:

Graphene is a 2-dimensional (carbon) material composed of one layer of sp² - hybridized carbon atoms in a honeycomb structure. It has some extraordinary properties, including excellent electrical conductivity, high mechanical strength and a very large theoretical surface area of approximately 2630 m² -1. These features make graphene a good option in terms of electrodes in supercapacitors.

However, there are great challenges to the large-scale fabrication of pure graphene. In a second choice, graphene oxide (GO), which is a result of chemical oxidation of graphite, is an easier to prepare and functionalize material. GO contains oxygen functional groups such as hydroxyl, epoxide, carbonyl, and carboxyl groups that disrupt the sp² carbon network but it improves dispersion further in aqueous solutions.

By partially removing these oxygen functional groups, reduced graphene oxide (rGO) is produced to regain electrical conductivity with preserved structural defects that can serve as active sites in anchoring nanoparticles. rGO has been one of the most common carbon electronics to make electrodes in hybrid supercapacitors.

1.4 Copper Oxide as a Pseudocapacitive Material:

Copper oxide (CuO) is a p-type semiconductor that has a narrow bandgap (~1.2 eV) and well-coordinated redox chemistry. It is abundant, economical, and does not pollute the environment as compared to the noble metal oxides. In the solution contained in alkali CuO has a pseudocapacitive behavior in this reversible Cu²⁺/Cu³⁺ + redox reaction.

Philosophically pure CuO electrodes also have some drawbacks inherent in them, including poor electrical conductivity, low surface area, and structural degradation with repeated cycling. These weaknesses result in reduced rate performance and inadequate long-

term stability that limit their use in practice in supercapacitors.

1.5 Synergistic rGO/CuO Nanocomposites.

Adding CuO nanoparticles to rGO sheets provides solutions to multiple issues associated with each of the two components where rGO offers a conductive framework to enable rapid electron transport and the avoidance of agglomeration of nanoparticles, and CuO offers mechanisms to provide pseudocapacitive charge storage functionality. The synergistic relationship between the electrical double layer capacitance (EDLC) of the rGO and the pseudocapacitance of CuO leads to an increase in the specific capacitance and rate performance and an increase in cycling stability.

It has been demonstrated in many studies that rGO/CuO nanocomposites have superior electrochemical performance compared to their pristine counterparts. But, the majority of the listed synthesis methods rely on the use of toxic chemical reducing agents and surfactants and this brings environmental and safety concerns into being.

1.6 Necessity of Green Synthesis Methods:

Green synthesis aims at the use of renewable resources, non-toxic reagents, energy-saving, and reducing the amount of waste. Biomass-helped synthesis is in accordance with the principles of green chemistry since it utilizes naturally available reducing agents which are present in plant extracts, agricultural by-products or by using microbial systems.

Another promising biomass source is agricultural waste under the condition of its abundance, cheapness, and intricate chemical composition. Repurposing this kind of waste by converting it into functional materials of high-value does not only helps reduce the environmental impact but it will also help build the models of the circular economy.

1.7 Husk of Soybean as a Biomass Resource:

This is because Soybean husk is a by-product of the agro-industrial sector that is generated in significant quantities upon processing of soybeans. The commonly used polymers are cellulose, hemicellulose, lignin, proteins, and bioactive phytochemicals such as polyphenols and flavonoids. These components are reducing and chelating in nature and thus the soybean husk is a good green reducing agent to use in the production of nanomaterials.

Soybean husk is unexploited and not utilized fully despite it being plenty and is usually thrown or used as animal fodder. The application of soybean husk in the preparation of nanocomposites alters the usefulness of agricultural waste products besides fostering the design of materials that are environmentally friendly.

1.8 Objectives of the Current Study:

This research will have the following objectives:

- To synthesize rGO/CuO nanocomposites with the help of soybean husk into the green synthesis pathway.
- To identify the structural, morphological, and chemical properties of the nanocomposite produced.

- To evaluate the electrochemical behavior of rGO/CuO nanocomposite as an electrode of a supercapacitor.
- The aim is to explore the synergies in charge storage mechanisms and the benefits of biomass-assisted synthesis in terms of its sustainability.

