

Facile Synthesis and Comprehensive Characterization of Reduced Graphene Oxide (rGO)/Manganese Oxide (MnO_2) Nanocomposites Derived from Graphite Powder Via Graphene Oxide Precursor Route for High-Performance Supercapacitor Electrode Applications

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ABSTRACT

A Simple in-situ synthesis technique in which reduced Graphene Oxide (rGO) and Manganese Oxide (MnO_2) were synthesized in the same pot was adopted for the synthesis of rGO/ MnO_2 nanocomposite because of its advantages being that it is one-pot, cost-effective, scalable, and there isn't any need for separate synthesis and post-assembly steps. The atomic structure, functional groups, surface morphology, specific surface area, and elemental composition of the rGO/ MnO_2 nanocomposites were analyzed by means of X – Ray Diffraction (XRD) spectroscopy, Fourier Transform Infra – Red (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), Energy Dispersion X-ray (EDX) spectroscopy and Brunauer–Emmett–Teller (BET) analysis. The SEM study inveterate the surface roughness which is advantageous to shortening the travel distance of electrons thereby enhancing good diffusion and increasing conductivity and presage the enhanced energy storage properties of the nanocomposites developed in this work. The BET surface analysis show that the material is mesoporous with the pore size found to be 2.115 nm and large surface area of $359.64\text{m}^2\text{g}^{-1}$. The electrochemical performance of the synthesized samples was investigated using cyclic voltammetry (CV) analysis and electrochemical impedance spectroscopy (EIS) technique. The result obtained show that, the composite has a high specific capacitance of 347.25Fg^{-1} , energy density of $\approx 107.1\text{WhKg}^{-1}$ which is near battery – level and power density of $\approx 55\text{KWKg}^{-1}$. The time constant of the nanocomposite is 3,507.7s or about 58.5 minutes which is the standard time it will take the supercapacitor to charge up to 63.2% of its maximum voltage, or discharge down to 36.8% of its initial voltage.

Keywords: Reduced Graphene Oxide; Manganese Oxide; rGO/ MnO_2 Nanocomposite; In-situ Synthesis; One-pot Technique; X-Ray Diffraction; Fourier Transform Infrared Spectroscopy; Scanning Electron Microscopy; Energy Dispersive X-ray Spectroscopy; Brunauer–Emmett–Teller (BET) Analysis; Surface Morphology; Mesoporous Structure; Specific Surface Area; Cyclic Voltammetry (CV); Supercapacitor Application.

1.0. Introduction

There has been concerted effort by scientists and researchers to mitigate the consequences of the world's ever-growing population coupled with technological revolution which has caused the scramble for energy, thereby causing a huge deficit in energy globally. In recent time, a host of technologies has become essential for everyday living. These includes, portable devices like mobile phones, tablets, hand – held tools and even some complex technologies like hybrid electric vehicles (HEV) [1, 2].

Hitherto, there has been overdependence on the energy from fossil fuel and others from environmental degradation which has great negative environmental impact and poses a lot of danger to both humans and the environment itself, hence the need for alternative source of energy [3, 4]. Renewable energy sources have evolved as the solution for these pitfalls of traditional energy sources although it has its own limitations which include; intermittent supply, regional dependence, and sometimes seasonal [5, 6]. For this reason, serious research interest has been ignited in the area of energy storage systems such as supercapacitors, fuel cells and batteries [7] with a view to produce cheap, efficient and sustainable energy storage devices to harness and store for future use this renewable energy [8].

Supercapacitor (Sc) also referred as ultracapacitors or electrochemical capacitors (ECs) [9] are charge storage devices that operate based on the electrical double layer on the electrode /electrolyte interface to store charges [10].

The electrode is an active part of the Sc, hence the material for the electrode should have a good electrical conductivity, large surface area, good porous structure and good redox property [11, 12]. Supercapacitor is at the forefront [13] of the energy storage revolution, offering high power density, rapid charging and discharging capabilities and extended life span over traditional batteries [9]. These features have positioned it to be indispensable in applications demanding quick energy bursts and high efficiency such as automobiles [14, 15], renewable energy systems, consumer electronics and industrial power management [16, 17]. Hence, there have been significant investment in research and development of supercapacitors to reduce manufacturing costs as the demand for efficient and sustainable energy solution intensifies because it belongs to the future energy. Summing it up, supercapacitors are unarguably a crucial part of future energy strategies because this evolving energy field is not only propelling technological innovation but also contributing substantially to the green energy transition [16, 18, 19, 20].

Reduced graphene oxide (rGO) shows strong supercapacitive properties due to its high surface area, electrical conductivity, and tunable oxygen functional groups. It mainly contributes to electric double-layer capacitance (EDLC) but can also exhibit pseudocapacitance when functionalized or hybridized with redox-active materials [21, 22, 23]. It can be advantageous for high power density, long cycle life, lightweight and flexible and above all can be produced from graphite oxide using relatively low-cost methods.

However, it suffers from restacking, which reduces the accessible surface area and results in low intrinsic capacitance, as the oxygen-containing groups may degrade during long-term cycling [24, 25]. On the other hand, MnO_2 which belong to the transition metal oxide (TMO) family is a promising pseudocapacitive material with high theoretical capacitance (~ 1370 F/g), environmental friendliness, low cost and exists in multiple polymorphs (α -, β -, γ -, δ - MnO_2) [26], each with distinct ion diffusion and capacitance properties. However, its poor conductivity and cycling stability limit practical applications. The most effective strategy is to use MnO_2 in composites with conductive supports like rGO, CNTs, or conducting polymers, which enhance electron transport and stabilize the structure [27, 26].

