

Evaluation of Power Density: Lead Ion Incorporated into the Network Reduced Graphene Oxide ($\text{Pb}_x\text{rGO}_{1-x}$) Composite Electrode

M. Alpha^a, A.Z. Ngari^a, A. I. Dan Asabe^a, T.O. Daniel^b and Eli Danladi^c

^aDepartment of Physics, Nigerian Army University Biu, Borno, Nigeria. ^bDepartment of Physics, Alex Ekwueme-Federal University Ndufu-Alike, Ebonyi State, Nigeria. ^cDepartment of Physics, Federal University of Health Sciences, Otukpo, Benue State, Nigeria.

Corresponding author: alpha.matthew@naub.edu.ng

Abstract

Pb ion has most often been used in various compounds with carbonaceous materials in a variety of technological applications, such as in batteries and electronics devices. Pb doped reduced graphene oxide was studied using scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy, and X-ray diffraction (XRD) techniques. It was found that when the concentration of Pb ions rises, so does the average size of the crystallites. The XRD result revealed the development of atomic layers with preferred reflection plane (111), which leads to a thermal energy gain system of atomic restructuring, increasing crystal size, and reducing crystal defects in the network of the composite material. From the EIS analysis, there is a correlation between a rise in the Pb^+ doping concentration in the composite electrodes and a rise in their power density. The composites; rGO, $\text{Pb}_{0.4}\text{rGO}_{0.6}$ and $\text{Pb}_{0.6}\text{rGO}_{0.4}$ gave respectively the following power densities, 91.49 W/kg, 241.05 W/kg and 361.58 W/kg.

Keywords: Lead Ion, Reduced Graphene Oxide, Doping, Composite, Power Density.

Introduction

Due to their notable qualities, including normalization, anti-corrosive properties, acid/base doping/de-doping, being thin, as well as being relatively inexpensive for production of composites, reduced graphene oxide (rGO) nano-composites have attained a unique significance in both scientific research and academia. Depending on the degree of charge transport and the synthesis process, they display considerable physical changes (Nakazawa *et al.*, 2014; Jeyaprakash *et al.*, 2010; Lowsk *et al.*, 2008).

Pb ion has been employed most frequently in composites with activated carbon in a variety of technological applications, comprising sensors, electrodes for supercapacitors, and optoelectronics. One such material that has drawn a lot of attention since its discoveries is graphene, which has a variety of applications and incredibly unique features (Skoog *et al.*, 2016; Madhu *et al.*, 2014; Martinelli *et al.*, 2009).

Via morphological modification and electronic interaction between constituents, Pb and rGO are mixed in various dopant concentrations to enhance conducting electrode materials and produce distinctive electronic properties. Although there are various ways to create rGO composites, one conventional and popular method is the hydrothermal reduction

process. The composites used in this investigation were made using this technique. The chemical compositions of the composite, its morphology, and its structural characteristics were examined using energy dispersive x-ray spectroscopy, scanning electron microscopy (SEM), and X-ray diffraction techniques, respectively (Alpha *et al.*, 2019; Daniel *et al.*, 2019; Largeot *et al.*, 2008) can be used to get the typical crystallite size (D).

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

Where θ = diffraction angle, λ = wavelength of the X-rays (1.5406 Å) and β = full width at half maximum (FWHM). Dislocation density δ can be calculated from (Daniel *et al.*, 2020).

$$\delta = \frac{1}{D^2} \quad (2)$$

The micro-strain ϵ can be evaluated from (Ilcan *et al.*, 2008).

$$\epsilon = \frac{\beta}{4 \tan \theta} \quad (3)$$

Where D, β and θ retain their meanings as defined for Eq. (1). The absorption coefficient α (alpha) can be calculated from (Mukherjee and Mitra, 2015).

$$\alpha = 2.303 \frac{A}{t} \quad (4)$$

Methodology

Synthesis of Reduced Graphene Oxide (rGO)

