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Research Article

Development and Synthesis of Composite Electrode (rGO/G/PANI) for Capacitor from Burnout Battery Powder

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Abstract: TrGO/G/PANI is a new composite that has been derived by the combination of graphene oxide, graphite, and polyaniline for usage in supercapacitor applications. By combing these three, a material can be produced that would be able to reach capacitances up to 421F/g. In this project, the techniques for producing and characterizing the composite were shown. Graphene oxide was synthesized using Hummer's method and subsequently reduced to rGO using hydrazine hydrate. Polyaniline was synthesized by chemical oxidative polymerization using MnO_2 and graphite from burnout battery powder in presence of graphene oxide to form a ternary nanocomposite. The resultant material was characterized by SEM, XRD, TGA, and CV test. The presence of spherical nanoparticles of polyaniline and sheets or graphene was confirmed by SEM, and XRD confirmed the appropriate compounds. CV test showed the capacitance of the material, which was 75.3F/g. These results indicate that this type of material can be used for fast storage and quick utilization of energy.

Keywords: Polyaniline, Reduced graphene oxide, Supercapacitor, rGO/G/PANI composite.

1. INTRODUCTION

Global warming has compelled us to think of alternative routes to produce our energy; ways that are clean and do not harm the environment as much as the current setups do. The answer to this clean energy question lies in the effective utilization of renewable resources such as wind energy, solar energy, and hydroelectric energy, and their proper storage. Supercapacitors have become quite popular in the technological age of current times because of their attractive properties such as high cyclic durability, reduced cost, and high charge/discharge rates. The 3 types of supercapacitors are EDLC capacitors, pseudo-supercapacitors, and hybrid supercapacitors. EDLC supercapacitors have good durability and high cyclic stability. Pseudo-supercapacitors are generally lower performance supercapacitors with properties lagging behind those of EDLC supercapacitors. Hybrid supercapacitors are a combination of the 2 previous types of supercapacitors. They provide the high gravimetric and volumetric density and can provide large amounts of currents [1]. To achieve these criteria, Polyaniline is considered as a viable electrode material. This conductive polymer's low cost, simple synthesis method, and high energy density make it a good candidate for supercapacitor materials. The only downside to PANI is that it has poor cyclic stability. This, however, is remedied by the addition of carbon nanomaterials such as graphite nanoparticles which provide support to the polymer. Polyaniline is produced by a process that is known as chemical oxidative polymerization of aniline monomers. Here, components of used batteries aka burnout battery powder, which are MnO2 and graphite nanoparticles, are used. The MnO_2 oxidizes the aniline whereas the graphite particles improve the cyclic stability as mentioned before. This material on its own has a capacitance of about 317 F/g. Another material that has attracted attention is graphene because of