There have been several synthesis protocols which many researchers have employed to synthesize the composite of rGO/metal oxides each of them is designed to combine the high conductivity and surface area of reduced graphene oxide (rGO) with the pseudocapacitive properties of metal oxides [28]. The most common methods include hydrothermal [29, 30], sol-gel [31], co-precipitation [32], in-situ reduction [33, 34], and chemical vapor deposition [35]. Each has trade-offs in scalability, uniformity, and electrochemical performance.

In-situ reduction is one of the most effective protocols for synthesizing rGO/metal oxide nanocomposites, because it simultaneously reduces graphene oxide (GO) to rGO while forming metal oxide nanoparticles directly on its surface. The oxygen functional groups on GO act as nucleation sites [36], ensuring nanoparticles are well-dispersed and strongly attached. This creates strong interfacial bonding and uniform dispersion, which are critical for electrochemical performance in supercapacitors. rGO provides a conductive backbone, while the metal oxides contribute pseudo capacitance. This process has also been proved to reduce synthesis steps, maximize active surface area for redox reactions which always result to higher capacitance and better cycling stability in

electrochemical performance [37, 38, 39]. However, it requires careful optimization to balance reduction degree, particle size, and dispersion [37, 40]. In our investigation we leverage on the overwhelming advantage of in-situ reduction method to synthesize and optimize our composite electrode. Additional benefits of in-situ reduction method are short diffusion routes [41] and less agglomeration [39], easy and affordable instrumentation [38], also the ability to synthesize complicated, very pure binary electrode material [40]. Hence, we have investigated rGO/MnO₂ nanocomposites for supercapacitive applications.

1.1. Objectives of the Study

The objectives of this study are;

- 1). To synthesize reduced graphene oxide (rGO) and manganese oxide (MnO₂) nanocomposites using a simple, one-pot in-situ technique that is cost-effective, scalable, and eliminates the need for separate synthesis and post-assembly steps.
- 2). To characterize the structural, morphological, and compositional properties of the rGO/MnO₂ nanocomposites using XRD, FTIR, SEM, EDX, and BET analyses.
- 3). To investigate the surface features, including mesoporosity, pore size distribution, and specific surface area, and correlate these with electron transport and conductivity.
- 4). To evaluate the electrochemical performance of the synthesized nanocomposites through cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS), focusing on specific capacitance, energy density, and power density.
- 5). To determine the time constant and charge–discharge behavior of the nanocomposites, assessing their suitability for supercapacitor applications.

2.0. Materials and Methods

2.1. Materials

The materials used in this work are; Graphite powder (Carbon – 98%, Fe – 0.4%, Sulphur – 0.1%, Ash content – 0.7%, Phosphorous – Traces), Potassium permanganate (KMnO₄), Sodium nitrate (Na₂NO₃), Hydrogen Peroxide (H₂O₂), sulphuric acid (H₂SO₄, 98%), Hydrochloric acid (HCl) Ethanol, Manganese sulphate (MnSO₄), L – Ascorbic acid and Distilled water which were of analytical grade and purchased from Molychem India. All the chemicals were utilized as obtained.

2.2. Synthesis of Graphene Oxide (GO) via Modified Hummer's Method

Graphene oxide (GO) was synthesized via the modified Hummers method [42], as earlier reported [43] “4 grams of graphite powder and 4 grams of sodium nitrate (NaNO₃) in the ratio of 4:4 were mixed in 150 ml of concentrated sulphuric acid (H₂SO₄)” in a beaker and placed in an ice bath and sonicated for about 30 minutes to ensure complete dissolution using EINS SCI Sonicator Model E-uc6-HD-D after which 20 grams of Potassium permanganate (KMnO₄) was slowly added to the mixture to enhance the oxidation process maintaining the temperature between 15°C - 20°C so as to control the exothermic reaction in the system. The solution was stirred at an elevated

temperature within the range of 35–50 °C for about three hours before diluting it with deionized water. In order to dilute and quench the reaction, deionized water was carefully added to the solution while stirring although at this point the temperature suddenly rose to about 80°C. Quenching the reaction was done by adding 30 ml of 35% Hydrogen peroxide (H_2O_2) and to remove any remaining Potassium permanganate. It was centrifuged using Cole model 0406-2 centrifuge set on 3000rpm for 15 minutes, followed by washing the synthesized graphite oxide with 10% Hydrochloric acid and then with deionised water to remove any residual acids and salts. The graphite oxide was collected as powder, dried at room temperature for 48hrs to bring the final product to graphene oxide (GO).

2.3. Synthesis of reduced Graphene Oxide/ Manganese Oxide Nanoparticles

Simple in-situ reduction method [44] in which rGO and MnO_2 were synthesized in the same pot was adopted for the synthesis of rGO/ MnO_2 nanocomposite which was earlier reported in the literature. 1000 mg of GO was dispersed in 200 ml of distilled water and sonicated for 30 minutes to achieve uniform GO suspension. Also in another separate beaker, 1000 mg of $MnSO_4$ was dispersed in 100 ml of distilled water and sonicated for 10 minutes after which, they were mixed together in a 1000 mL beaker and sonicated for 5 minutes. Then 1000 mg of L – Ascorbic acid was added to the mixture and sonicated for 120 minutes. The reaction was monitored to observe the colour changes indicating the reaction that was taking place after which it was centrifuged for 20 minutes at 10,000 rpm to collect the rGO/ MnO_2 nanocomposite. The collected composite was washed with distilled water and then with ethanol to remove impurities and dried at 60°C for 60 minutes to ensure that it is completely dried.

