

rGonanostructure Electrode Material for High-Performance Super-Capacitor Application

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Abstract

Carbon materials are usually light, non-toxic, and environmentally friendly with excellent reinforcement ability. They are very promising materials for the development of future generation electronics and energy storage devices. rGO has attracted interest as electrode material for energy storage owing to its high surface area, stability, and porosity. A facile method to prepare reduced graphene oxide with enhanced performance has been synthesized by chemical reduction and exfoliation from graphene oxide and exfoliated to rGO. Their efficient chemical and electrical properties, such as high conductivity efficient electrochemical stability, enable them to fulfill the requirement of modern electronic industries. The morphology showed that rGO possessed a multiple-layered structure and is porous. The electrochemical properties of rGO have been investigated as supercapacitors. The electrochemical performance of rGO showed that a specific capacitance of 70F/g at 1 A/g, capacitance retention curve showed 69.4% retention after 1000 charge-discharge cycles at 1 A/g indicating rGO can be used in high-performance supercapacitor applications. The CV curves of rGO can keep rectangular even at a scan rate of 200 m/s; this can be due to the presence of a residual oxygen-containing functional group.

Keywords: Graphene-oxide, supercapacitor, and storage devices.

Introduction

The Graphene term was first coined by Boehm and co-workers and was derivative of graphite, the ene suffix denotes polycyclic hydrocarbons. Graphene, in turn, was derived from the Greek word "Graphiène" which means to write. Graphene which originated from graphite can be obtained physically from graphite as graphene is a single layer of graphite. Graphene when introduced as graphene oxide with the basal plane of the hydroxyl group and peroxide groups. But Graphene has gained worldwide attention in 2004 when an article was published in the science journal "Electric field effect in atomically thin carbon films."

This article focuses on the graphene synthesis, their modifications as graphene oxides and their potential use as electrochemical supercapacitors. Supercapacitors are materials that can store and release charge from high surface area conducting supports and are finding interest in the field of electronic applications and energy grid storage. Graphene materials with high electrical conductivity and increased surface area have exceptional mechanical properties and best for electronic applications. The reduced graphene oxide increases charge storage and transport in the electrical double layer at the electrode-electrolyte interface. Graphene and metal oxide increases energy density and capacitance with traditional supercapacitors.

Graphene is known as one of the best electronic materials with a high specific area making them highly desirable for use as a two-dimensional carbon and its related materials are attracting tremendous attention in the scientific community because of its exceptional properties and unique morphology. Its properties were enhanced when mixed with inorganic nanomaterials, thus offers many opportunities in the field of research including many applications such as for drug delivery through encapsulation, Photocatalysis, sensors, photovoltaic devices, and other energy storage systems. Graphene is superior to Carbon nanotube as its electrical conductivity and mechanical properties are better and have an extensive surface area. Reduced graphene is a kind of carbon that is processed by chemical, thermal and other methods to reduce the oxygen content and exfoliate to graphene oxide from graphite oxide with consequent chemical reduction. Graphite oxide was synthesized from natural graphite by the electrochemical exfoliation process in which deionized water used as a solvent. There are two routes production of graphene a bottom-up approach by chemical vapour deposition and a top-down approach starting from graphite to graphene sheets by exfoliation procedure. In this, we prepared graphene which was exfoliated as supercapacitor electrodes which have high specific capacitance. Exfoliated Graphene oxide as supercapacitor electrodes has high specific capacitance with good electrochemical and mechanical properties

and suitable for energy storage devices. An advanced supercapacitor can be prepared by developing an active electrode with oxygen functional groups, here we report to synthesize exfoliated Graphene oxide using reduction and investigated their performance.

Experimental section

Precursor details

Natural graphite flakes (99.9% purity, -250 mesh size), Sodium Nitrate (NaNO₃) (FINAR Reagents), Potassium Permanganate extra-pure (KMnO₄) (SRL), Sulfuric acid (98%) (H₂SO₄) (SDFCL), Hydrogen Peroxide (H₂O₂) 30% (EMPARTA), Hydrochloric acid (35.4%) (HCl) (SDFCL), Liquor ammonia [25% NH₃] (Fisher scientific), Hydrazine Hydrate (NH₂.NH₂.H₂O) (Qualgens), Multiwall carbon nanotubes (CVD-assisted synthesized CNT), Nitric acid (69-72%) (HNO₃) (SDFCL), and Potassium Hydroxide (KOH) (Fisher Scientific) were consumed for this research work.

