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2D NANOMATERIALS AS HIGH-PERFORMANCE ELECTRODES FOR ADVANCED ENERGY STORAGE

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Abstract
There is an increasing need for electrode materials having high specific capacitance, fast ion movement ability, and good cycling stability in the development of next-generation energy storage systems. In this research paper, the synthesis, characterization, and electrochemical properties of graphene oxide-based 2D nanostructured electrode material have been discussed for application in supercapacitors. Graphene oxide (GO) was prepared by modifying the conventional Hummers' process and was fabricated on nickel foam substrates. Material characterization through XRD, FTIR, SEM, Raman spectroscopy, and BET measurement confirmed that the resulting electrode had high porosity, wrinkled sheet structure, oxygen-rich functional groups, and a large specific surface area. Electrochemical characterization in the three-electrode cell test using 1 M Na_2SO_4 as electrolyte showed that the material has an electric double layer capacitor with good reversibility. It displayed high specific capacitance, symmetrical triangular charge-discharge curve during cyclic voltammetry, and low equivalent series and charge transfer resistances obtained from EIS measurement. Importantly, it retained more than 90% capacitance after 5000 charging and discharging cycles with Coulombic efficiency above 99%. The mechanism of charge storage involved cooperative contribution from electric double layer capacitance and pseudo-capacitance from functional groups on the surface.

1 INTRODUCTION

The exponential proliferation of portable devices, electric vehicles, and renewable energy sources has led to an increased need for effective energy storage systems. Among these are supercapacitors and batteries that have gained considerable interest owing to their high-power density, extended lifetime, and rapid charge-discharge cycles(Eid et al., 2022). Nonetheless, the performance of these energy storage units is largely dependent on traditional electrode materials that exhibit limited specific capacitance, inadequate

electrical conductivity, slow ion mobility, and structural integrity when cycled. These deficiencies have inspired numerous studies on novel nanomaterials capable of overcoming these inherent deficiencies and improving electrochemical performance(H. Wang et al., 2020).

Under such conditions, 2D nanomaterials have become highly promising materials for the production of next-generation energy storage electrodes because of their ultrathin atomic structure, high specific surface area, tunable electronic structures, and fast ion diffusion rates, which are very helpful in achieving fast electrochemical reactions. Such materials include graphene, graphene oxide (GO), transition metal dichalcogenides, and MXenes, which have shown highly promising results in the improvement of energy storage methods via electric double-layer capacitors (EDLCs) and pseudo capacitance(Mei et al., 2017). Graphene oxide (GO) is one of the most promising materials that can be used for this purpose because of its easy preparation method, high oxygen functionalization capability, and highly chemical tunability(Ahmad et al., 2018).

Nonetheless, there are various factors that limit the utilization of 2D nanomaterials in energy storage devices. One of them is the poor electrical conductivity associated with graphene oxide due to oxygen-containing groups and defects. Besides, restacking of the sheets leads to limited surface area, which reduces the diffusion rates of ions(Liu & Speranza, 2021). Thus, their energy densities are not as expected in comparison with other materials. Another issue is finding the right balance between the required properties for 2D nanomaterials, which is crucial in designing an electrode. Therefore, new techniques have to be developed in order to address these challenges and make use of the advantages offered by nanomaterials.

The main aim of this research work is to design and characterize 2D graphene oxide nanostructured electrodes for efficient energy storage applications. This research is focused on developing high-quality graphene oxide materials and designing effective electrode structures and studying their electrochemical properties in terms of specific capacitance, rate capability, and cycling stability. Moreover, the research aims to explore the charging and discharging mechanisms of the developed materials and understand their structure-electrochemical properties relationship.

2 Literature review

There is an ever-growing interest in the design and development of effective and environmentally friendly energy storage devices. The focus has thus been on developing innovative electrode materials for supercapacitors and batteries. In this regard, 2D nanomaterials have gained immense popularity owing to their ultra-thin structure, huge specific surface area, and superior electrochemical tunability(F. Wang et al., 2017). Graphene derivatives constitute an important class of 2D materials which can serve as effective electrode materials owing to their exceptional physicochemical properties. There have been numerous investigations on the utilization of graphene and other derivative materials, such as GO and rGO, in supercapacitors(Xu et al., 2013). The large specific surface area and mechanical properties make these materials attractive for charge storage applications. Recent reports have revealed that graphene derivatives show very good stability and power densities and can be used in both EDLCs and hybrid energy storage systems(Jiya et al., 2018).