2.4. Characterization of the Synthesized Nanoparticles

The Graphene Oxide synthesized was analyzed using several techniques. X-ray diffraction (XRD) analysis was carried out using a Rigaku Miniflex-II diffractometer operated at 40 kV and 15 mA with $CuK\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Data were collected over the 2θ range of 20°–70° with a scanning speed of 20° min^{-1} and a step size of 0.02°. Williamson–Hall (W-H) Equation (4) linked the peak broadening to micro strain as well as the crystallite size [45] hence separates size and strain contributions to peak broadening, offering a more in-depth analysis to crystallite size than what was believed. it therefore means that;

Total broadening (β_τ) = broadening due to crystallite size (β_D) + broadening due to micro strain (β_ϵ)

i.e.

$$(\beta_\tau) = (\beta_D) + (\beta_\epsilon) \quad (1)$$

Also, the broadening due to micro strain [45] is given as

$$\beta_\epsilon = 4\epsilon \tan \theta_D \quad (2)$$

Where:

β_ϵ = broadening due to strain, ϵ = the strain, θ = peak position in radians.

From Scherrer's Equation [46], (1) now becomes

$$\beta_\tau = \frac{k\lambda}{D \cos \theta} + 4\epsilon \tan \theta \quad (3)$$

Linearizing equation (3) will give Williamson–Hall (W-H) equation as shown in (4);

$$\beta_{\tau} \cos \theta = \frac{k\lambda}{D} + 4\epsilon \sin \theta \quad (4)$$

Where;

D = average crystallite size, K = shape factor (~0.9), λ = X-ray wavelength (0.15406),

β = full width at half maximum (FWHM) of the peak (in radians), θ = Bragg angle,

ϵ = micro strain [45].

Equation (4) is now a straight-line equation where we can extract the micro strain as the slope while the crystallite size was deduced from the intercept [45]. The elemental composition and surface morphology of all samples were characterized using a SEMPLOR NANOS SEM-EDS system, which integrates scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDX). Furthermore, Fourier Transform Infrared Spectroscopy (FT-IR) was employed to identify functional groups, with measurements conducted on a Perkin Elmer FTIR spectrometer. The BET-specific surface area was evaluated from low-temperature nitrogen adsorption isotherms obtained using the Nova Station A instrument (Quantachrome Instruments, version 11.0, Miami, FL, USA). The pore diameter, reported in nanometers (nm), was determined by applying the Barrett–Joyner–Halenda (BJH) method with the same instrument.

3.0. Results and Discussion

3.1. X – ray Diffraction Spectroscopy (XRD)

The most veritable tool for determining the crystal structure of a material which include; the interplanar spacing, the crystallographic orientation, and the crystallite size is the X – ray Diffraction Spectroscopy [47]. From fig 1, the XRD diffraction pattern of the rGO/MnO₂ nanocomposite show that the characteristic peak of graphitic plane is restored at $2\theta \approx 23.26^\circ$ and the other peaks indicating the polymorphs of MnO₂ which agree with literature [48, 49]. As shown in Table 1, the diffraction peaks reveal a broad reflection at $2\theta = 23.26^\circ$, which corresponds to the (002) plane of reduced graphene oxide. This feature suggests that the material undergoes partial restoration of its graphitic structure, highlighting the reorganization of carbon layers during the reduction process [50, 51]. During the reduction of GO, oxygen-containing functional groups are removed, causing the peak to shift from a low angle of $\approx 9^\circ - 12^\circ$ (which is characteristic of GO) to the higher angle of $\approx 23^\circ - 26^\circ$ range. The peaks at 37.54° and 69.98° match β -MnO₂, a common polymorph with good electrochemical properties as referenced by standard JCPDS card No 24-0735 and the peak at 55.37° matches with γ -MnO₂, often present in nanocomposites due to its layered structure and high surface area which is referenced by standard JCPDS card No 44-0141. These results as summarized in *table 1*, were similar and in agreement with the experimental result of Yugambica and his colleagues, and also Saravanan and coworkers [52, 50]. Again, in *table 1*, there was a decrease in interplanar spacing of rGO prompting short ionic diffusion distance thereby causing increase in conductivity. This angular shift corresponds to a sharp decrease in the interlayer distance of GO (d – spacing). The spacing shrinks from

roughly ≈ 0.959 nm in GO down to ≈ 0.382 nm in rGO which is the reason for the observed broad peaks in the XRD diffractogram. It is evidence of nanoscale effects as observed by *Okorie & Buba 2023*, [53] and is responsible for expanding the interlayer distance, which is typical for graphene oxide and its partially reduced forms. The advantage is improved ion accessibility and electrolyte diffusion, which results in good electrochemical performance.

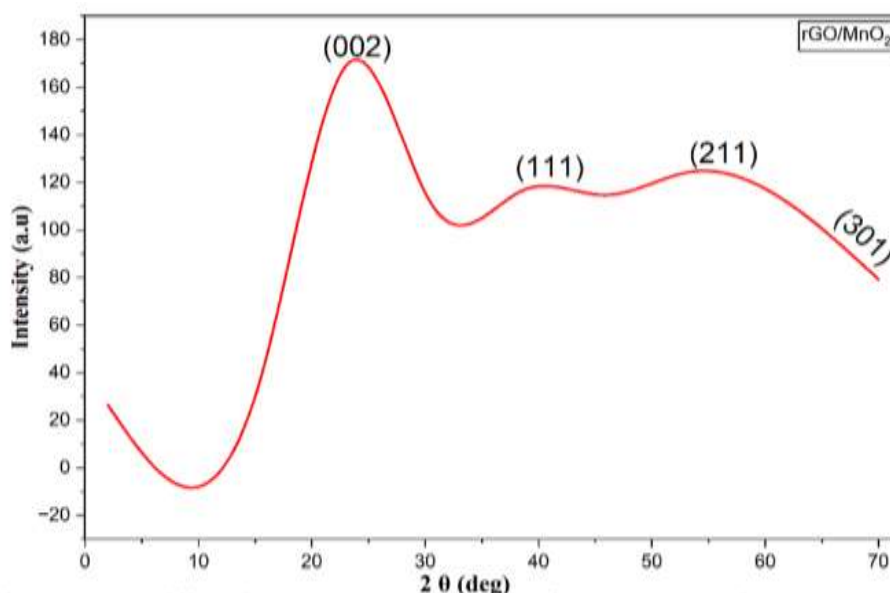


Figure 1. X-Ray diffraction patterns of rGO/MnO₂ Nanocomposite.