Synthesis of Reduced Graphene Oxide (rGO)

The rGO was synthesized using the modified Hummers method with some changes in the pre-oxidation step as discussed below.

Pre-Oxidation process

2 g of natural graphite flakes and 1 g of NaNO₃ were dissolved in the concentrated H₂SO₄ and the mixture was stirred for 18 hours. A black thick solution was observed at this stage. Later the beaker was shifted into a container with ice cubes to maintain a temperature < 10 °C. While maintaining the temperature (<10 °C), 3 g of KMnO₄ was added slowly to the mixture under slow and steady stirring. Later, the ice-cube container was removed and the stirring was continued until the beaker was restored to room temperature (this process continued for 3 hours). The thin greenish colour slurry was observed at this stage.

After that, 92 ml of deionized (DI) water was added to the greenish slurry slowly for 30 min. under constant stirring and the temperature was maintained at 40 °C. Next, the temperature was set to 60 °C and stirred for another half an hour. Finally, the temperature was set at 90 °C and stirred for half an hour. During this stage, the mixture turned from greenish to thick brownish paste-like colour.

To the above mixture, a solution of 292 ml of DI water and 9 ml of 30% H₂O₂ was added to terminate the reaction. The light yellowish colour was observed indicating the formation of graphene oxide. The solution was put aside overnight. Later the mixture was treated with 10 wt% of HCl and then washed with DI water using vacuum filtration to obtain pH 7. Finally, the wet powder was dried overnight in a vacuum at 90 °C. The product thus obtained was termed as graphene oxide (GO).

Reduction of GO to rGO

300 mg of GO was dispersed in 300 ml of DI water and sonicated for 30 minutes to obtain a thick brown homogeneous suspension. A small amount of liquor ammonia was added to the above solution under stirring to obtain a value of pH 10. Next, 0.5 ml of hydrazine hydrate (N₂H₄) was added to the above suspension and heated at 80 °C for 2 hours under stirring. Finally, the solution was cooled and washed several times with DI water to obtain pH 7. The product thus obtained was labelled as reduced graphene oxide (rGO).

Characterization

The characterization techniques utilized in this work along with the sample preparation techniques are given below. The crystallographic detail of rGO was obtained from D8 Advance, Bruker Powder X-Ray Diffractometer with Cu K α radiation, the wavelength of 1.54056 Å and scan rate of 1.20/min (step size of 0.02 deg for 1 sec). Finely ground powder of the sample was used for this analysis. The Raman analysis was done using Witech alpha 300 with a 532 nm laser. The sample for the analysis was a fine powder. The morphology of rGO was obtained from ZEISS Gemini SEM 500 field emission scanning electron microscopy (FESEM). The sample for analyses was in the form of fine powder. TECNAI G2-Transmission electron microscopy (TEM) equipped with a field emission electron gun operated at 200 kV was used for microstructural studies. The finely ground powder was dissolved

in isopropyl alcohol (IPA) to get a uniform dispersion utilizing ultrasonication. 2-3 drops of the suspension were then dropped on a carbon-coated grid of 300 meshes and dried overnight for the analysis.

Finally, the electrochemical analysis of the materials was done using BioLogic Science Instruments. The detailed electrode preparation, electrochemical cell fabrication, electrochemical analysis input parameters utilized for the analysis and the analytical formulae used for the calculation of different parameters of supercapacitor are given below.

Electrode/Cell preparation

The active material, carbon black as a conductive agent, and polyvinylidene difluoride (PVDF) as a binder is taken in an 8:1:1 ratio. These were ground to a fine powder using mortar and pestle. A few drops of N-methyl 2-Pyrrolidone (NMP) were added to transform into a slurry. The slurry was later coated on a 350 μ thick graphite foil. The foil was dried in vacuum at 800 C overnight. The two-electrode cell was arranged using a Swagelok cell. The cell consists of two electrodes separated by a Whatman separator wetted with 3M KOH electrolyte.