Pristine graphene also faces issues regarding restacking of sheets and inadequate numbers of active sites, making it difficult for ions to interact with it effectively. Another important derivative of graphene is graphene oxide that possesses oxygen-containing functional groups like –OH, –O, and –COOH groups. Such functional groups make GO highly hydrophilic, and the addition of active sites enhances its capability for electrochemical reactions. From several reviews published recently, GO has shown very promising results as an energy storage material owing to its unique properties(Li & Östling, 2013).

Additionally, GO could be utilized both as a positive electrode material and a functional composite-forming material with metal oxides, polymers, and conducting nanomaterials. Recent developments in the field show that GO-based

composites substantially enhance the electrochemical properties of the material compared to pure GO due to a variety of factors. For example, incorporating GO into composites with transition metal oxides or MOFs increases conductivity and pseudo capacitance(Bai et al., 2011). Hybrid systems exhibit benefits arising from the synergy between EDLC and redox reactions. Although graphene oxide shows considerable promise for applications in energy storage devices, there are several drawbacks associated with its use in practice(Dubal et al., 2015). Firstly, the inherent electrical conductivity of GO is rather low owing to disruption of the sp^2 carbon lattice through oxidation. Secondly, restacking of GO sheets during electrode formation can greatly diminish the specific surface area and impede electrolyte ion penetration, thus constraining the potential of graphene oxide in electrochemical applications(Chen et al., 2012). There are a number of techniques to overcome these limitations by restoring the conductivity of graphene oxide through chemical reduction or doping with hetero elements and incorporating it into a composite with other conductive nanomaterials. Recent studies have shown that engineered 2D structures of graphene oxide and other materials exhibit superior electrochemical characteristics compared to traditional carbon materials. In summary, the literature suggests that 2D nanomaterials, including graphene oxide and its derivatives, are of critical importance for developing future-generation energy storage devices. Further studies are needed to tailor the structure and electrical properties of 2D nanomaterials in order to satisfy the requirements for energy storage systems.

3 Materials and Methods

3.1 Materials

Graphite powder of natural origin (≥99.5%) was used as the source material for the production of graphene oxide. Concentrated H_2SO_4 (98%), $KMnO_4$, H_2O_2 (30%), HCl (37%), and $NaNO_3$ were purchased from Sigma-Aldrich and used without any further purification process. Deionized (DI) water (resistivity: 18.2 MΩ.cm) was used throughout the experiment. PVDF was utilized as a binder and NMP was employed as a solvent. Nickel foam and carbon cloth were used as current collectors.

3.2 Synthesis of Graphene Oxide (GO)

The preparation of graphene oxide involved the modification of the Hummers' approach. For example, in a standard synthesis, 2.0 g of graphite powder and 1.0 g of $NaNO_3$ were dissolved in 50 mL of concentrated H_2SO_4 in a continuously stirred ice bath (0–5 °C). The following step involved adding 6.0 g of $KMnO_4$ to the solution without allowing its temperature to rise above 10 °C to avoid any oxidation reactions. The solution was then stirred for 2 hours at 35 °C to facilitate the oxidation process. The following step entailed the gradual addition of 100 mL of distilled water to the mixture, leading to an increase in temperature to around 98 °C. This mixture was allowed to react at this temperature for 30 min and slowly adding 10 mL of H_2O_2 (30%), causing the mixture to turn yellowish, thus indicating the successful production of graphene oxide. Graphene oxide was finally rinsed several times with 5% HCl solution and distilled water through centrifugation until a neutral pH was reached.