Table 1. The Interlayer Spacing and Miller Indices of rGO/MnO₂ Nanocomposite.

2θ (°)	θ (°)	FWHM (°)	d-spacing (nm)	Likely (hkl)
23.26	11.63	10.69061	0.382	(002) – Characteristic rGO
37.54	18.77	17.96896	0.239	(111) – β-MnO ₂ (JCPDS No. 24-0735)
55.37	27.685	20.04502	0.166	(211) – γ-MnO ₂ (JCPDS No. 44-0141)
69.98	34.99	16.55721	0.134	(301)/(220) β-MnO ₂ (JCPDS No. 24-0735)

Also, specific d-spacings match known MnO₂ polymorphs which are β-MnO₂ that is dense, tunnel-structured phase with good conductivity and γ-MnO₂ which is the layered phase with high surface area, useful for catalysis and energy storage. Smaller d-spacings of 0.134 nm at higher angles is an indication of tight atomic packing, typical of well-crystallized MnO₂ domains. With these properties there is composite synergy indicating that MnO₂ nanoparticles provides redox activity, while rGO nanoparticles enhance conductivity and mechanical stability. These structural findings are corroborated by recent works [54, 50, 52]. As discussed above, the d-spacing values

and peak assignments are consistent with those reported in previous studies, confirming the reliability of our analysis. Using the Williamson–Hall (W–H) method, the crystallite size was estimated to be 6.93 nm with an associated strain of 0.0362. These values fall within the range typically observed for reduced graphene oxide, thereby reinforcing our earlier assertion regarding the partial restoration of graphitic domains. The study is relevant for supercapacitor performance, and our material has shown that it has good structural property that is beneficial for use in supercapacitors.

3.2. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectrum provides a graphical representation of the sample's absorption of infrared radiation, with the absorption bands expressed in terms of wavenumbers (cm^{-1}). The wavenumbers (cm^{-1}) usually plotted on the x – axis, corresponds to energy levels whereas the absorbance or transmittance is on the y – axis and the peaks in the spectrum reflects specific molecular vibrations [55].

In order to interpret the FT-IR spectrum, four key wavenumber ranges are of great importance and they are [56]; Single-Bond Region ($4000\text{--}2500\text{ cm}^{-1}$) which are the stretching vibrations of O–H (hydroxyl) group, N–H (Amines) group and the C–H (mostly aromatics), Triple-Bond Region ($2500\text{--}2000\text{ cm}^{-1}$) absorption in this range correspond to triple bond stretching typical of $\text{C}\equiv\text{C}$ (alkynes) and $\text{C}\equiv\text{N}$ (nitriles) the third important region is the Double-Bond Region ($2000\text{--}1500\text{ cm}^{-1}$) the dominant group in this region are; $\text{C}=\text{O}$ (carbonyl) and $\text{C}=\text{C}$ (alkene and aromatic) which can also be found in the single – bond – region. The fourth region is Fingerprint Region ($1500\text{--}500\text{ cm}^{-1}$), and the dominant group is complex absorption patterns unique to individual compounds [57, 55, 58]. Broad peaks between 3650 cm^{-1} and 3250 cm^{-1} are typical of hydrogen bond and the functional group are common in hydroxyl ($-\text{OH}$) and amine ($-\text{NH}$) groups, where extensive intermolecular interactions lead to a wider peak [56]. From the above explanation, the functional groups are as shown in fig 2, the broad peak observed at 3161.98 cm^{-1} is an indication of a single bond which have the hydroxyl group, and the weakness of the peak is due to the reduction of the oxygen group and the result is typical of rGO/ MnO_2 nanocomposites [59, 52, 60].