Analysis of input parameters

Three techniques were utilized primarily for the electrochemical analysis namely, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and Potentiostat electrochemical impedance spectroscopy (PEIS). The CV curve was plotted between potential (x-axis) and current (y-axis) with an input ramp (potential window) of 1 V, at different scan rates of 5, 10, 20, 50, 100, 200, and 500 mV.s⁻¹. The GCD analysis was done at different current densities of 0.25, 0.5, 1, 2, 5, and 10 A.g⁻¹ using static input current, based on the weight of active material in an electrode. The GCD plot was made between time (sec) and potential (V). This same technique was used here for determining cyclic stability. PEIS was done with an input sine wave of V rms of amplitude 5 mV, frequency range from 0.1 Hz to 10 kHz, and initial voltage equals to an open-circuit voltage of the electrochemical cell just before this analysis. Nyquist plot (-Im(Z) vs Re(Z)) is plotted using PEIS.

Analytical formulae

The specific capacitance, energy density and power density of electrochemical cell are calculated from cyclic voltammetry using the following equations.

Specific capacitance:

$$C_s = \frac{\int Idv}{V_s \times \Delta V \times m} \text{ – Eq. 1 units: F. g}^{-1}$$

Where C_s is the specific capacitance, $\int Idv$ is the integral area under the CV curve, V_s is the scan rate, ΔV is the potential window, and m is the total mass of active material in the electrochemical cell.

Energy density:

$$E_d = \frac{1}{2} \times C_s \times \Delta V^2 \times \frac{1000}{3600} \text{ – Eq. 2 units: Wh. kg}^{-1}$$

where C_s is the specific capacitance obtained from Eq. 1, and ΔV is the potential window.

Power density:

$$P_d = \frac{1}{2} \times C_s \times \Delta V \times V_s \text{ – Eq. 3 units: W. kg}^{-1}$$

where C_s is the specific capacitance obtained from Eq. 1, ΔV is the potential window, and V_s is the scan rate.

Similarly, the specific capacitance, energy density, and power density are calculated from GCD as per the following equations given below [24].

Specific capacitance:

$$C_s = \frac{I \times \Delta t}{\Delta V \times m} \text{ – Eq. 4 units: F. g}^{-1}$$

$$C'_s = 4 \times C_s \text{ – Eq. 5 units: F. g}^{-1}$$

Where C_s is the specific capacitance of the full cell and C'_s is the specific capacitance of single electrode. The cathodic current applied, Δt is the discharging time, ΔV is the discharging voltage, and m is the mass of the total active material in the electrochemical cell.

The energy density is calculated with a similar equation as given in Eq. 2, except that C'_s is used obtained from Eq. 5.

Power density

$$P_d = \frac{E_d \times 3600}{\Delta t} \text{ -- Eq. 6 units: W. kg}^{-1}$$

Where E_d is the energy density obtained from the GCD analysis and Δt is the discharging time.

Results and discussion

Raman analysis

With modified Hummer's method, we synthesized Reduced Graphene Oxide.

In Fig 1, Raman spectroscopic analysis of rGO25, the D band indicates the extent of defects whereas G band indicates graphite nature if D peak intensity is higher the sample is having more defects in the graphite nature and if G band is prominent sample is said to be crystalline. Raman spectra of D 1339 cm^{-1} and G 1585 cm^{-1} respectively. The existence of D and G bands confirms the formation of graphite with lesser defects and crystalline structure. The decrease in the D band and increased G band shows that reduced GO has to increase graphitic domain size and also its thickness. A universal observation is that higher disorder in graphite leads to a broader G band as well as to a broad D band of higher relative intensity compared to that of a G band.

ID/IG ratios of 1:1 confirm the rGO layer.