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its high surface area, great mechanical properties, and good conductivity. It is mixed with the G/PANI in the form of graphene oxide because the oxygen functional group allows it to be mixed in with polyaniline. The graphene oxide however can have improved capacitive properties in the form of reduced graphene oxide. This was reported by adding the mixture of GO/G/PANI with hydrazine hydrate which reduces the GO to rGO which is more conductive. This rGO/G/PANI material has capacitive properties that are greater than that of the Graphene oxide nanocomposite. Since this process used burnout battery powder as raw material, it can be thought of as a sort of recycling process as the SBP is being utilized rather than being discarded and causing pollution and harm to human health [2]. For the production of rGO, different processing techniques like Hummer's method, Hummer's modified method, and Improved Hummer's method were reported [3]. In the production of graphene oxide, graphite oxide is used as a precursor. Graphite oxide contains several different functional groups on the edges with the oxygen group attached to it. Graphite oxide has the following characteristics: it can be dispersed in water and a few organic solvents, it can have versatile properties depending on the functional group present and large amounts of graphene can be synthesized from the low-cost graphite by using chemical methods [4]. The GO possesses properties such as increased electron mobility (close to 200 000 $cm^2/v^{-1}s^{-1}$), high thermal conductivity (near 5000 $Wm^{-1}k^{-1}$) high theoretical surface area which is approximately 2 630 m^2g^{-1}. It has Young's modulus of 1100 Gpa which is almost 200 times greater than that of steel. This shows us that graphene is immensely strong [5]. The common misconception relating to graphene oxide is that it is an electrical insulator. It is not and this property directly depends upon the level of oxidation present in the compound. If the solution has highly oxidized graphene oxide, the overall electrical conductivity is reduced. If the oxygen functional groups are reduced, the electrical conductivity increases but not as high as the high-quality graphene monolayers. However, the conductivity along with other properties of the GO or rGO, such as dispersibility, hygroscopicity, and toxicity, can be modified by processes known as functionalization [6]. Polyaniline (PANI) has the form of powder material. PANI can have different colors depending upon the amount of oxidation. For example, Pernigraniline that is oxidized fully has a violet color whereas Leucomeraldine that is fully reduced has a pale-yellow color. This changing of color depending on the redox state allows PANI to be used in electrochromic applications. PANI is not very soluble in solvents as it forms very fine suspensions of colloidal particles. Methods such as grafting aniline monomers to other suitable functional groups have been suggested to overcome this non-soluble behavior. The powdery form however allows the PANI to be electrodeposited. This is done on electrodes to create PANI films [7]. Since PANI is in the form of a powder, its mechanical properties are usually determined by making PANI infused polymer composites or fibers that are electrospun on varying core materials. PANI pellets show good mechanical properties, around the range of 0.9 up to 1.3 GPA [7]. The overall mechanical strength of PANI is dependent on the compactness of the PANI powders. The higher the compactness, the better the mechanical properties could be achieved. PANI shows electrical conductivities ranging from 10-6 to 102 Scm^{-1}. Values within this range can be produced by doping. It is also dependent upon the type of doping, conditions of synthesis, and oxidant type. For example, doping with HCL produces conductivity of around 270 Scm^{-1} whereas doping with H_2SO_4 gives a conductivity of around 320 Scm-1 [7]. This reaction uses an electrochemical cell with an aqueous solution. This is used to remove the oxygen functional groups. The process of thermal annealing is used to reduce GO. Because of this extensive heating, the oxygen functional group decomposes into a gas. This gaseous expansion causes the total GO structure to expand and exfoliate. Reducing agents such as hydrazine hydrate, Lithium aluminum hydride, and sodium borohydride ($NaBH_4$) are used to reduce the GO to rGO [8]. The fabrication technique greatly influences the overall performance of the composite. Efficient strategies include attaching PANI on to graphene, producing nanostructured PANI to be attached to rGO or novel ideas such as nanofiber PANI combined with N-doped graphene hydrogels. Forming 3D rGO for 3D graphene / PANI nanocomposites proves to be a very costly method but produces a good capacitance of 596.1 F g^{-1}. Varying the component ratios also produces variable results. Using the methods of self-assembly, the compositions of PANI to graphene can be altered to produce varying results. For example, increasing the PANI percentage up to 50% produces a capacitance value of 921 F g^{-1}, and PANI percentages below 50% are okay for usage in flexible supercapacitors. It should be noted that the capacitance levels increase only up to 54% PANI. Increasing the percentage beyond 54% led to reduced capacitance values. By using a different electrolyte, or by adding a component, or by changing the way of compositing, we can achieve different synthesis conditions. For example, a graphene/PANI nanocomposite is made in which the graphene oxide is reduced by hydrazine hydrate was able to produce specific capacitances of around 480 Fg^{-1} [9].