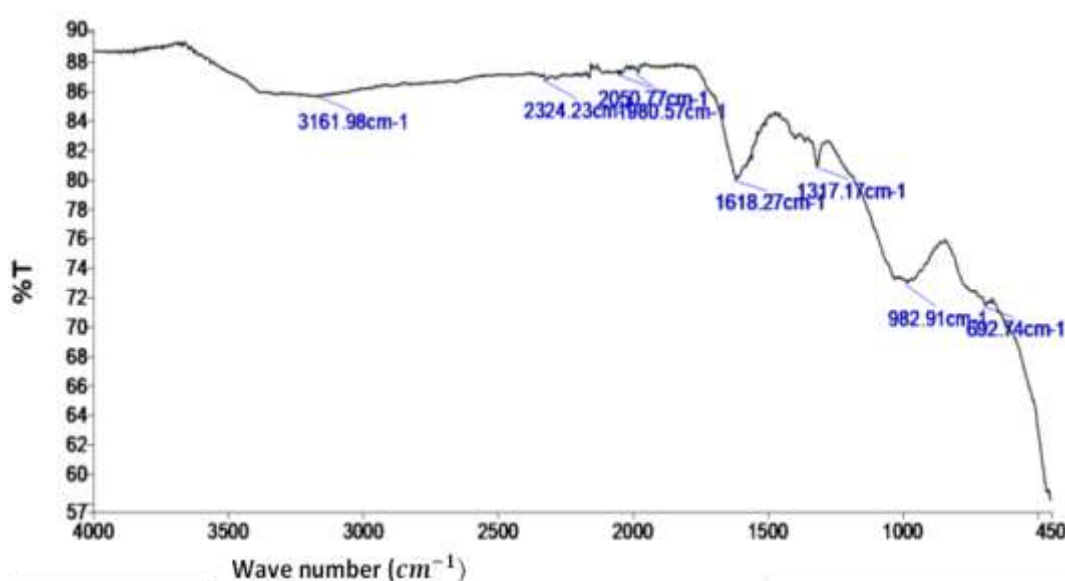


Figure 2. FTIR Spectrum of rGO/ MnO_2

3.3. Scanning Electron Microscope (SEM) Analysis

Scanning electron microscopy (SEM) determines surface morphology and the layer composition of graphene. It is achieved by focusing a beam of energetic electrons onto the sample and in our work, we used Nanos Sempior scanning electron microscope. SEM was used to check the morphologies of rGO/MnO_2 nanocomposite. The micrograph obtained from SEM at 750 magnifications of image on the surface of the material is shown in figure 3. The micrograph of the nanocomposite as observed, show good surface roughness. The implication of this roughness is an increase in effective surface area exposing more active sites that allow better interaction between electrode and electrolyte which will lead to higher current density and improved charge storage or catalytic activity. This property is essential for higher specific capacitance and energy density [61, 62]. Other benefits include enhanced ion transport because micro- and nano-scale roughness creates porous pathways that facilitate ion diffusion hence reduces resistance and improves rate capability, especially in fast-charging applications.

Rough surfaces can lower activation energy for redox reactions by increasing contact points which accelerates electron transfer and enhances electrocatalytic efficiency, crucial for sensors, fuel cells, and supercapacitors. Zhao et al [63], Albina et al [64] and Kunduraci et al [65] reported similar results.

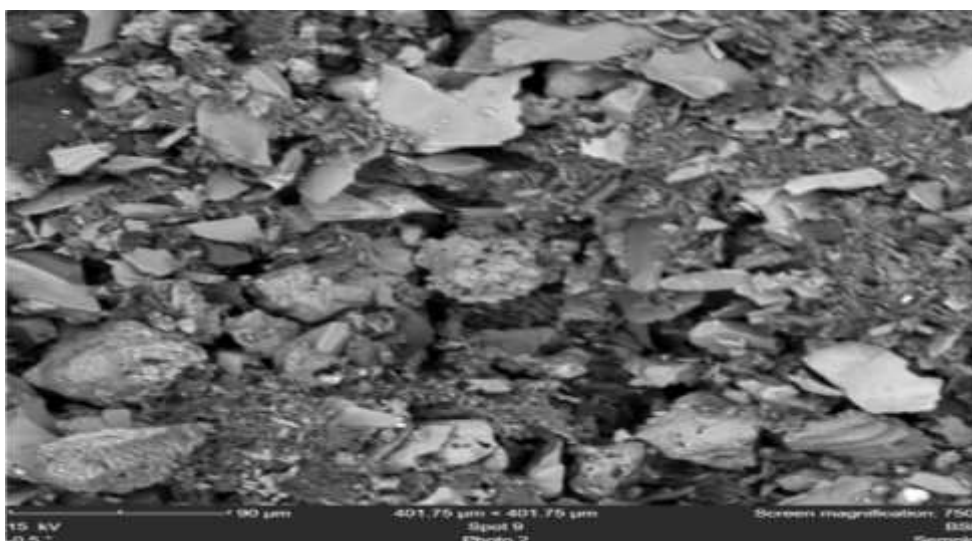


Figure 3. Micrograph of rGO/MnO_2 Showing the Surface Roughness.

3.4. Energy Dispersion X-ray Spectroscopy (EDX) Analysis

The elemental composition, quantitative analysis, purity and contamination checks are essential in material characterization. Given these considerations, it is essential to perform energy-dispersive X-ray spectroscopy (EDX) to verify the elemental composition and to determine the relative quantities present, as well as the purity level of our material [66, 13]. Figure 4 shows the material composition of the synthesized rGO/MnO_2 nanocomposite which confirm the dominant elements as shown in table 2. Most prominent element by percentage weight is Carbon consistent with rGO being the matrix of the composite, also strong Manganese (Mn) as expected for MnO_2 , reasonable Oxygen (O) signifying that Mn is bonded oxygen, the Silicon (Si) traces could be as a result of substrate wafer or glass, the traces of potassium (K) and Iron (Fe) can be residual salts and are negligible which can be ignored. The result is consistent with the fact that Oxygen content of GO is reduced in the composite

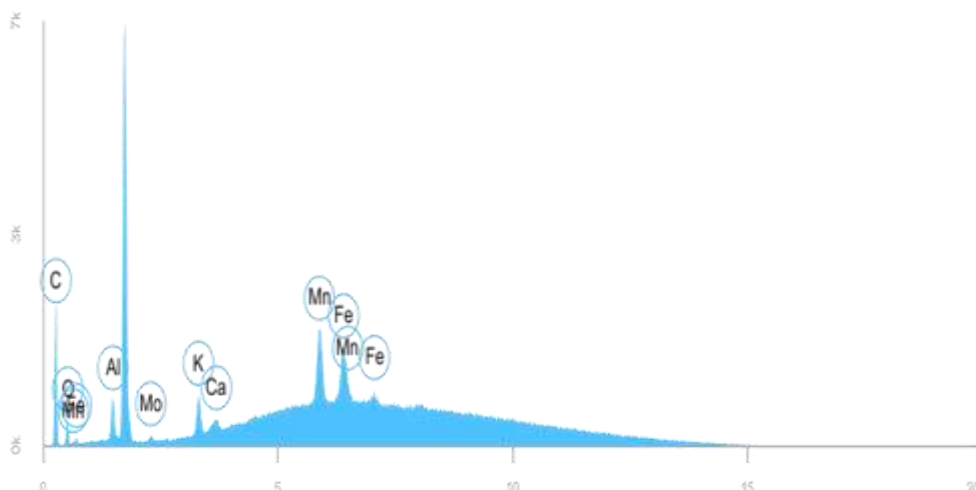


Figure 4. EDX Spectroscopy of rGO/MnO₂

Table 2. Percentage Elemental Composition of Synthesized rGO/MnO₂

Element	C	Mn	O	Si	K	Fe	Mo	Al	Ca
Weight %	43.0	29.3	17.4	8.9	0.9	0.5	0.0	0.0	0.0
Atom %	74.0	7.8	15.6	2.2	0.3	0.1	0.0	0.0	0.0