5 g of graphite was measured and another 2.5 g of sodium nitrate were added together. 115 ml of concentrated sulfuric acid was added to the mixture and sonicated at 60 °C for thirty minutes. Once the mixture was placed in an ice bath, 15 g of potassium permanganate was gently added while the temperature was kept below 20 °C. Following the addition of the potassium permanganate, the temperature was then increased to 35 °C by sonication for 20 minutes. The mixture was gradually given 100 ml of distilled water and 1 g of ascorbic acid to aid in reduction. The temperature was then raised to 95 °C, and it was agitated for an additional 15 minutes. To help with homogeneity, 100 ml of distilled water and 10 ml of hydrogen peroxide were added to the mixture at the conclusion of the 15 minutes. Following filtering, the solution was rinsed with 100 ml of distilled water and 1 M of hydrochloric acid (8.3 ml of hydrochloric acid to 100 ml of water yields 1 M). When the residue was combined with polyvinyl chloride and bound with four drops of toluene, it was

spread out on slides and dried in an electric oven at 60 °C for eight hours to produce graphene oxide.

Synthesis of Lead Doped Reduced Graphene Oxide ($\text{Pb}_x\text{:rGO}_{1-x}$) ($0.4 \leq x \leq 0.6$)

For the synthesis of $\text{Pb}_x\text{:rGO}_{1-x}$ composite in the following compositions; $\text{Pb}_{0.6}\text{:rGO}_{0.4}$ and $\text{Pb}_{0.4}\text{:rGO}_{0.6}$, 100 ml of distilled water and Pb_2CO_3 , a source of Pb and rGO, were combined. Using an agate mortar and pestle, the rGO cake was thoroughly crushed. The mixture for each composition was then sonicated for 30 min. at 60 °C before being put into a stainless-steel autoclave for a hydrothermal reaction at 160 °C for six hours. With 50 ml of 0.1 M potassium hydroxide, the pH was brought to a neutral state (1.12 g of potassium hydroxide diluted with 200 ml distilled water). Using distilled water, the finished product was washed and collected. After placing the mixture on glass slides, it was combined with polyvinyl chloride and four drops of toluene. The mixture was then dried in an electric oven at 60 °C for two hours. A composite of $\text{Pb}_x\text{:rGO}_{1-x}$ ($0.4 \leq x \leq 0.6$) was produced after it had dried.

Results

The XRD

Figure 1 shows the XRD pattern for rGO, $\text{Pb}_{0.4}\text{:rGO}_{0.6}$ and $\text{Pb}_{0.6}\text{:rGO}_{0.4}$ composites scanned across a 2 theta angle of 20° to 80° range.