rGO: When graphene oxide is reduced into just graphene, many new avenues of usage appear. Since reduced graphene oxide is conductive, it is used in high-speed electronics due to its virtually zero band gaps. Another use case of rGO is for supercapacitors. In its nanosized form, reduced graphene oxide can provide very large specific surface areas. This benefits the ability of charge retention and thereby improves the overall performance of the supercapacitor. These graphene-based supercapacitors are used in electric cars as well as in civil aviation aircraft, such as the Airbus A380. [13]. PANI: According to Waghuley at el [14], Polyaniline has received critical acclaim from researchers owing to its amazing optical and electrical properties, ability to remain stable in response to environmental effects, and its capability to handle doping elements. Here, the ability of Polyaniline to detect harmful gases such as NO_2, CO, SO_2 is used to make a gas sensor. The interaction of these gases with the surface of this active membrane produces different electronic changes in resistance, voltage, and conductance. Different gases cause these parameters to vary at different rates [15]. Like rGO, Polyaniline is also used in the making of supercapacitors. Because of PANI's low cost and fast charge/discharge rates, they are usually combined with different materials to produce capacitive properties. These properties have become apparent due to continued research on PANI composites and their behavior with different electrically active materials [16]. This work aimed to synthesize a nanocomposite electrode for the application of the capacitor by using rGO/G/PANI materials.

2. MATERIALS AND METHODS

a) Preparation of Graphene Oxide

Graphene Oxide was made using Hummer's method, here an ice-bath is used to keep the temperature of the reaction mixture low. An aliquot of 25 ml of H_2SO_4 is added to the beaker in which 1 gm graphite powder is added with 0.5 gm Sodium Nitrate ($NaNO_3$). The ratio for graphite powder and $NaNO_3$ is 2:1. Stirring is done by way of a magnetic stirrer with the temperature maintained below 20°C, using the aforementioned ice bath. Stirring is done for 30 mins to obtain a suspension. After a suspension is obtained, 3 gm of $KMnO_4$ is added to the suspension gradually with the continued stirring for 2 hours. $KMnO_4$ is used for oxidation of graphene oxide and $NaNO_3$ was used as a secondary oxidizer. Due to the addition of $KMnO_4$, and the reaction of $KmNO_4$ with $NaNO_3$, a green color due to MnO_2 was observed. After stirring for 2 hours, the ice bath was removed and the temperature is maintained at 35 - 40^0C with continued stirring. Stirring was continued for 1 ½ hour until a thick dark brown paste was obtained. Then, 46 ml distilled water was added, effervescence is observed due to temperature increased, light brown liquid is produced. Stirring was continued for 30 mins, the temperature is maintained at 85-90°C. Finally, 100 ml H_2O and 4 ml H_2O_2 are added to the solution to terminate the oxidation reaction, light brown color is observed. This is a positive indication for the synthesis of Graphene Oxide. Stirring is continued for further 15 mins for solution homogenization. Graphene oxide is washed, first with 1 Mol HCL, and distilled water afterward, using a centrifuge. The settled particles are then dried at 50°C for 5 hours using a drying oven.

b) Preparation of GO/G/PANI and rGO/G/PANI

For preparing the nanocomposite, burnout Battery Powder was extracted from local used batteries ("R14C" batteries used to extract the burnout battery powder). The burnout battery powder contained MnO_2 and graphite particles. This powder is then washed using a centrifuge and dried in a drying oven. The centrifuged and dried, burnout battery powder was mixed with 50 ml of water. This was then pre-cooled before mixing with Aniline and

GO. The ratio of MnO_2 and Aniline was kept at 1:1 so that no excess MnO_2 was present in the final solution. This ensured good electrical properties were achieved. The Aniline to GO mass ratio was kept at 1:10 and was added with HCL. Nonetheless, the pre-cooled burnout battery powder mixture was mixed into the Aniline/GO mixture to initiate chemically oxidative polymerization. This process was carried out at 0 ^{0}C for 8 hours which produced a colloidal solution of GO/G/PANI nanocomposites particles of bluish. Then, the solution was washed using 1 Mol HCL and deionized water several times using a centrifuge. The rotational speed was set to 7000 rpm and the time was set to 20 mins. The settled particles were collected in a conical flask. Finally, the particles were dried at 85^{o}C for 4½ hours and collected in a Petri dish. For the preparation of rGO/G/PANI, the synthesized dried and agglomerated form of GO/G/PANI was then reduced using the reducing agent Hydrazine Hydrate to rGO/G/PANI.