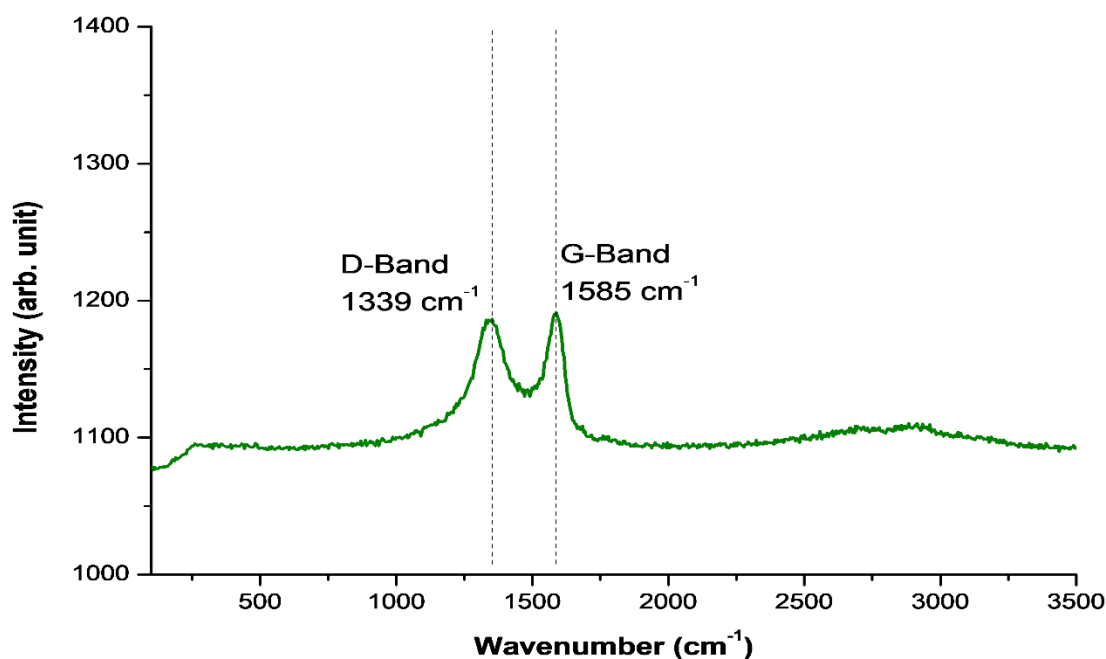


Fig. 1. Raman spectroscopic analysis of rGO

XRD Analysis

Figure 2 shows the XRD pattern of reduced graphene oxide exhibited a peak at $2\theta=25$ with an interlayer distance of 0.34 nm approximately with D spacing of 0.4nm implying a successful reduction of graphene to reduced graphene oxide during intercalation process. The result shows that after the chemical reduction process to remove carbonyl, hydroxyl functional groups on the surface the peak showed the characteristic of a reduced graphene oxide material.