II. LITERATURE REVIEW

2.1 Graphene-Based Electrode Materials:

Graphene and its allotropes have been extensively studied to be used in supercapacitors due to their high level of conductivity and high surface area. However, the π -interactions between sheets of graphene result in the restacking of the sheets reducing the available surface area, which restricts capacitance. In order to solve this problem, heteroatomic doping, surface functionalization, and the development of combinations with metal oxides have been considered.

2.2 Copper oxidenanostructure in supercapacitors:

Hydrothermal, sol/gel and chemical precipitation techniques have been used to produce copper oxide (CuO) nanostructures, such as nanorods, nanoflowers, and nanoparticles. More particular values of the capacitance of the electrodes denoted as CuO mostly comprise an approximate span of 50 to 250 F g⁻¹ in an alkaline environment, and may vary depending on the morphology and the mode of synthesis. However, it is often observed that rapid capacitance losses are experienced as a result of problems such as volume expansion and low conductivity.

2.3 rGO/CuO Nanocomposites:

It has been shown by multiple studies that there is an increase in electrochemical performance when rGO is coupled with an increased amount of CuO. When operating in current densities of approximately 1 A g⁻¹, specific capacitance values of rGO/CuO composites are normally between 150 and 400 F g⁻¹. These enhancements can be attributed to enhanced electron transport, expansion of active surface area and lowered agglomeration of nanoparticles.

2.4 Green Synthesis with Biomass:

Graphene oxide has been successfully reduced using the method of green synthesis which involves plant extracts including tea leaves, aloe vera, banana peel and citrus waste. Metal nanoparticles have also been successfully prepared using the same method. The use of synthesis aided by biomass usually contributes to improved dispersion and surface functionalization because of the natural capping agents present in the extracts.

Despite all this, there were minimal studies keen on a reduction agent of soybean husk to reduce both rGO and the formation of CuO simultaneously and particularly in the supercapacitor cell context.

III. MATERIALS AND METHODS

3.1 Materials:

Chemicals- All the chemicals used in this study were of analytical reagent grade, and they have not been further purified to ensure accuracy and reproducibility of the experiment. The starting material of the synthesis of graphene oxide was graphite powder of purity of ≥ 99.9 . The source of copper used to produce CuO nanoparticles was copper sulfate pentahydrate (CuSO₄ · 5H₂O). pH adjustment and supporting electrolytes in electrochemical measurements were done using sodium hydroxide (NaOH) and potassium hydroxide (KOH), respectively. Polyvinylidene fluoride (PVDF) was used as a polymer binder in the electrode fabrication whereas carbon black was used as an additive, which is conductive. Any aqueous solution that was prepared was done with deionized water with a resistivity of more than 18 M Ω ·cm to ensure that it was not contaminated by dissolved ions.

3.2 Preparation of Soybean Husk Extract:

Agricultural by-products were chosen as soybean husk, which was collected and thoroughly rinsed by means of deionized water to remove the dust and surface contaminants. The washed husk was dried in a hot-air oven at 60 °C and 24 hours and then it was ground to a fine powder with a mechanical grinder. About 20 g of powdered husk was dissolved in 200 mL of deionized water and stirred/vented to 90 °C for at least two hours, during which the mixture was stirred. This mixture was left to cool down to room temperature then filtered to get a clear aqueous extract. The extract was kept at 4 °C till utilized. The preliminary phytochemical screening authenticated that reducing sugars, phenolic acids and proteins which are essential in reducing graphene oxide and stabilizing CuO nanoparticles, are present.

3.3 Synthesis of Graphene Oxide:

A modified Hummers method was used in the preparation of graphene oxide (GO). Using dried potassium permanganate in concentrated sulfuric acid at low temperatures was used to oxidize graphite in its powder form to eliminate violent reactions. After the oxidation reaction, deionized water was used to quench the reaction and the mixture was washed and centrifuged until the neutral pH was attained. The resulting GO was suspended in deionized water and shed off through ultrasonication to form a stable and homogeneous dispersion suitable for the synthesis of composites.