For reducing the sample of GO/G/PANI, the sample was first added with hydrazine hydrate to 70 ml of deionized water, then stirred for 2 h at 95-100^{o}C. Then the final synthesized solution was obtained, washed with deionized water, and dried in an oven at 40^{o}C for 12-14 hours.

c) Synthesis of an electrode for CV test

Chemicals used for the synthesis of the electrode were carbon black, DMF (N, N- Dimethyl Formamide), rGO/G/PANI composite in powdered form, and PVP (polyvinylpyrrolidone). rGO/G/PANI, Carbon black, and PVP were mixed in the ratio of 80:10:10. DMF was used as a dispersant because of its high solubility of inorganic compounds and its application in the production of high potential capacitors. It was then magnetically stirred for 2 hours. After the thoroughly mixing, the solution is poured into the small container made up of aluminum foil for the carbon plate to be used as an electrode. This was done to contain the liquid in a small space to ensure full coverage of the nanocomposite on all sides of the carbon plate. It was then dried in a drying oven at 40^{o}C for 8 hours.

Cyclic Voltammetry is an electrochemical analytical technique which is extensively used in chemical science for the calculation of current for electrochemical cell in an exorbitant condition of voltage. In this test, the current was determined by the passing of potential to the working electrodes in a cyclic manner. It used a three-electrode system namely, a counter electrode, working electrode, and reference electrode. The counter electrode or auxiliary electrode was for the balancing of the current of the working electrode or the stabilization of the potential between the reference electrode and the working electrode. The last functional electrode was the reference electrode with the constant potential which was used as a standard reference against the other electrodes in the electrochemical cell. The result for the CV test was obtained in the form of a voltammogram (graph between current and potential against the reference electrode) in which reduction and oxidation took place, so the potential would scan negatively first and then positively. In some cases, oxidation occurs first reversing the scanning of the potential. The TGA/DSC was used for thermal analysis of the sample in which the mass of the sample and heat flow were measured continuously concerning the temperature.

Table 1. Chemical used in the synthesis

S. No.	Raw material used	Company	Concentration (%)
1	Expandable Graphite	Daejung	Industrial grade
2	H_2SO_4	Labchem	98%
3	$KMnO_4$	Sigma Aldrich	99%
4	Deionized water	Daejung	Industrial grade
5	H_2O_2	Merck	30%
6	Aniline	Daejung	Industrial grade
7	Hydrazine hydrate	Daejung	Industrial grade
8	HCl	Merck	37%
9	DMF (N, N- Dimethyl Formamide)	Daejung	99.5%
10	$NaNO_3$	Labchem	Industrial grade
11	PVP (polyvinyl)	Labchem	Industrial grade

3. RESULTS AND DISCUSSIONS

3.1 EDX/Microstructure

EDX result shows the elemental mapping of the microstructure determining the % mass of Carbon and % mass of Oxygen. A high % of Oxygen confirms that GO/G/PANI is not fully reduced to rGO/G/PANI. There may also be a problem with the % mass of Oxygen because the error % for detection for Oxygen is high. Nitrogen in composite couldn't be detected as the vacuum chamber of SEM is vent with Nitrogen gas (Fig 2 , Table 2).

3.2 Scanning Electron Microscopy

The SEM images taken for the sample was made up of rGO/G/PANI with a ratio of 1:10 for GO/Aniline. It can be inferred from the SEM images showed in the Fig. 1 that the ternary nanocomposite has formed successfully based on the sheets seen in the images were flakes of rGO and upon these flakes, the G/PANI nanoparticles were observed. G/PANI nanoparticles appeared around the flake shape, as indicated the sizes in the nanoscale in Fig. 1 (c-d). There is significant agglomeration [17-18] among these particles because of the high GO/Aniline mass ratio of 1:10 and because the Aniline particles, when mixed with GO, get absorbed into the oxygen functional groups of the GO flake. These oxygen functional groups provide the nucleation sites for the formation of the rounded G/PANI particles.

3.3 XRD Results

XRD technique was used to confirm the composition of synthesized GO and rGO/G/PANI. The parameters for the performed test were; Start Position [2θ] =10.0125, end position [2θ] =79.9875, step size [2θ] = 0.0250, scan step time [s] = 0.6000, scan type= continuous and Cu was the anode target material.