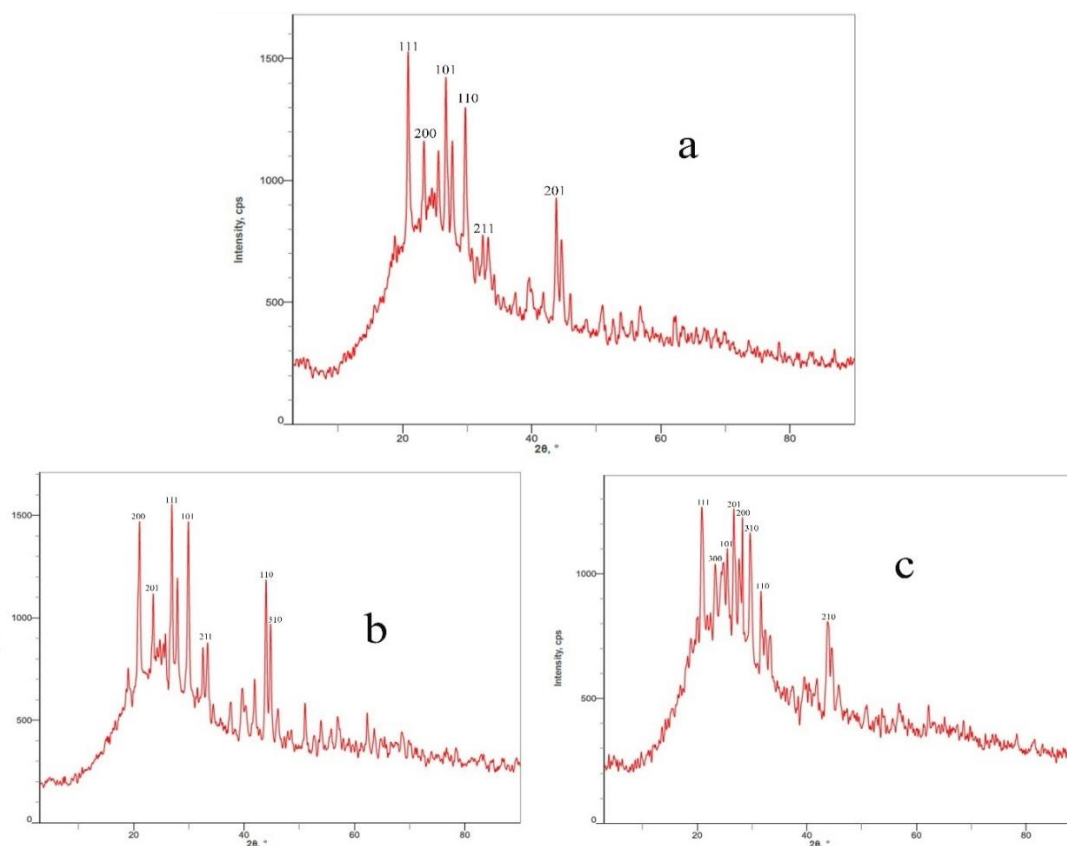


Figure 1: XRD Diffractogram for (a) rGO; (b) $\text{Pb}_{0.4}\text{:rGO}_{0.6}$; (c) $\text{Pb}_{0.6}\text{:rGO}_{0.4}$

The composites' peaks were examined and catalogued using the herzenbergite mineral's characteristic pattern (JCPDS PDF card 39-0355). The production of crystalline with few peaks is visible in the XRD pattern. For the equivalent composites rGO, $\text{Pb}_{0.4}\text{:rGO}_{0.6}$, and $\text{Pb}_{0.6}\text{:rGO}_{0.4}$, at 2 theta angles of 20.10, 24.00, and 22.00, respectively, the peaks with preferred orientation diffracted along the planes (111). The average size of the crystallites was found to increase as Pb ion concentration increased. This promoted the formation of atomic layers, which resulted in thermal energy gain system of atomic realignment that increased crystal size and decreased microstructures in the system of the composite material.