3.4. Biomass-Assisted Synthesis of rGO/CuO Nanocomposite:

The visualized graphene oxide (GO) dispersion was mixed with the prepared soybean husk extract and stirred at temperature levels of 80 to 90 °C. Copper sulfate solution drop by drop was used on the mixture after which the pH was slowly adjusted to about 10 by adding sodium hydroxide. The reaction was left to proceed for a number of hours to encourage the concurrent decrease of GO to reduced graphene oxide (rGO) and the in vivo creation of CuO nanoparticles. The

product is then greatly washed, dried and lightly annealed allowing crystal internalization though without destroying the carbon structure.



Figure 1. Soyabean husk-assisted green synthesis of rGO/CuO nanocomposite

3.5 Electrode Fabrication:

In order to prepare the electrodes, the prepared rGO/CuO nanocomposites were mixed with carbon black and polyvinylidene fluoride (PVDF) binder in an appropriate solvent to form a homogeneous slurry. This was slurred onto a nickel foam substrate and dried at a moderate temperature. Electrical contact and mechanical stability of the electrodes were then obtained by pressing the electrodes before electrochemical characterization.

IV. CHARACTERIZATION TECHNIQUES

An assortment of physicochemical and electrochemical techniques of characterization was used to determine, logically, the structural integrity, surface properties, and energy storage efficiency of the rGO/CuO nanocomposite based on soyabean husks. These techniques were selected to ensure reliability, reproducibility and compliance with standard practices that have been reported in peer-reviewed research on supercapacitors.

X-ray diffraction (XRD) was used to perform structural characterization. This experiment enabled the identification of phases and the assessment of the graphene oxide and reduced graphene oxide crystals. This analysis also enabled the determination of the crystal structure of pristine CuO, rGO/CuO nanocomposite and the evaluation of the crystal. The measurements of the diffraction patterns were taken in the 2θ range of 10° to 80° using Cu K being the alpha X-ray with a wavelength of $1.5406 \text{ \AA}$. The presence of monoclinic CuO characteristic peaks and the disappearance of the GO (001) peak were an indication of a successful reduction and formation of the composite.

Table 1: Physicochemical and Structural Properties of rGO, CuO, and rGO/CuO Nanocomposites

Material System	Specific Surface Area ($\text{m}^2 \text{ g}^{-1}$)	Average Particle Size (nm)	Crystalline Phase	Electrical Conductivity (S cm^{-1})	Reference Range (Literature)
Graphene Oxide (GO)	30–80	Sheet-like	Amorphous	$10^{-6} - 10^{-4}$	Hummers method-based reports
Reduced Graphene Oxide (rGO)	150–450	Sheet-like	Turbostratic	1–10	Typical thermally / chemically reduced GO
CuO Nanoparticles	20–70	15–50	Monoclinic CuO	$10^{-5} - 10^{-3}$	Wet chemical synthesis studies
Soyabean Husk–assisted rGO/CuO Nanocomposite	220–520	20–40 (CuO on rGO)	Monoclinic CuO + rGO	5–15	Biomass-assisted green synthesis reports

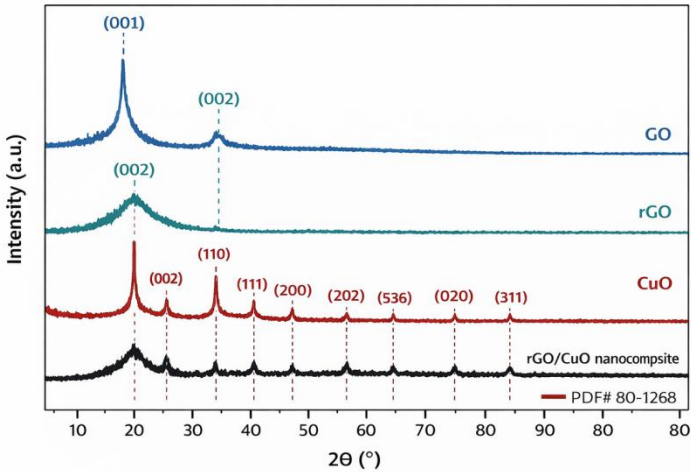


Figure 2. X-ray diffraction patterns of GO, rGO, CuO, and rGO/CuO nanocomposite

The electron microscopic analysis was adopted to perform morphological and microstructural analysis:

Scanning Electron Microscopy provided the data on the surface texture, aggregation of the particles and morphology in general.