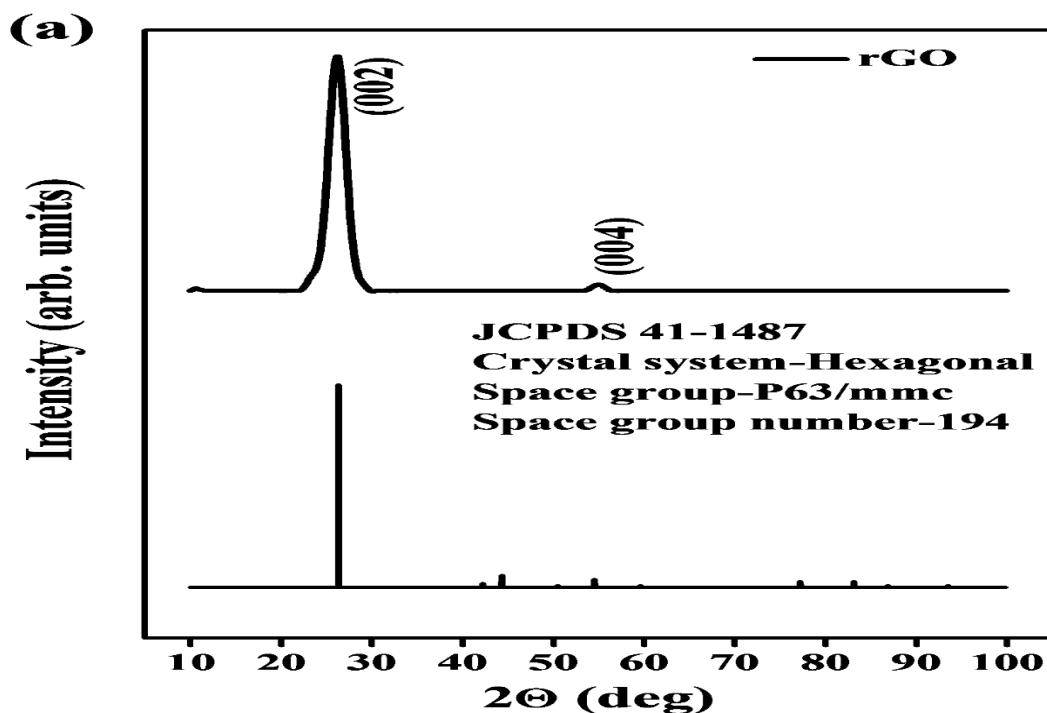


Fig.2. X-ray diffraction pattern of rGO

SEM Analysis

The SEM analysis shows go by modified Hummer's method, which can be obtained by various methods, the sample demonstrates ultrathin graphene films. The films of rGO appear as randomly aggregated with distinct edges wrinkled surfaces and folding, the main sheet dimension to about $10\mu\text{m}$. The result shows that rGO microparticles have similar morphology to that of GO microparticles. The oxygen content decreased and C/O increased.

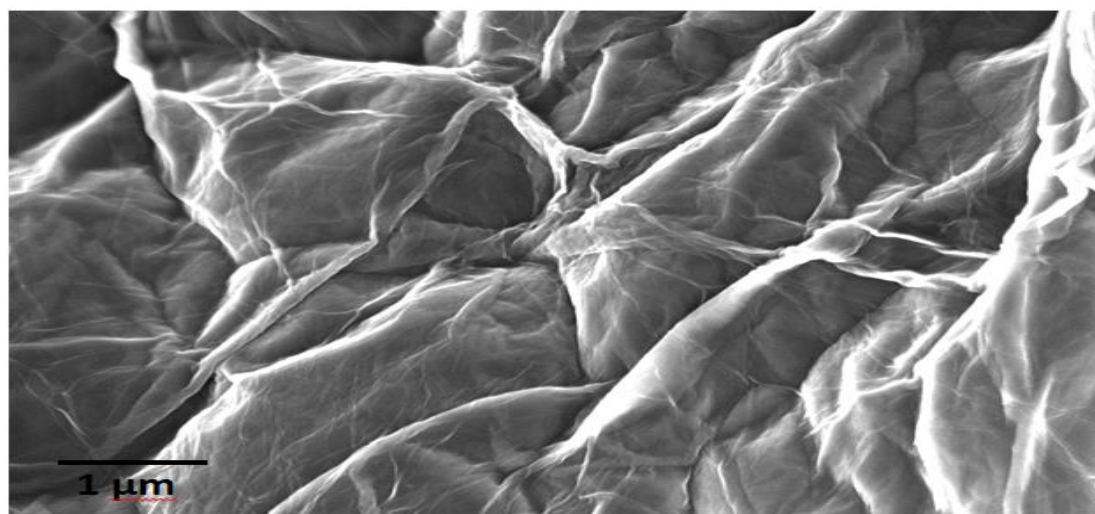


Fig. 3.FESEM image of rGO

TEM Analysis

The micrograph of the rGO sheet clearly shows the lattice fringes of graphene. The TEM image indicates different thickness, dark areas indicate the thick stacking structure of several rGO with the amount of oxygen functional groups and higher transparency areas indicate much thinner films of few layers of rGO resulting from graphene exfoliation. It also shows a significantly higher surface area of high transparency indicating lamination due to reduction.