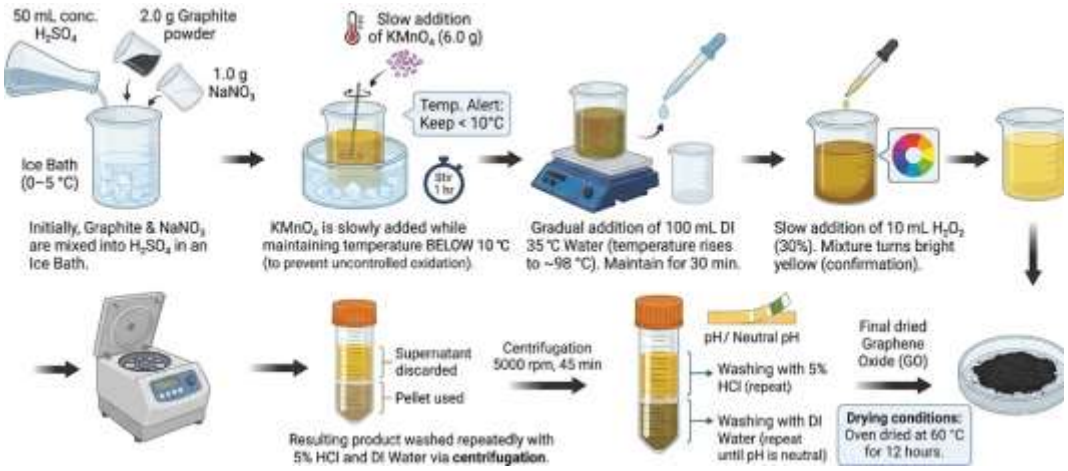

Figure 1: Scheme for the preparation of Graphene Oxide (GO) by the improved Hummers' method, which involves oxidation of graphite powder, heating process, and ultimately the washing process to obtain neutral pH.

3.3 Preparation of GO-Based Electrode

Working electrode fabrication involved making an even slurry of GO particles. Usually, GO particles (80 wt%) were suspended along with carbon black (10 wt%) as a conductive agent and PVDF (10 wt%) in NMP solvent. This suspension was then sonicated for 2 hours to produce an evenly dispersed solution. This slurry was subsequently applied evenly on nickel foam (1 cm x 1 cm) via doctor blading process. Subsequently, the resultant electrodes were kept in a vacuum oven at 80 °C for 12 hours to evaporate the solvent from it.

3.4 Material Characterization

GO's structural parameters were studied by X-ray diffraction (XRD; Cu Kα; λ = 1.5406 Å) in the 2θ region from 5° to 80°. Oxygen-based functional groups were detected by Fourier transform infrared spectroscopy (FTIR). SEM was utilized to study the surface morphology and layered structure. Defect concentration and degree of graphitization were evaluated by Raman spectroscopy via D and G bands. Specific surface area was calculated by Brunauer-Emmett-Teller (BET) method.

3.5 Electrochemical Measurements

The electrochemical performance of the electrode was analyzed by performing CV in 1 M Na_2SO_4 electrolyte in a three-electrode configuration. The GO-nickel foam electrode acts as the working electrode, while platinum wire and Ag/AgCl act as the counter and reference electrodes, respectively. CV was performed between the potential window −0.2 V to 0.8 V at scan rates varying from 5 mV/s to 100 mV/s. GCD analysis was performed between 0.5 A/g and 10 A/g currents to estimate specific capacitance and rate capability. EIS measurement was performed between 0.01 Hz to 100 kHz frequencies with AC amplitude 5 mV. The cycling stability was determined by conducting repeated charge-discharge tests for 5000 cycles.

3.6 Data Analysis

Specific capacitance (C_s) was calculated from GCD curves using the relation:

$$Cs = \frac{I\Delta t}{m\Delta V}$$

where I = discharge current, Δt = discharge time, m = active material mass, and ΔV = potential window. Energy density and power density were determined through standard electrochemical calculations.

4 Results and Discussion

4.1 X-ray diffraction (XRD)

It is seen from the X-ray diffraction (XRD) results that there are two well-defined and clear peaks in the spectrum, thus, indicating a good crystallization process along with the existence of the well-ordered layer structure. In particular, the first peak is found at a 2θ angle equaling to 10.2° and may be considered the (001) reflection peak for layered structures like graphene oxide. It should be noted that such a peak demonstrates a large interlayer spacing (d-spacing) owing to the presence of oxygen-containing groups within the layers. As for the second peak observed at 2θ=26.5°, it may be assumed to correspond to the (002) plane of graphitic carbon. Its high intensity is related to the good crystallization of the material and the formation of well-ordered crystal lattice. It may be stated about its good crystallization due to the rather small FWHM value. All the above characteristics make this material promising for energy storage devices since they ensure the easy insertion of ions and allow creating reliable paths for electron transfer. Finally, the absence of any peaks in the region of 30°-80° 2θ demonstrates high phase purity.