3.5. Surface Area Assessment

The specific Surface Area (SSA) and pore size is an important property of a supercapacitor electrode which facilitates the performance of supercapacitor electrode. For energy storage the pore size of electrode material is expected to be within a certain value hence materials are classified as microporous ($\leq 2nm$), mesoporous ($2nm \sim 50nm$) and macroporous material which is $50nm$ and above [67]. The standard method and most widely used procedure for surface area assessment is the BET analysis [68, 69]. A high surface area combined with well-defined porosity is critical for maximizing the capacitance of supercapacitors. These structural properties can be quantitatively assessed through Brunauer–Emmett–Teller (BET) analysis, which measures nitrogen adsorption and desorption to provide insight into the material's energy storage potential. Linearization of the BET equation [70] within the relative pressure range of $0.05 < P/P_0 < 0.35$ enables the transformation of experimental adsorption data into a straight-line plot, thereby facilitating accurate determination of surface area and porosity, from which the monolayer capacity (v_m) and BET constant (C) are extracted which was used to calculate the specific surface area of the material, making the BET method a cornerstone in surface science and materials characterization. The linearized form of BET equation is given in (5).

$$\frac{P}{V(P_0 - P)} = \frac{1}{V_m C} + \frac{C - 1}{V_m C} \cdot \frac{P}{P_0} \quad (5)$$

Where:

P = equilibrium pressure of adsorbate; P_0 = saturation pressure; V = volume of gas adsorbed; V_m = monolayer adsorbed gas volume; C = BET constant (related to adsorption energy).

From (5) it is easy to extract the monolayer, adsorbed gas volume and the BET constant through linear – regression which gives V_m and “C” as;

$$V_m = \frac{1}{\text{slope} + \text{intercept}}$$

And
$$C = 1 + \frac{\text{slope}}{\text{intercept}}$$

and hence, the specific surface area is determined which is given by (6).

$$S_{BET} = \frac{V_m N_A \sigma}{v} \quad (6)$$

Where;

V_m = monolayer capacity (volume of gas adsorbed at STP, in cm^3/g); N_A = Avogadro's number (6.022×10^{23}); σ = molecular cross-sectional area of adsorbate (in m^2); v = molar volume of gas at (STP) $22,414 (\text{cm}^3/\text{mol})$ [71, 70].

The specific surface area is expressed in units of area per unit mass (m^2/g) and can be converted to a volume-specific surface area by multiplying by the material density. In this study, nitrogen adsorption–desorption isotherms were recorded at 77.350 K, and the Brunauer–Emmett–Teller (BET) method was employed to determine the specific surface area. The BET equation was subsequently applied to the adsorption data to obtain reliable surface area values.

The BET linear plot Figure 5, was constructed by plotting $1/[V((\frac{P_0}{P}) - 1)]$ against $(\frac{P}{P_0})$. The linear region between 0.05–0.30 relative pressure was selected for fitting, yielding the result as presented in table 3. From these values, the monolayer capacity V_m was calculated; the BET surface area was also calculated. The BET constant (C) was also recorded, indicating strong adsorbate–adsorbent interactions. From literature, rGO/MnO_2 composites, the BET surface areas were between $200 - 500 \text{ m}^2 \text{ g}^{-1}$, highlighting the synergistic effect of MnO_2 nanoparticles in enhancing accessible surface sites [72, 50, 73, 74].

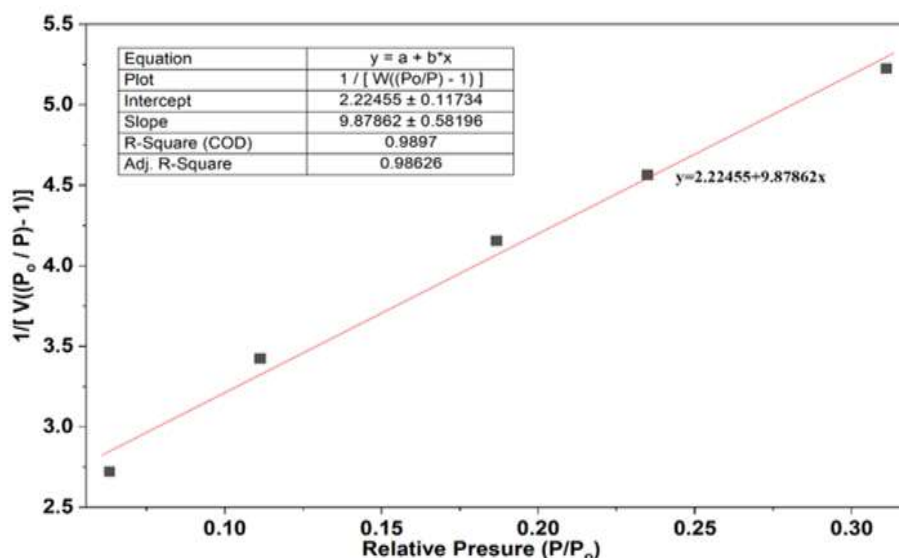


Figure 5. BET Linear Plot for Nitrogen Adsorption on rGO/MnO₂ Nanocomposite at 77.350 K.

Table 3. BET/BJH Analysis.