FTIR Results

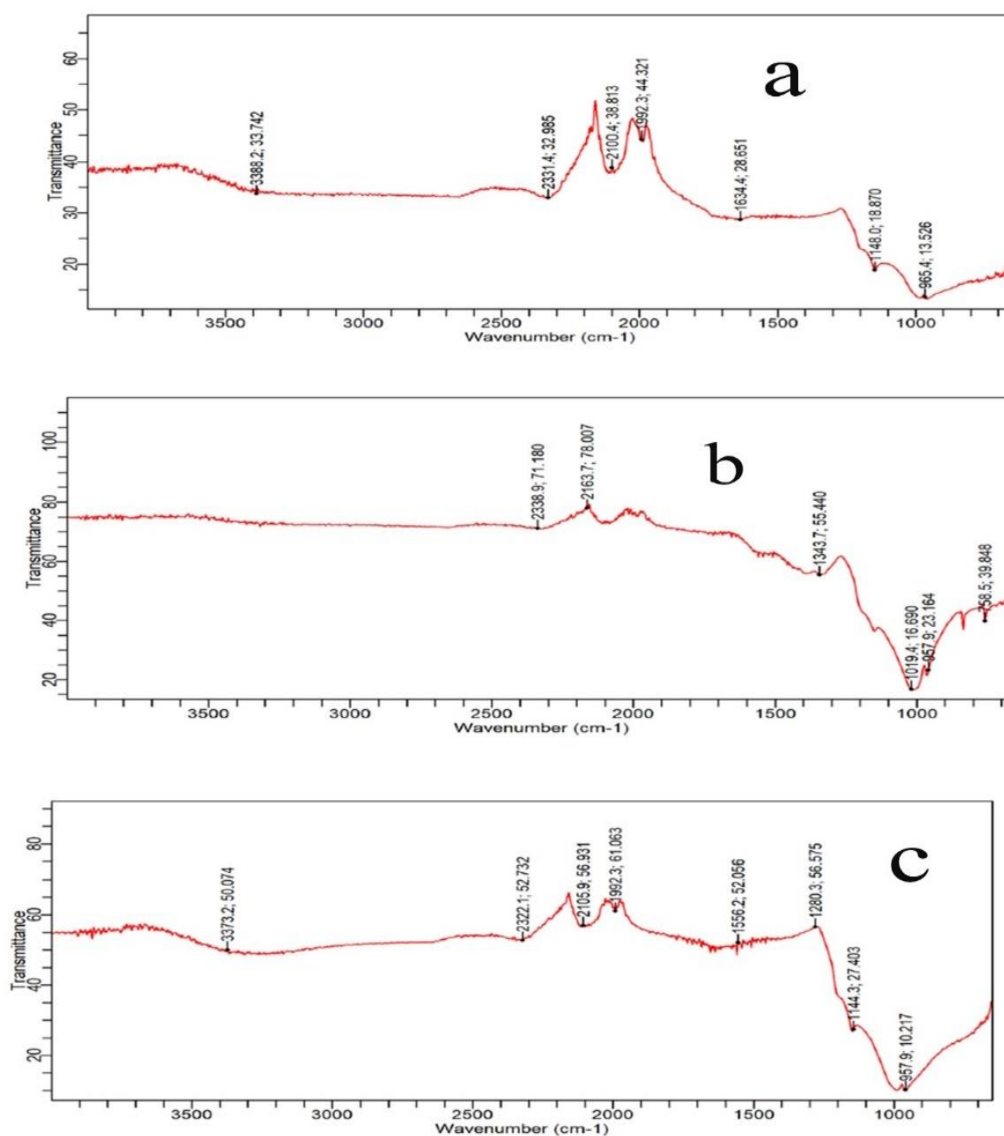


Figure 2: FTIR Spectra: (a) rGO; (b) $\text{Pb}_{0.4}\text{:rGO}_{0.6}$; (c) $\text{Pb}_{0.6}\text{:rGO}_{0.4}$

There are four distinct regions in the FTIR spectrum. The size of the first region is 4000 to 2500 cm^{-1} . The second zone is 2500 to 2000 cm^{-1} in size. The third region is 2000 to 1500 cm^{-1} in size. From 1500 to 400 cm^{-1} , the fourth region is measured (Raymundo-Pinero *et al.*, 2004). According to (Ilican *et al.*, 2008), the spectrum for these composites exhibits characteristic peaks between 2500 and 1280 cm^{-1} , which correspond to absorption brought on by C-O bond molecules in the composite. The spectrum also exhibits typical peaks between 1144 and 957 cm^{-1} that correspond to Pb-C bond molecules in the composite's absorption. The shift in peaks observed in the spectra can be attributed to a change in the hybridization state or electron distribution within the chemical bond of the composite material.

This confirms the findings of (Siamak *et al.*, 2015; Ilican *et al.*, 2008). The intensity of the peak influences the structural alterations and the total carrier concentration of the composite, which in turn influences the mobility of the surrounding Pb ions within the network of the rGO.

SEM Result

The SEM images of rGO, $\text{Pb}_{0.4}\text{:rGO}_{0.6}$, and $\text{Pb}_{0.6}\text{:rGO}_{0.4}$ are shown in Figures 3, 4 and 5. All materials used in this experiment had their morphology and microstructure examined using a Phenoworld Pro X Model scanning electron microscope (SEM) in secondary electron detection mode at 2 kV.