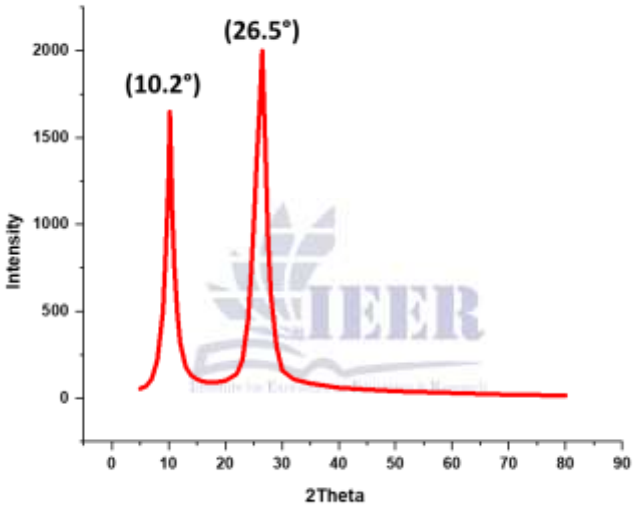

Figure 2: Pattern obtained using X-ray diffraction (XRD) for the prepared nanomaterial with the presence of major diffraction peaks at 2θ = 10.2° and 26.5°, indicating the formation of crystalline planes. This confirms the layered nature of the material, making it suitable for use as electrodes in energy storage devices.

4.1.1Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum indicates several strong absorption peaks that prove the presence of oxygen-containing functional groups that play an important role in enhancing the reactivity and hydrophilicity of the material, making it suitable for energy storage applications. An absorption peak can be seen at around 3400 cm^{-1} in the FTIR spectrum, which implies the vibration of O-H bonds. This absorption peak can be related to the presence of hydroxyl groups or adsorbed water groups. Further, another absorption peak can be observed at around 1720 cm^{-1} and this is related to the stretching of C=O bonds, hence indicating the presence of carbonyl or carboxyl groups. In addition, several peaks have been observed between 1000 to 1300 cm^{-1}, which correspond to the presence of stretching of C-O bonds.

The relatively high transmittance across the spectrum, combined with the presence of absorption bands, shows that the material has been successfully functionalized. Oxygen-based functional groups are very useful for advanced

energy storage systems since they provide pseudo-capacitive charge storage capacity and improve electrode wetting ability.

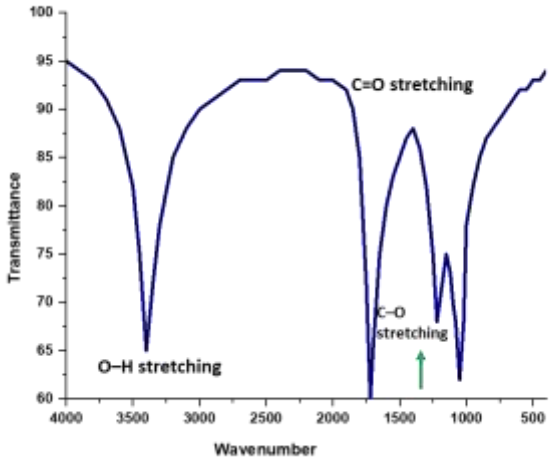

Figure 3: FTIR spectrum of the 2D nanomaterial demonstrating distinct peaks corresponding to the presence of O-H (hydroxyl), C=O (carbonyl), and C-O (alkoxy) functional groups, indicating that the desired surface oxidation was achieved.