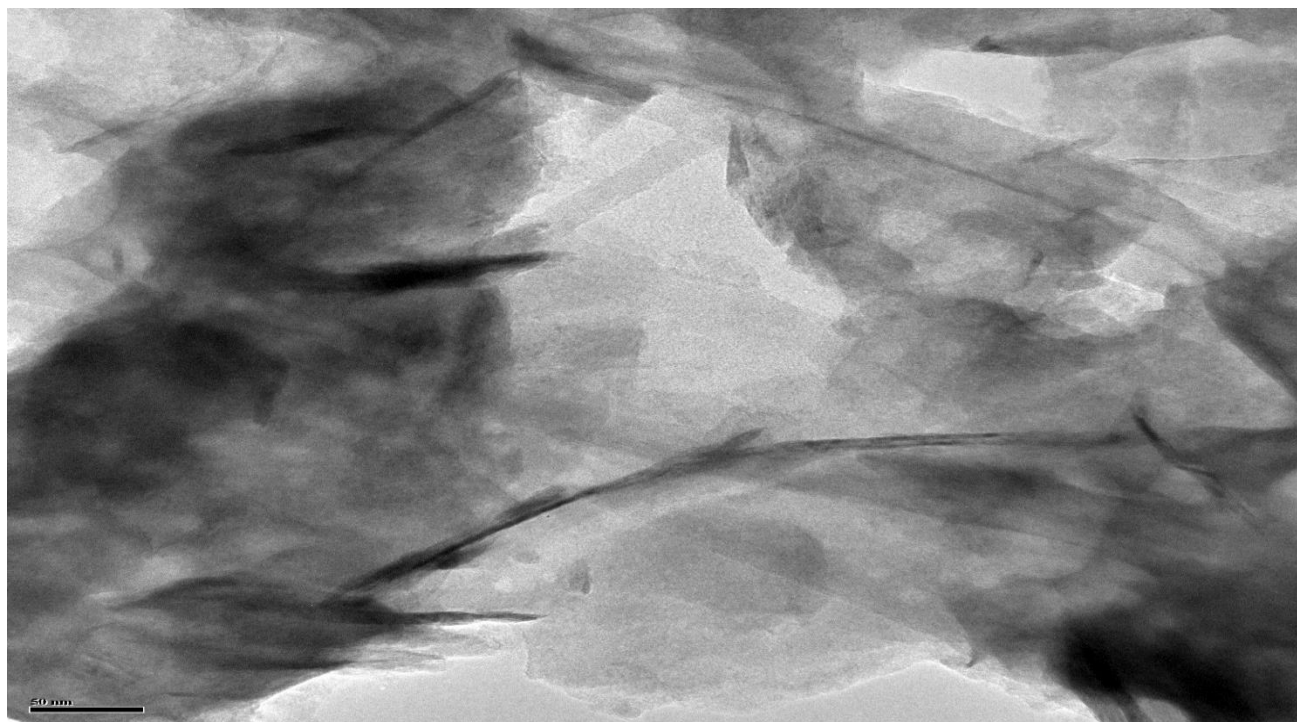


Fig. 4. TEM image of rGO

Electrochemical analysis

Three techniques were utilized primarily for the electrochemical analysis namely, cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) and potentiostatic electrochemical impedance spectroscopy (PEIS). The CV curve was plotted between potential (x-axis) and current (y-axis) with an input ramp (potential window) of 1 V, at different scan rates of 5, 10, 20, 50, 100, 200, and 500 mV.s⁻¹. The GCD analysis was done at different current densities of 0.25, 0.5, 1, 2, 5, and 10 A.g⁻¹ using static input current, based on the weight of active material in an electrode. The GCD plot was made between time (sec) and potential (V). This same technique was used here for determining cyclic stability. PEIS was done with an input sine wave of V_{rms} of amplitude 5 mV, frequency range from 0.1 Hz to 10 kHz, and initial voltage equals to an open-circuit voltage of the electrochemical cell just before this analysis. Nyquist plot (-Im(Z) vs. Re(Z)) is plotted using PEIS.

Electrochemical Performances

The CV curves and specific capacitance

The capability of rGO used as electrode material was tested through cyclic voltammetry. The figure 5(a) shows the CV curve of rGO measured at different scan rates of 5, 10, 20, 50, 100, 200 mv/s with an input ramp of 1V potential window shows a higher current response, the CV curve was plotted between potential on the x-axis and current on the y-axis. The integrated area of the curve of rGO shows better super capacitance behaviour, while the CV curve of rGO is approaching a rectangular shape, this can be attributed to EDLC behaviour contributed by GO. The role of rGO in enhancing the electrochemical has been revealed. This proves the ability of rGO used as an electrode material as supercapacitors 26-28

Effect of scan rate

To investigate the effect of a scan of rGO material at different scan rates of 5,10,20,50,100,200 and 500 mV/s on the x-axis and specific capacitance on the y-axis decreases as the scan rate increases, which shows that rGO material used as a supercapacitor.

Galvanostatic charge-discharge

One of the most efficient methods to evaluate durability and the performance of the electrode material is galvanostatic charge-discharge under controlled conditions. The rGO electrode at a current density of 1 A/g for 1000 cycles. The test results are presented in Figure 5 (c), the result shows that voltage and time relationship plots at different charge/discharge cycles are similar. The results show the relationship voltage and time relationship plot of at different charge /discharge summary deduce that a linear and nearly symmetrical triangular shape which is typical of an ideal capacitor. The GCD also demonstrates good durability, the stability of the material for use as a supercapacitor.