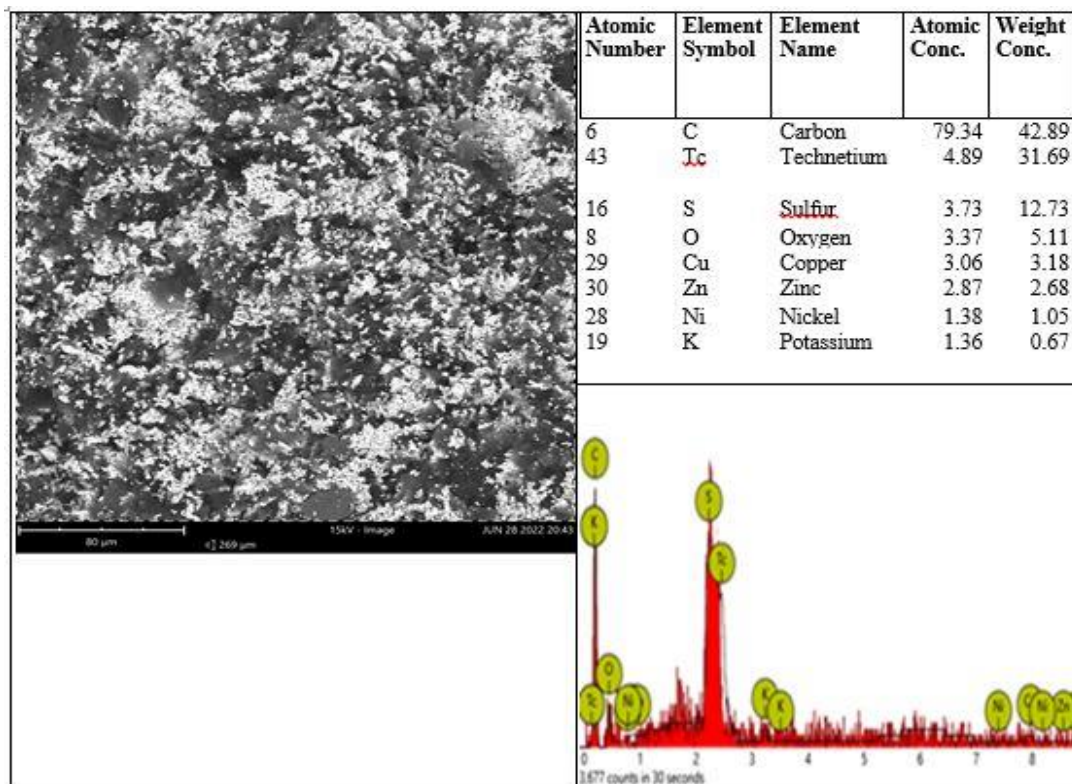


Figure 3: SEM Image and EDX for rGO

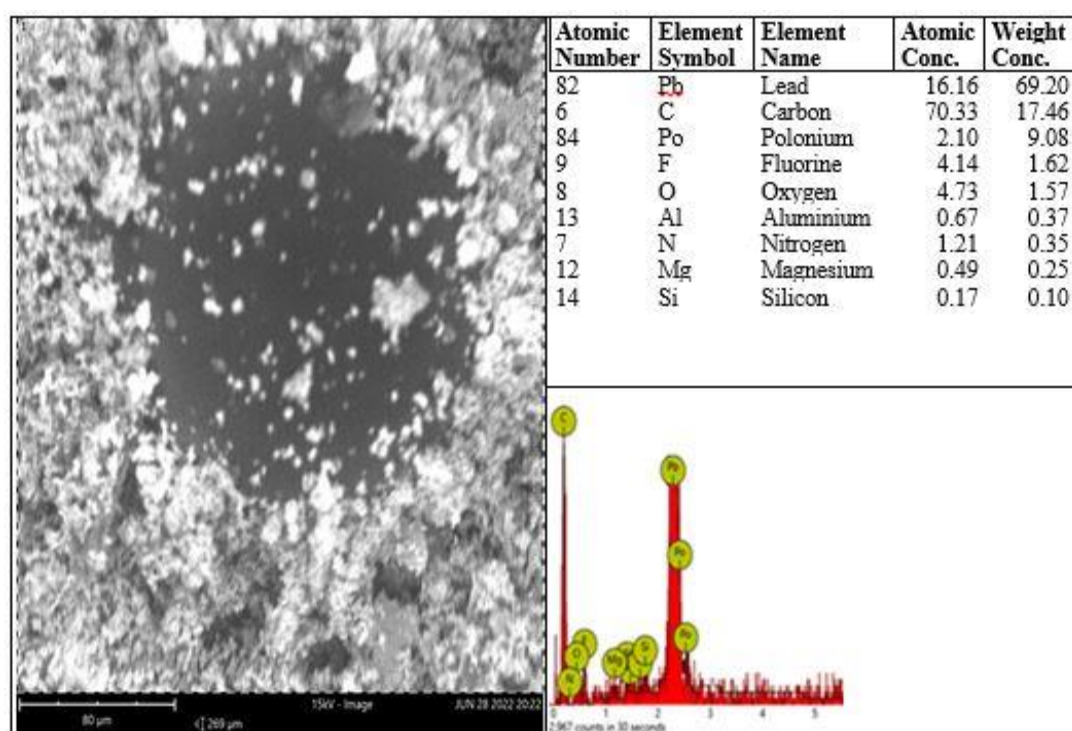


Figure 4: SEM Image and EDX for Pb_{0.6}:rGO_{0.4}

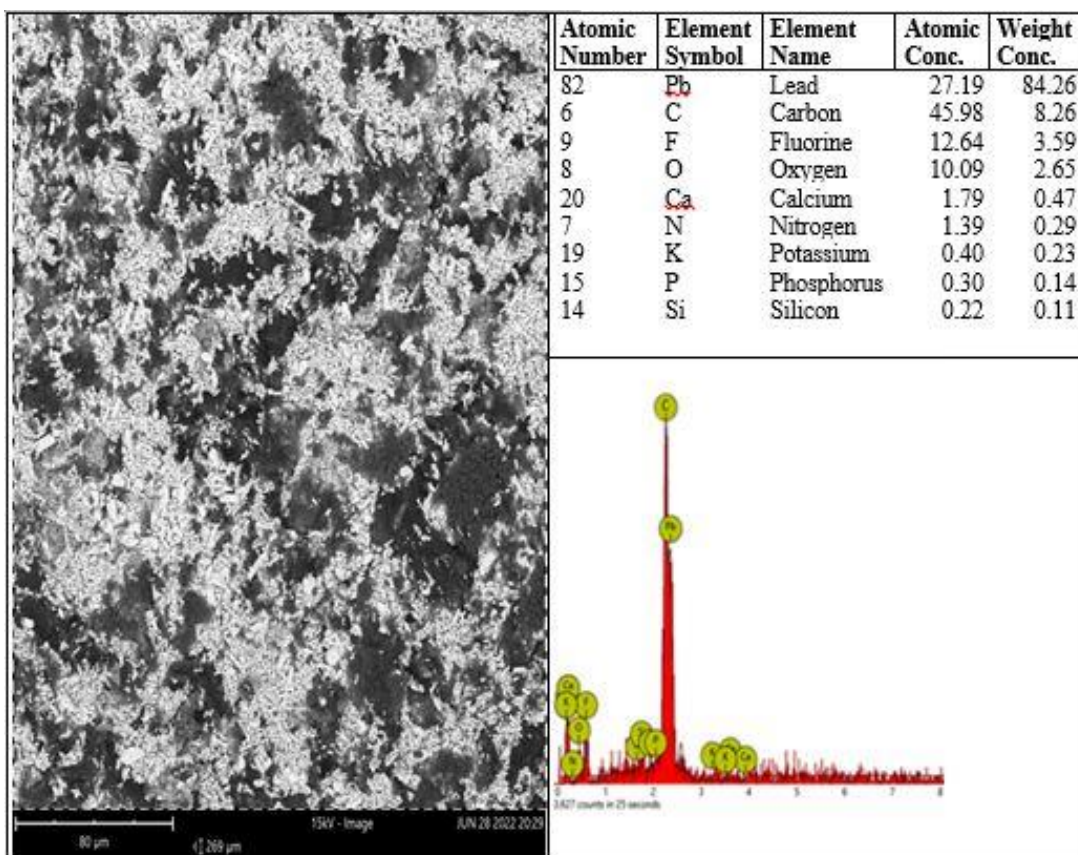


Figure 5: SEM Image and EDX for Pb_{0.6}:rGO_{0.4}

The doped Pb atoms are chemically connected between the layers of the reduced graphene oxide, as seen in the SEM images in figures 4 and 5, resulting in a composite material with excellent dispersion. The doped composites' porous surface patterns were remarkably consistent and were seen to enlarge with rising dopant concentrations. The SEM image demonstrates how, as the concentration of dopants rises, the surface coalescence gradually breaks down and the number of grain boundaries steadily rises. However, regardless of the level of doping, every doped composite displayed a strikingly similar microstructure and surface morphology because all samples were prepared under the same conditions. The interaction of reduced graphene oxide layers through van der Waals forces (Uwe, and Neil, 2011; Pandolfo *et al.*, 2006) creates a porous structure that can allow electrolyte ions to easily access the surfaces of the composite, this can improve the electrochemical utilization of Pb nanoparticles into the system of the reduced graphene oxide.