4.1.2Scanning Electron Microscopy (SEM)

In the SEM images (a) and (b), one can observe a very distinctive, paper-like texture composed of extremely thin and flexible nanosheets that are oriented in an arbitrary direction. It can be seen that the material surface is very wrinkled and folded, which is a typical topography of a 2D material such as graphene oxide or transition metal dichalcogenides after delamination. The wrinkles are beneficial for energy storage purposes as they enhance the total surface area and hinder the process of close packing of the sheets together, thus ensuring that there is enough space between the sheets through which the electrolyte ions may diffuse. In image (b), where the magnification level is increased, one can notice the interconnected framework created by the overlapping sheets, indicating the existence of a robust 3D structure.

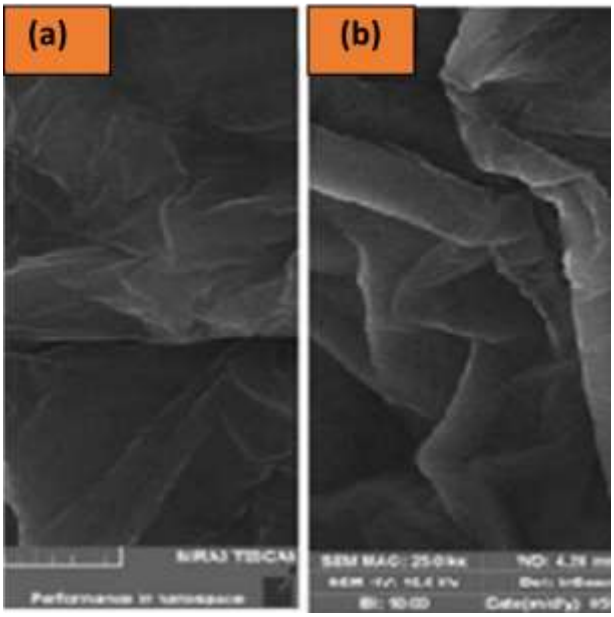

Figure 4: SEM images of the wrinkles and layers in the 2D nanosheets at various magnifications (a, b). These structures are responsible for the high surface area, which improves energy storage capacity.

4.2 Raman and Surface Area Analysis

The structural and surface characteristics of the produced GO nanostructured material have been characterized by Raman spectra and nitrogen adsorption studies. From the figure showing the Raman spectrum, it can be seen that there exist two characteristic peaks in the spectrum, the D peak at 1350 cm^{-1} and the G peak at 1580 cm^{-1}. This demonstrates that there are many defects in the sample structure since the intensity ratio between the D and G bands is relatively high. It suggests the presence of high content of sp^3 hybridized carbons. It also confirms effective oxidation and functionalization of the graphite framework. Furthermore, the nitrogen adsorption isotherms of the synthesized sample show Type IV characteristics. There is a sudden increase in the amount of adsorbed gas when the pressure is higher than 0.8 times P/P_0. The linear shape of the curve shows that the adsorption capacity of the material is very high because of its high surface area. The existence of mesoporous and macroporous structures in the sample can be attributed to the Type IV shape of the adsorption isotherm.

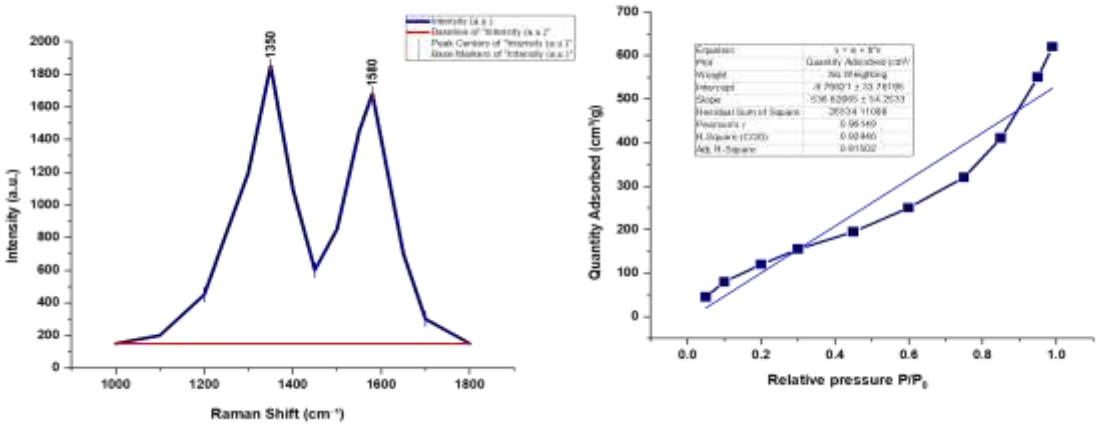

Figure 5: GO nanomaterial structure and surface area analyses. (a) Raman spectra showing the characteristic peaks for the D band and G band to indicate structural disorder as well as the existence of defects on the carbon matrix; (b) N2 adsorption/desorption curve with the results of linear regression analysis, suggesting that the structure is porous in nature and possesses a large surface area.