Electrochemical results of rGO

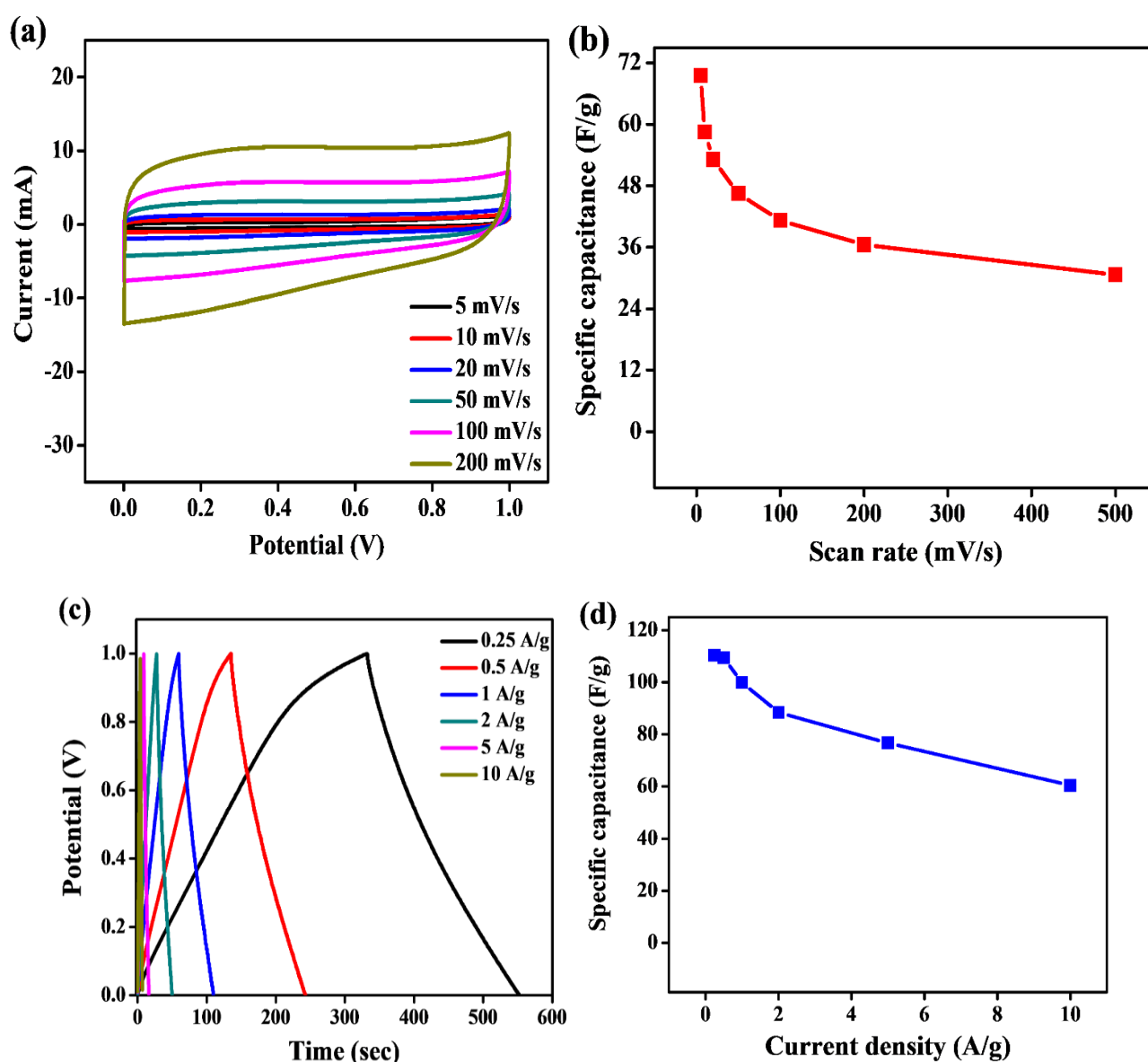


Fig. 5. Electrochemical analysis of rGO (a) cyclic voltammogram analysis at different scan rates (b) plot between specific capacitance and scan rate (c) galvanostatic charge-discharge analysis at different current densities (d) plot between specific and current density