Table 2. EDX data corresponding to the SEM image

Element	(keV)	mass%	Error%	At%
C K	0.277	65.03	1.01	71.24
O K	0.525	34.97	7.58	28.76
Total	-	100.00	-	100.00

GO: In the XRD result showed in fig. 26, the peak between 8-16 o shows the confirmation of the presence of GO in the sample [3].

RGO/G/PANI: The XRD result showed in fig. 4 confirms the presence of the PANI with a peak between 20-30^{o}, confirming that the MnO_2 particles are consumed in the oxidative polymerization of Aniline

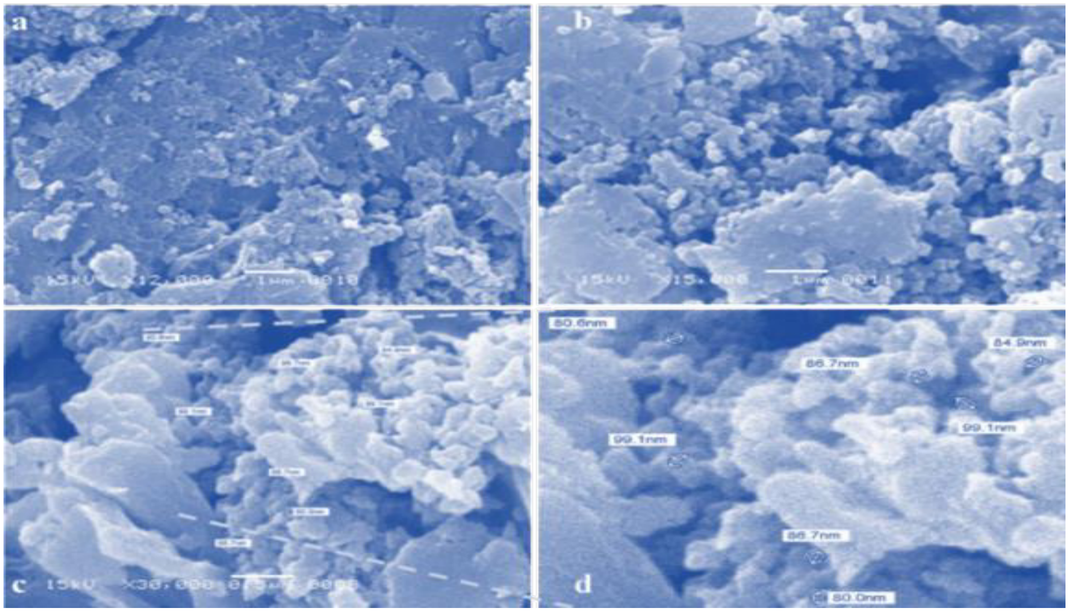

Fig 1. SEM images of rGO/G/PANI sample at (a) 12,000X, (b) 15,000X, (c) 30,000X and (d) zoom inset of (c)