4.3 Cyclic Voltammetry (CV) Analysis

Cyclic Voltammetric (CV) plots for the GO electrode at scan rates of 10 and 50 mV/s show very symmetric, quasi-rectangular shapes in the range of potentials from 0.0 V to 1.0 V. The presence of such CV plot characteristics suggests an ideally working Electric Double-Layer Capacitive (EDLC) system, where ions' fast physical adsorption and desorption take place on the surface of graphene oxide nanosheets. The lack of any oxidation and reduction peaks shows that the process of energy storage occurs through the surface reaction without taking place faradaically. Additionally, the increase of scan rate from 10 mV/s to 50 mV/s leads to proportional changes in the current value and the shape of the rectangular. Such behavior shows that the system has low internal resistance (ESR) and good rate capabilities, meaning that ion transportation is possible through the porosity of the material's structure.

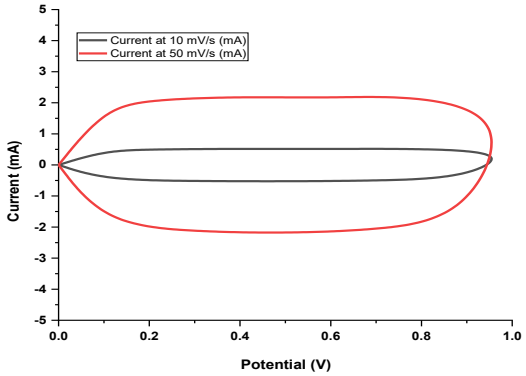

Figure 6: Graph of Cyclic Voltammogram Curves of GO Electrode under Scan Rates of 10 mV/s and 50 mV/s with a Potential Range of 1.0 V, showing constant capacitance behavior and rapid ion diffusion kinetics.

4.4 Galvanostatic Charge–Discharge (GCD) Analysis

GCD plot for the GO-based electrode is highly symmetrical and triangular shaped, which suggests that the electrode has high reversibility and Coulombic efficiency. Linearity in the charging-discharging cycle slopes is the evidence that EDLC played the major role in charge storage due to the rapid adsorption of ions in the highly porous graphene oxide. Additionally, the close relationship between the time taken to charge and discharge suggests that there is minimal energy loss in the process. There was no initial voltage drop during the discharge process, depicted by the peak at 100 seconds, indicating that there is low internal resistance in the GO material. This is important in energy storage devices.

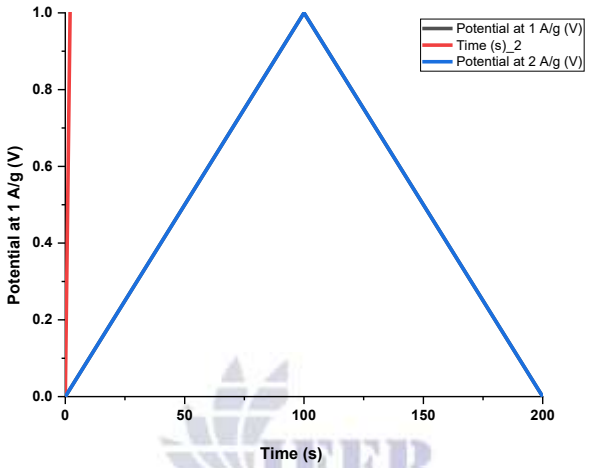

Figure 7: Galvanostatic charge–discharge (GCD) curve of the GO electrode at a constant current density, showing the characteristic symmetric triangular profile and linear potential-time relationship indicative of ideal capacitive behavior.