Sample	BET ($m^2 g^{-1}$)	area	BJH ($m^2 g^{-1}$)	area	BJH pore size (nm)	BET Constant	R^2
rGO/MnO ₂	359.64		346.6		2.115	5.441	0.99

The BET constant for our sample shown in table 3 suggest relatively uniform adsorption sites, while the high correlation coefficient also recorded in table 3 confirm the reliability of the BET fits. The high value of the surface area of the composite is particularly significant for applications in supercapacitors, where higher surface area directly translates to improved performance. Javanbakht et al [73] demonstrated Facile In-Situ Electrosynthesis of *MnO*₂–graphene Nanocomposite with surface areas in the range of 284.6 m²/g and it exhibited superior electrochemical performance compared to either component alone. The synergistic effect arises because graphene prevents *MnO*₂ particle agglomeration, while *MnO*₂ introduces additional porosity and redox-active sites. Our composite's BET result recorded in table 3 falls squarely within this optimal range, suggesting strong potential for high specific capacitance and excellent cycling stability.

3.6. Electrochemical Analysis

3.6.1. Cyclic Voltammetry (CV) Analysis

Cyclic voltammetry (CV) was employed to investigate the electrochemical behavior of rGO/MnO₂ using a BioLogic SP-200 potentiostat with a saturated calomel electrode (SCE) as reference. Figure 6 is the cyclic voltammogram of rGO/MnO₂ sample as recorded. The potential was cycled between –2.50 V and +2.50 V at a scan rate of 50 mV s^{–1} over 10 successive cycles. The voltammogram revealed an anodic peak at +1.19 V and a cathodic peak at –0.30 V, corresponding to a peak separation (ΔE_p) of ~1.49 V. This value of ΔE_p and asymmetry in peak currents is an indication that there was electron transfer process. The findings suggest that the sample undergoes redox behavior as expected due to the presence of *MnO*₂ in the matrix, which will promote pseudocapacitive property of the material.

From the CV analysis we can calculate the specific capacitance of the material as shown below.

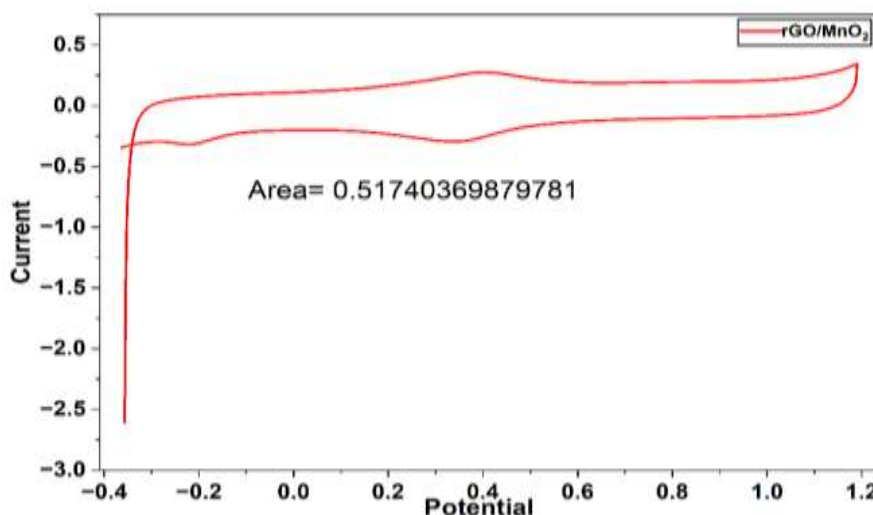


Figure 6. Cyclic Voltammogram of rGO/MnO₂ Nanocomposite.

$$C_{SP} = \frac{Q}{mv} \quad (7)$$

Where; Q is the charge, m is the mass and v is the voltage

$$Q = It$$

$$C_{SP} = \frac{It}{mv} = \frac{I}{m\left(\frac{v}{t}\right)}$$

$\frac{v}{t}$ is the scan rate from the instrument which we can represent as K .

$$C_{SP} = \frac{I}{mK}$$

$$\therefore I = C_{SP}mK \quad (8)$$

From integrating (8) over the surface area we got (9) which is the specific surface of the composite

$$C_{SP} = \frac{A}{2mK \Delta V} \quad (9)$$

Where A , is the integral charge surface area of the CV curve; K , is the scan rate and 2 is an indication of the forward and reverse sweeps [75, 76].

From (9) the specific capacitance of the electrode was calculated to be 347.25 F/g . This high value, is at a scan rate of 50 mV/s and it suggests strong pseudocapacitive contributions of MnO_2 due to the synergistic properties of the composite. This value for rGO/MnO_2 electrodes is fully consistent with published literature, which generally reports values between $300\text{--}700 \text{ F/g}$ for similar composites. This places our result in the credible range and shows our material performs better comparably to other optimized MnO_2/rGO systems with an advantage of synthesis procedure.

3.6.2. Electrochemical Impedance Spectroscopy (EIS) Analysis

Electrochemical impedance spectroscopy (EIS) was carried out using a BioLogic SP-200 potentiostat over the frequency range of 100 kHz to 0.1 Hz with an AC perturbation amplitude of 10 mV . Data were acquired with logarithmic spacing (10 points per decade) and subsequently plotted, as shown in Fig. 7. From the Nyquist plot of rGO/MnO_2 (Fig. 7), the Equivalent Series Resistance (ESR) and Charge Transfer Resistance (Rct) were directly extracted. The ESR corresponds to the high-frequency intercept of the Nyquist plot with the real axis ($\text{Re}(Z)$), yielding a value of 10.14Ω . The Rct is represented by the diameter of the semicircle along the real axis, obtained as the difference between the low-frequency and high-frequency intercepts, with a value of 66.2Ω . Notably, this Rct is exceptionally low compared to values reported in previously published studies. The implication of the above result is that the composite has good interfacial kinetics and lower internal resistance which will translate into better rate performance, higher power density, and more efficient charge/discharge cycles. A comparative look at our result and existing literature as shown in table 4, our result show better conductivity and faster charge transfer than many reported rGO/MnO_2 composites, which often exhibit ESR values in the range of $10\text{--}15 \Omega$ and Rct values in

the range of 80–120 Ω . This result suggests our electrode material is closer to optimized designs with improved kinetics and lower interfacial resistance. This is because Lower ESR minimizes energy loss, while lower Rct accelerates redox reactions.