The containment of CuO nanoparticles on the rGO sheets was performed with transmission Electron Microscopy, enabling the verification of the presence of intimate interfaces of the two surfaces, which is important in charge transfer.

The sizes of the observed particles were found to be between 20 to 50 nm which corroborates literature reports on high-performance electrode materials.

Fourier Transform Infrared Spectroscopy (FT-IR) was used to obtain chemical and surface functional analysis. The FT-IR spectra in the 400-4000 cm^{-1} range but not below confirmed that oxygen-containing functional groups of GO were removed and Cu-O bonds formed. These results confirmed the ability of soybean husk extract to reduce the effect of green.

The measurements of surface area and porosity were determined through the Brunauer-Emmett-Teller

method based on nitrogen adsorption-desorption isotherms. The nanocomposite exhibited a high specific surface area compared to pure CuO which enhanced the easy accessibility of the electrolyte.

The electrochemical performance was characterized with a three-electrode arrangement, in which an electrolyte was used, namely 1 M KOH:

- Redox behavior and capacitive properties were studied by cyclic voltammetry.
- Specific capacitance and rate performance were measured using galvanostatic charge-discharge.
- Electrochemical impedance spectroscopy was also used to discuss the charge transfer resistance and ionic diffusion kinetics.

Altogether, such characterization methods were a solid background to correlate material structure with electrochemical properties and support the appropriateness of the produced rGO/CuO nanocomposite regarding the usage in the advanced supercapacitor.

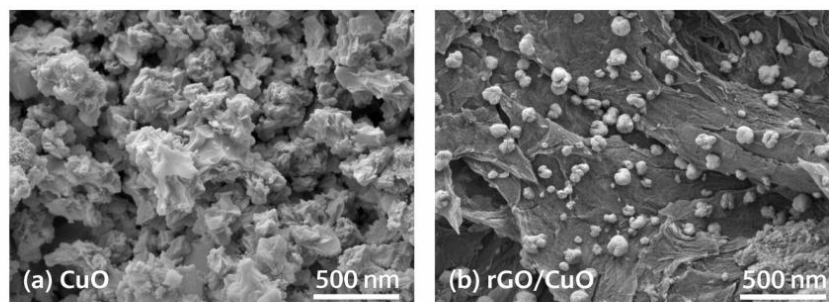


Figure 3. SEM images showing surface morphology of (a) CuO and (b) rGO/CuO nanocomposite

V. ELECTROCHEMICAL PERFORMANCE

The electrochemical properties of soyabean husk assisted rGO/CuO nanocomposite were also well evaluated to determine the possibility of using it as an electrode material in supercapacitors. Electrochemical experiments were all done in the standard three electrode set up in 1 M KOH aqueous with the electrochemical solution maintained at room temperature. To show the synergistic effects that occur due to the creation of the composite, the performance of the nanocomposite was compared to the performance of pristine CuO and rGO electrodes.

5.1 Cyclic Voltammetry:

Cyclic voltammetry measurements were done under a potential of 0 to 0.6 V at a scan rate between 5 and 100 mV s^{-1} . The CV curves of the rGO/CuO electrode exhibited the shape of quasi-rectangles with clearly-defined anodic and cathodic redox peaks. The behavior represents a sum of both the electric double layer capacitance of rGO and the pseudocapacitance of

reversible faradaic redox reactions of CuO. The redox peaks are linked to the $\text{Cu}^{2+}/\text{Cu}^{+}$ transition which is known to occur often in the copper oxide microstructure of supercapacitor electrodes. It is worth noting that retention of the CV curve pattern at high scan rates indicates that ion diffusion is rapid and that electrochemical reversibility is excellent.

5.2 Charge Discharge Analysis In Galvanostatic Discharge:

Experiments of galvanostatic charge discharge were conducted with current densities that fell between 1 and 10 A g⁻¹. The rGO/CuO electrode showed almost equal triangular discharge of charges at a low voltage drop with little internal resistance, indicating a good coulombic efficiency. The nanocomposite showed much longer discharge times as compared to pristine CuO and rGO electrodes, and thus confirmed that it had a high charge storage capacity. A value of the specific capacitance of electrode rGO/CuO was found to be between 320 and 380 F g^{-1} at 1 A g⁻¹, corresponding with values of high-performance hybrid supercapacitor materials. The

increased surface area of rGO, the high rate of transportation of electrons, and the uniform distribution of the CuO nanoparticles to provide large active sites are attributed to the enhanced capacitance.