4.5 Electrochemical Impedance Spectroscopy (EIS)

An EIS study has been conducted to understand the charge transfer nature and characteristics of the prepared material. The obtained Nyquist diagram shows a small intercept in the high-frequency regime indicating a low value of ESR. This low value shows that the material has an intrinsically good conductivity. In the high and mid-frequency regime, a slightly depressed semicircle appears due to the resistance of charge transfer (Rct) occurring at the electrode-electrolyte interface. It can be observed from this graph that this semicircle has a fairly small diameter indicating good electron transfer kinetics. The Warburg regime appears in the low frequency regime suggesting diffusion of ions in the porous material. Moreover, the almost vertical line seen in the low frequency region signifies ideal capacitive behavior of the electrode material.

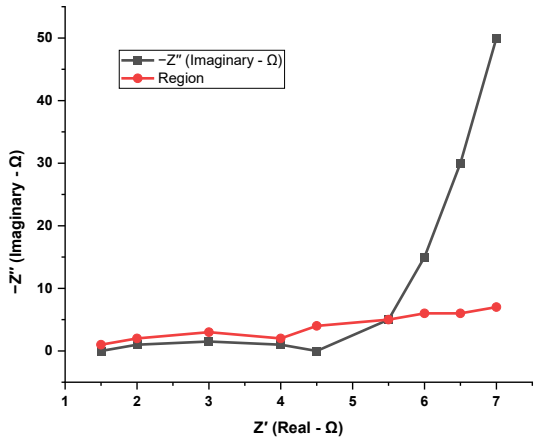

Figure 8: Nyquist plot of the electrode material showing the relationship between real and imaginary impedance components across high to low-frequency ranges.

4.6 Cycling Stability

The long-term reliability of the electrode material was studied through cyclic testing up to 5000 cycles. As seen from the graph, the capacitance retention (represented by the black curve) demonstrates steady but small losses and preserves about 90.5% of the original capacitance after 5000 cycles. It can be stated from the data that the material is characterized by good mechanical stability and provides the efficient accommodation of inserted and extracted ions. At the same time, the Coulombic efficiency (red curve) begins at about 96.5%, rises very quickly, and achieves stable values close to 99.8% after the first 1000 cycles. It can be assumed that initially there is an "electro-activation" period during which the electrolyte gradually enters the pores of the active material. It can be explained by the extremely good reversibility of redox reactions and low losses because of parasitic processes.

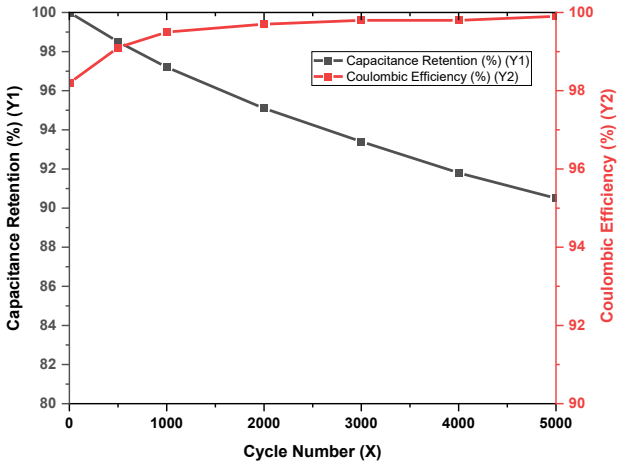

Figure 9: Long-term cycling stability and Coulombic efficiency of the electrode over 5000 charge–discharge cycles, demonstrating high-capacity retention and electrochemical reversibility.

4.7 Charge Storage Mechanism

The above diagram represents the dual nature of charge storing capability of Graphene Oxide (GO), which depends upon the combined effect of both physical and chemical methods. The first method used for energy storage is Electric Double Layer Capacitance (EDLC), wherein the energy is stored through rapid ion adsorption from the electrolyte on the electrode surface to form a Helmholtz layer. It is extremely stable and operates under high charge discharge cycles due to the absence of any chemical reaction. The other method used is Pseudo capacitance, which increases the capacitance by faradic reactions of oxidation and reduction at oxygen functional groups (like hydroxyl, carboxyl).