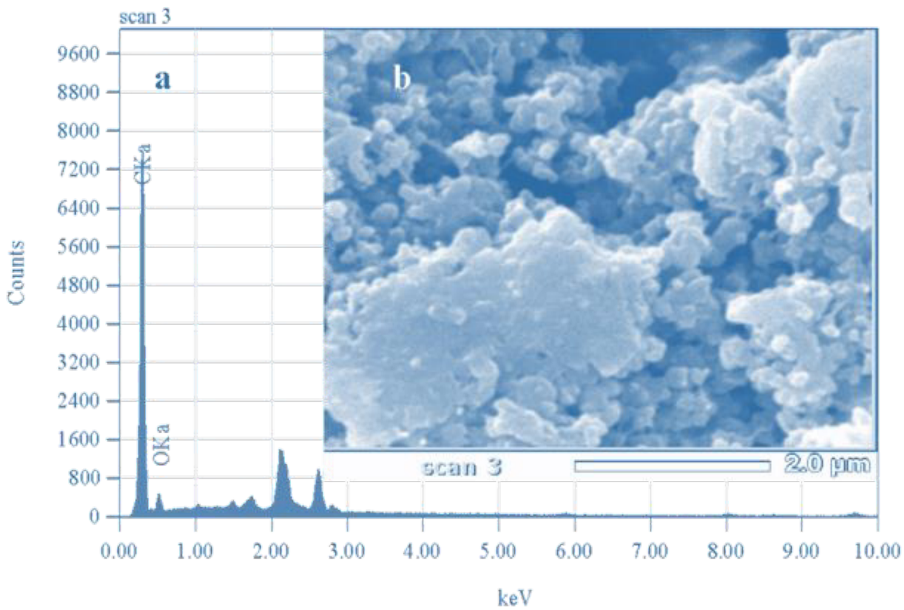

Fig 2. Shows (a) corresponding EDX data of (b) SEM image

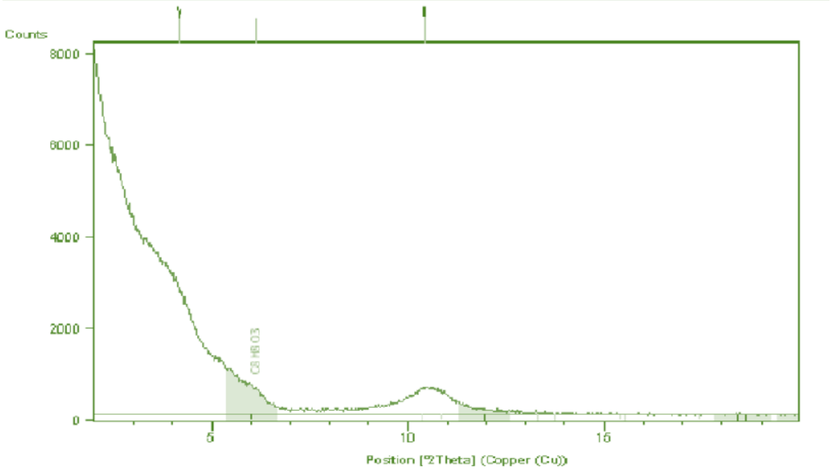

Fig 3. XRD Pattern of GO

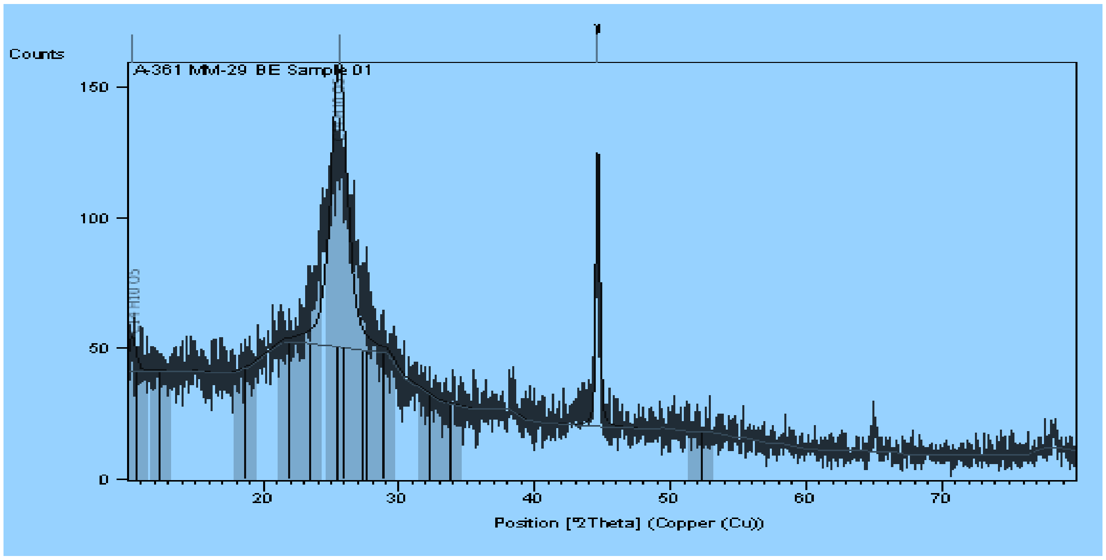

Fig 4. XRD pattern of rGO/G/PANI

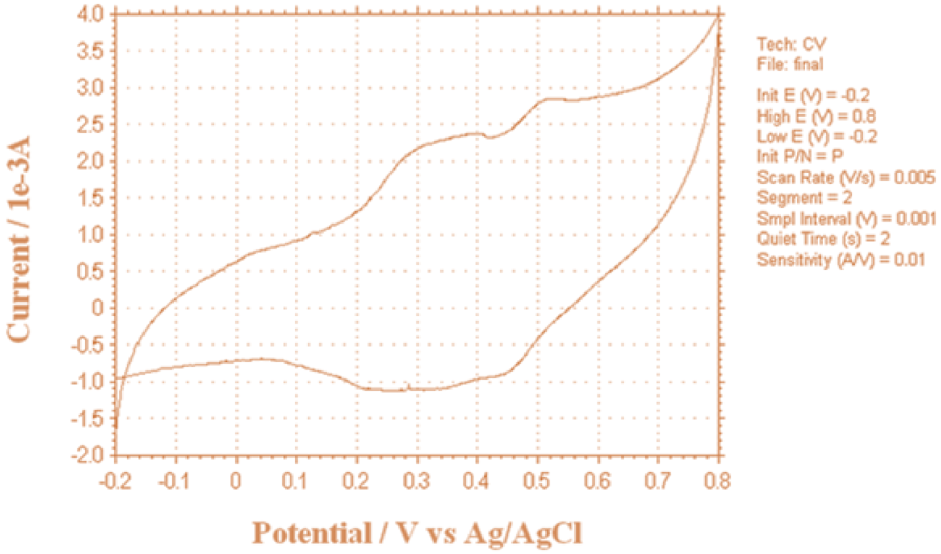

Fig 5. Voltammogram of rGO/G/PANI

3.4. CV (Cyclic Voltammetry)

The CV test was carried out for the Nano-composite electrode. A scan rate of 0.005 V/s was used in 1 M H_2SO_4 solution. The reference electrode, counter electrode, and working electrode used for this test were made of Ag, Pt. wire, and carbon electrode coated with rGO/G/PANI respectively.

The specific capacitance was calculated for this composite according to the following relationship [2] as per the data shown in Fig. 5;

$$Cm = \frac{\int_{-0.2}^{0.8} I\,dv}{m(s)(\Delta v)}$$

Where; $Cm =$ Specific capacitance, $\int_{-0.2}^{0.8} I\,dv =$ Area under the positive sweep, $m =$ Mass of active material (rGO/G/PANI), $s =$ Scan rate, $\Delta v =$ Potential window.

Therefore, we have:

$\int_{-0.2}^{0.8} I\,dv = 1.883 \times 10^{-3}$, $m = 0.005$, $s = 0.005$,

$\Delta v = 0.8-(-0.2) = 1$

4. CONCLUSION

The Ternary nanocomposite GO/G/PANI for supercapacitor was successfully synthesized through chemical oxidative polymerization of aniline using the oxidizing agent MnO_2, extracted from burnout battery powder, with the addition of Graphene Oxide flakes. In Addition to this, this composite was reduced by using hydrazine hydrate, where GO/aniline was taken in 1:10, showed a good specific capacitance, and finally, it was done to find out the specific capacitance, which was 75.3 F/g. TGA/DSC results showed the thermal stability phenomenon of the ternary nanocomposite electrode material at low temperatures. [3, 17, 18, 19 and 20]. This kind of composite material would also be a good initiative to overcome the energy storage demands for future technology. rGO/G/PANI. XRD result confirmed the synthesis of the ternary nanocomposite rGO/G/PANI. CV test was done to find out the specific capacitance, which was 75.3 F/g. This kind of composite material would also be a good initiative to overcome the energy storage demands for future technology.

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