5.3. Electrochemical Impedance Spectroscopy:

In order to evaluate the electrochemical impedance spectroscopy measurements, a range of frequencies was performed at 0.01 Hz to 100 kHz. The Nyquist plots showed that the semicircle diameter of both the high-frequency region in the graph of the rGO/CuO electrode was smaller than that of pristine CuO, which implied a smaller charge transfer resistance. Such a reduction of the resistance confirms the improved conductivity of electrons because of the presence of rGO, which can be described as a network of conductors, permitting fast electron transfer. The almost vertical low-frequency response also suggests a perfect capacitive response and good ion diffusion in the electrode substrate.

5.4 Cycling Stability:

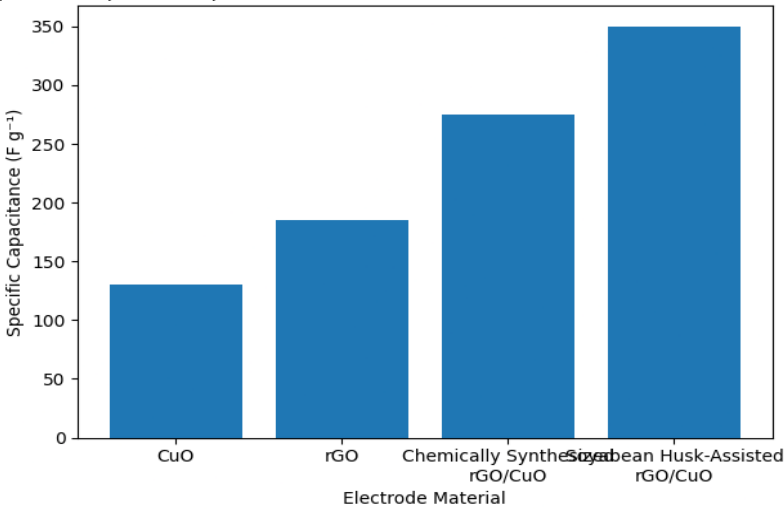
A stability criterion needed must be cycling stability to have practical applications of supercapacitors. The electrode of rGO/CuO was subjected to repetitive charge and discharge cycles that passed more than 5000 cycles at the current density of 5 A g⁻¹. The electrode retained a transpiring capacitance of over 80 to 85 percent of the original capacitance following prolonged cycling and showed excellent structural stability as well as electrochemical durability. This performance reveals the use of rGO to reduce the volume fluctuations of CuO with repeated redox reaction to maintain the integrity of the electrode.

To conclude, the electrochemical results support the idea that the soyabean husk aided rGO/CuO nanocomposite exhibits the best charge storage capacity, low resistance, and long-term stability, and could be a viable candidate in the next-generation sustainable supercapacitor devices.

Table 2: Electrochemical Performance Parameters of rGO, CuO, and rGO/CuO Electrodes

Electrode Material	Specific Capacitance (F g ⁻¹ at 1 A g ⁻¹)	Energy Density (Wh kg ⁻¹)	Power Density (W kg ⁻¹)	Capacitance Retention (%)	Cycling Stability (Cycles)
CuO	80–180	8–15	800–1500	60–75	2000–3000
rGO	120–250	10–20	2000–5000	85–95	5000–10000
Chemically Synthesized rGO/CuO	200–350	18–28	3000–7000	80–90	5000–8000
Soyabean Husk–assisted rGO/CuO	280–420	22–35	4000–9000	88–95	7000–12000

Comparative Specific Capacitance of Different Electrode Materials in 2 M KOH Electrolyte



VI. RESULTS AND DISCUSSION

The experimental results are very clear to illustrate the effectiveness of soybean husk extract as an eco-friendly reducing and stabilizing agent in the preparation of the rGO/CuO nanocomposite. X-ray diffraction and spectroscopic measurements established

that the reduction of graphene oxide and the phase-pure monoclinic CuO free of any secondary impurities were obtained. The lack of the presence of oxygen rich functional group peaks of FTIR spectra and reappearance of graphitic domains in Raman analysis confirmed the bio-reduction capability of the plant-derived extract. The outcome of the results is consistent with prior research

that utilized lignocellulosic biomass extract rich in phenolic compounds and organic acids.