PEIS measurement

The electrochemical performance, electrical conductivity, and transport charge of the electrode were further experimented by electrochemical impedance spectroscopy. The EIS is measured in the frequency range 0.1 Hz to 10 kHz, and the initial voltage equals an open-circuit voltage of the electrochemical cell just before this analysis. Nyquist plot ($-\text{Im}(Z)$ vs. $\text{Re}(Z)$) is plotted using PEIS. The result shows the impedance spectra are similar in form. The impedance increases with charge/discharge cycles. After 1000 cycles the electrode material still exhibits high conductivity or low internal resistance due to the removal of some oxygenated functional groups by electrochemical reduction.

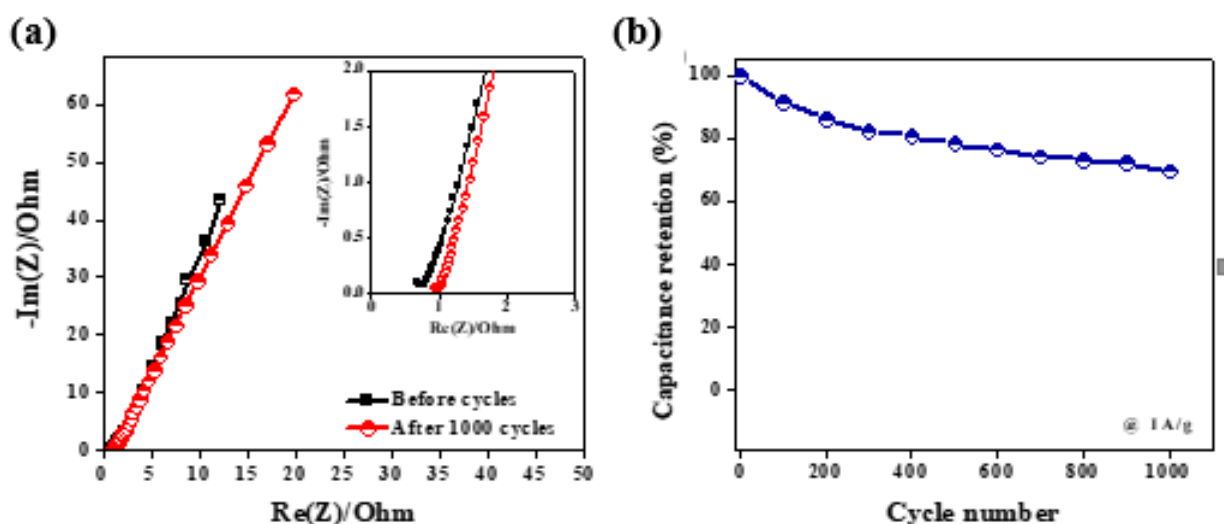


Fig. 6 (a) Nyquist plot before and after 1000 cycles with inset showing the plot at higher frequencies (b) capacitance retention curve showing retention of 69.4 % after 1000 charge-discharge cycles.

Conclusion

In the present study, we focused on the preparation of graphene from graphite flakes with the facile and affordable method. The properties of rGO were discussed based on Raman, XRD, SEM, TEM and also discussed its electrochemical properties for energy storage as supercapacitors, the results showed specific capacitance of rGO is better than graphene oxide as it exhibited high conductivity and electrochemical stability. rGO with its unique features and capacities is a future generation storage device.

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Conflict of Interest

Authors do not have any conflicts of interest with any person, institution, or agency.

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Authors Biography

Shikha Chander is an Assistant Professor of Chemistry at St. Francis College for Women with 22 years of teaching experience and authored materials for undergraduate levels and, currently am a research scholar under the supervision of Dr.Meenu Mangal, and my research is based on a synthesis of reduced graphene oxide electrode as supercapacitor applications and carbon for electrochemical energy storage and conversion.

Dr.Meenu Mangal is a Professor in Chemistry at Poddar College Jaipur, with experience in Teaching and published many papers in International Journals, She has authored and co-authored many books on Heterocyclic compounds, Water Sampling and her current area of research are on Nanotechnology and Asymmetric synthesis.