EIS Result

The EIS evaluation is a potent and insightful method that establishes the rate at which the electrode material can be charged or discharged, making it a crucial integral part in calculating the power density and assessing the conductivity and charge transport characteristics in the electrode/electrolyte interface (Low *et al.*, 2014)

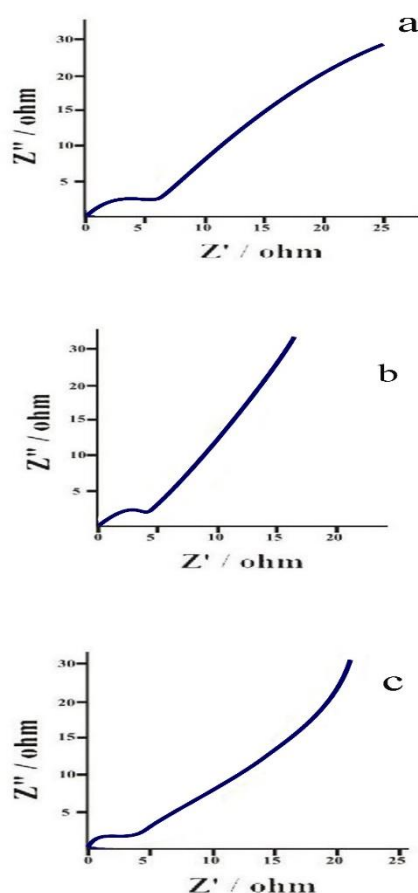


Figure 6: EIS graph: (a) rGO; (b) $\text{Pb}_{0.4}\text{rGO}_{0.6}$; (c) $\text{Pb}_{0.6}\text{rGO}_{0.4}$

Since the current collector, electrode, and dielectric material make up a capacitor in most cases, there will be an equivalent series resistance (ESR) from these parts. Power is the amount of energy consumed every second. As a result, the maximum power density that the cell can deliver is shown as follows:

$$P_D = \frac{V^2}{4(ESR)M} \quad (5)$$

(Yang *et al.*, 2013; Bello *et al.*, 2014)

Where P_D is the power density in Wkg^{-1} , M is the entire mass of active material, and ESR is the equivalent series resistance.

The two electrodes' combined mass and the entire cell capacitance are changed to the half-cell capacitance and single electrode mass using the factor $1/4$ (Yang *et al.*, 2013)

Equation 5 makes it evident that the capacitor's maximum power is dependent on the ESR, hence it is preferred to keep this number as low as possible for high power density. The kind of electrode material employed and the electrolyte's operating voltage have an impact on power density. The level at which the solvent in the electrolyte decomposes mostly determines the maximum operating voltage (in the case of aqueous electrolytes). Equations (5) show that the operating voltage affects the capacitor's power densities. Consequently, using highly capacitive materials with a wide potential window is crucial for achieving increased power densities that are higher than in batteries.

Table 1: Values of ESR for the composites

Composite	Mass (g)	ESR (Ω)	Operating Voltage (volt)	P_D (W/kg)
rGO	0.411	6.0	0.95	91.49
Pb _{0.4} :rGO _{0.6}	0.312	3.0	0.95	241.05
Pb _{0.6} :rGO _{0.4}	0.312	2.0	0.95	361.58

From table 1, there was a correlation between an increase in the doping concentration of cation (Pb^+) in the composite electrodes and an increase in the power density of the composite electrodes. This is because the introduction of Pb^+ into the reduced graphene oxide network resulted in an enlargement of the active sites, hence increases conductivity.

Conclusion

When the concentration of Pb ions rises, so does the average size of the crystallites. The XRD results showed the development of atomic layers with preferred reflection plane (111), which leads to a thermal energy gain network of atomic restructuring, increasing crystal size, and reducing crystal defects in the network of the composite material. From the EIS findings, there is a correlation between an increase in the Pb^+ doping concentration in the composite electrodes and a corresponding rise in their power density. This is because the

addition of Pb⁺ caused the active sites in the network of reduced graphene oxide to expand, thus increasing conductivity.

Acknowledgement

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Conflict of Interest

The authors declare that there are no conflicts of interest.

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