Table 4. Comparative Study of ESR and Rct with our Work.

Study	Reported ESR (Ω)	Reported Rct (Ω)	Notes
rGO-anchored MnO ₂ nanorods	~12 Ω	~95 Ω	Good stability, but relatively high Rct due to poor conductivity [54]
MnO ₂ /rGO composites	~11–14 Ω	~80–110 Ω	rGO improved ESR, but Rct remained high without nanostructuring [52]
Green electrodeposited rGO/MnO ₂	~10 Ω	~70–90 Ω	In situ deposition, reduced Rct compared to conventional synthesis [44]
This work (In Situ reduction) rGO/MnO ₂ Nanocomposite	10.1 Ω	66.2 Ω	Lower ESR and Rct → improved conductivity and faster charge transfer

Using the result of specific capacitance, the energy density E_d measured in Watt-hours per kilogram (Wh/kg) and power density P_d measured in Watts per kilogram (W/kg) can also be evaluated using equation (10) and (11) respectively [77, 78].

$$E_d = \frac{1}{2} C_{sp} (\Delta V)^2 \quad (10)$$

$$P_d = \frac{V^2}{4 \times ESR \times m} \quad (11)$$

Where ESR is the equivalent series resistor

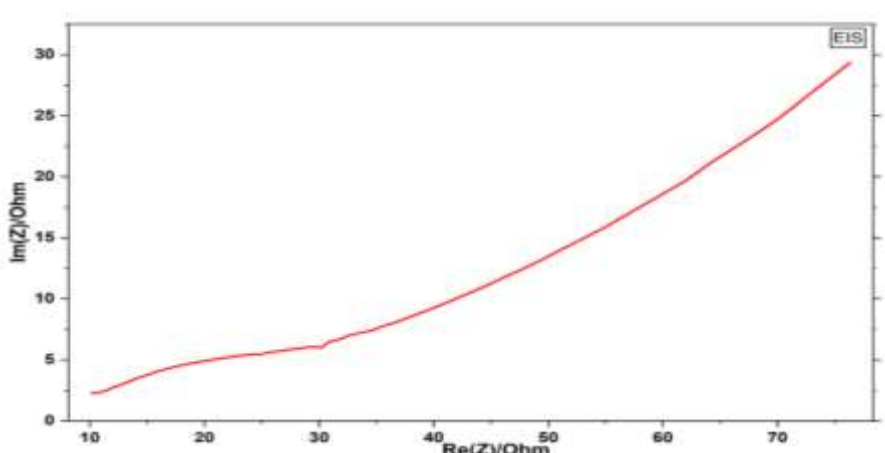


Figure 7. EIS Nyquist Plot of rGO/MnO₂

The value of the energy density of rGO/MnO₂ nanocomposite was found to be $\approx 107.1 \text{ WhKg}^{-1}$ which is near battery – level and power density of $\approx 55 \text{ KWKg}^{-1}$ which is extremely high. The implication of the result is that

the electrode material combines battery-level energy density with supercapacitor-level power density, which is an excellent balance and it is ideal for high-rate applications [44]. This suggests that the rGO/MnO₂ nanocomposite is highly promising for next-generation energy storage devices.

Also from the above results, the time constant of the supercapacitor, tau (τ) which is a measure of the speed of charging or discharging in a resistor – capacitor circuit is determined from (12).

$$\tau = R_{ESR} \times C \quad (12)$$

Where R is the electrochemical series resistance and C is the capacitance [79].

The time constant of the composite is 3,507.7s or about 58.5 minutes which is the time it takes for the supercapacitor to charge to 63.2% of its maximum voltage, or discharge to 36.8% of its initial voltage.

4.0. Conclusions

In conclusion, rGO/MnO₂ nanocomposite was successfully prepared using In-Situ reduction method with GO and MnSO₄ as our major precursors and L – Ascorbic acid as the reducing agent. The structural and morphological analysis were successfully determined which practically indicated its suitability for supercapacitor application. The result of the electrochemical characterization equally supported the assertion.

5.0. Recommendations for Future Research

In view of the findings of this research, the following recommendations are made:

- 1). Optimization of synthesis parameters: Investigate how variations in temperature, precursor concentration, and reaction time affect the morphology and electrochemical performance of rGO/MnO₂ nanocomposites.
- 2). Exploration of alternative metal oxides: Extend the one-pot synthesis approach to other transition metal oxides (e.g., NiO, Co₃O₄, Fe₂O₃) to compare performance and identify superior candidates for energy storage.
- 3). Hybrid nanocomposites: Study ternary or multi-component composites (e.g., rGO/MnO₂ combined with conducting polymers or other oxides) to enhance capacitance and stability.
- 4). Long-term cycling stability: Conduct extended charge–discharge cycling tests to evaluate durability and performance retention over thousands of cycles.
- 5). Device-level integration: Scale up the synthesis for prototype supercapacitor devices and assess real-world performance under practical operating conditions.
- 6). Environmental and economic assessment: Perform life-cycle analysis and cost–benefit studies to evaluate the sustainability and commercial viability of the one-pot synthesis method.

Declarations

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Competing Interests Statement

The authors declare that they have no competing interests related to this work.

Consent for publication

The authors declare that they consented to the publication of this study.

Authors' contributions

All the authors took part in literature review, analysis, and manuscript writing equally.

Availability of data and materials

Supplementary information is available from the authors upon reasonable request.

Institutional Review Board Statement

Not applicable for this study.

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