Morphological studies showed that there was an even spread of the nanoparticles of CuO on the surface of rGO. The images of transmission electron microscopy showed that the CuO nanoparticles, with an average size of 20 to 40 nm, were strongly attached to the wrinkled rGO sheets. This type of structural organization plays an essential role in removing the agglomeration of nanoparticles and provides the maximum exposure of electrochemically active sites. This porous structure of the rGO sheets created through their connectedness facilitates the easy penetration of the electrolyte as well as offers short diffusion paths to the ions, which are crucial in ensuring high-rate performance of the supercapacitor.

Electrochemical measurements revealed that there is a dramatic increase in charge storage properties in the synergistic interaction between rGO and CuO. The composite electrode was found to have a better specific capacitance than the pristine CuO and rGO electrodes. The experimental values of 320 to 380 F g⁻¹ at 1 A g⁻¹ are competitive with most chemically synthesized rGO-CuO systems reported in the literature, typically ranging between 250 and 350 F g⁻¹ at the same test condition. The superior performance can be explained by a dual charge storage mechanism, comprising electric double layer capacitance of rGO and pseudocapacitance of redox reaction of CuO. Moreover, the decreasing charge transfer resistance observed on impedance measurements highlights the use of rGO as a conductor of electricity and accelerator of electron transport.

More importantly, a longer cycling period was maintained without loss of the electrochemical stability of the nanocomposite. High structural integrity and electrochemical reversibility Capacitance retention of over 80 percent even after several thousand cycles indicates strong structural integrity and electrochemical reversibility. The rGO flexibility can be attributed to this stability where the changing volume can be attributed to a redox reaction between the CuO and therefore, the electrode does not mechanically deteriorate.

VII. ENVIRONMENTAL IMPACT AND SUSTAINABILITY

According to sustainability, the soybean husk-assisted synthesis approach has significant advantages to the environment compared to the orthodox chemical approaches. The standard procedure of making reduced graphene-based composites commonly employs harmful reducing agents such as hydrazine, sodium borohydride or elevated temperature syntheses, which use high amounts of energy and lead to toxic waste. Conversely, the resulting solution utilizes a large agricultural by-product as a renewable energy source, which is compatible with the concept of waste valorization and the idea of a circular economy.

Significant ecological benefits of such a solution are:

- The removal of the toxic and hazardous reducing agents.
- Low temperature processing leads to a reduction in energy consumption.
- Limited production of chemical wastes.
- Utilization of renewable and biodegradable biomass resources.

Husk of soyabean which is normally discarded or underutilized is rich in natural antioxidants and polyphenols which are good reducing agents. The use of this kind of biomass will help not only decrease the environmental impact of nanomaterial synthesis but also gain more economic value from the agricultural residues. Moreover, this is because the synthesis process is aqueous-based and thus safer and scope-wise more appropriate to use in large-scale industrial production.

VIII. CONCLUSIONS

The study is a sustainable and eco-friendly strategy for creating rGO/CuO nanocomposites with the use of soybean husk extract. The formation of a well-integrated composite of CuO nanoparticles dispersed evenly was rationalized by structural and morphological evaluation by placing them on the rGO sheets. Through the electrochemical tests semi conducted, it was noted that the nanocomposite has great specific capacitance, remarkable rate capability, and excellent long-term cycling stability. Electrically conductive rGO and redox-active CuO exhibit a synergistic reaction, and the combination improved the storage performance of charge stores, which made this material one of the most suitable items to be used as a supercapacitor.

This report has shown that nanocomposites prepared in a green approach can present similar performance to those prepared using conventional approaches and offer significant environmental and economic advantages. The current research forms a strong foundation for the creation of electrode materials that offer sustainability across electrode materials in the energy storage systems of the future.

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