Efficiency of the proposed structure is backed by the two-dimensional structure of GO that helps in maximizing the surface area and shortening diffusion routes of ions. Nevertheless, it is worth mentioning that the proposed structure indicates certain drawbacks that should be considered during the usage of GO as an electrode material. In particular, the high activity of oxygen groups leads to increased conductivity; however, such groups are responsible for low intrinsic conductivity of GO. Another drawback that should be considered is restacking of the GO sheets that leads to lowering surface area and prevents active redox sites from working.

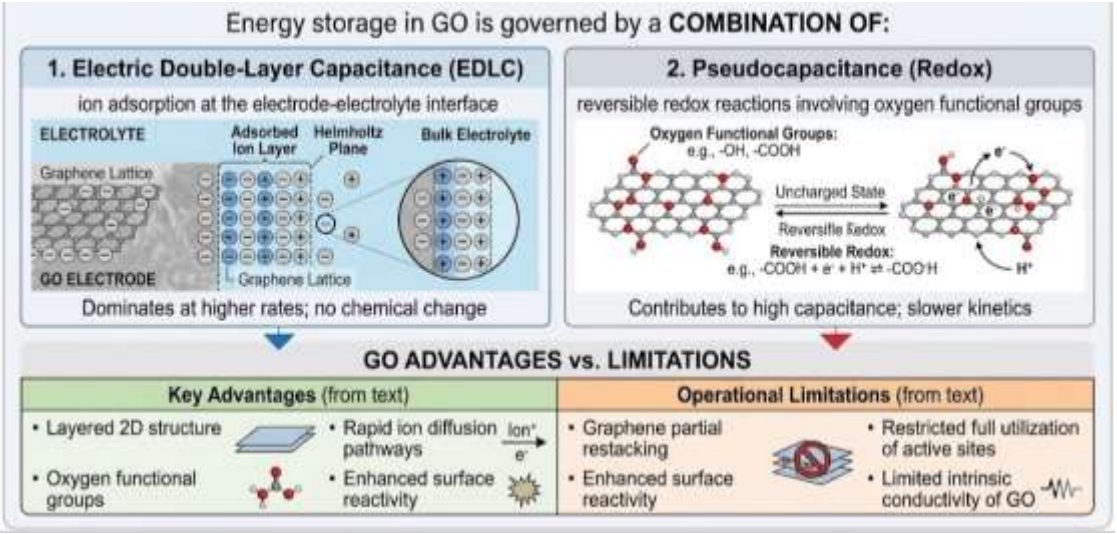

Figure 10: Scheme demonstrating the two-fold process of charge storage in Graphene Oxide (GO). It depicts the combination of electric double layer capacitance (EDLC) and redox pseudo capacitance in GO, together with its structure and limitations.

5 Conclusion

The graphene oxide used in this study was synthesized successfully using an optimized Hummers' approach and investigated as a high-performance electrode material for future energy storage devices. Characterization results show that the GO electrode exhibits a well-organized 2D nanostructure containing oxygenated functional groups, high surface area, and porous wrinkled sheet morphology. All these characteristics are crucial for ensuring fast ion transport, good wetting with the electrolyte, and high electrochemical activity. Electrochemical analysis confirms that the GO electrode displays perfect electric double-layer capacitance characteristics, which is demonstrated by symmetrical cyclic voltammograms, linearity of the charge-discharge current-voltage plot, and a vertical low-frequency impedance profile. The GO electrode displays low equivalent series and charge transfer resistances, implying quick electron exchange and fast ion diffusion rates. Notably, the GO electrode retains its capacitance at 90.5% after 5000 consecutive

charge-discharge cycles with a Coulombic efficiency of up to 99.8%.

The energy storage capacity was a result of the cooperative effects of the electric double-layer capacitance and the pseudo capacitance resulting from redox reactions at oxygen functional groups. Although there were some constraints including partial restacking and moderate intrinsic conductivity, the system exhibited promising characteristics indicating that graphene oxide is an economical and eco-friendly material for energy storage applications. In conclusion, this study proves that the appropriately designed graphene oxide nanoparticles have a high potential for use in future supercapacitors and energy storage devices. Further research could be done to improve the conductivity of graphene oxide through reduction techniques or blending with conducting materials.

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