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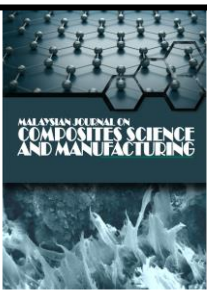
ISSN: 2716-6945



Cyclic Voltammetry and Galvanostatic Charge-Discharge Analyses of Polyaniline/Graphene Oxide Nanocomposite based Supercapacitor

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ARTICLE INFO

Article history:
Received 27 July 2020
Received in revised form 07 September 2020
Accepted 15 September 2020
Available online 30 September 2020

ABSTRACT

Supercapacitor is an energy device that is applicable in numerous fields because of its excellent reversibility, long life and high power density. Nevertheless, its universal use is restricted by the reduced energy storage capacity and its high crossed series compared to batteries. Even with the relatively high-level output and extensive use of supercapacitor, there is still substantial doubt and ambiguity as to their efficiency in general, especially when it is compared to lithium-ion batteries. The inconsistencies are attributable both to the lack of standardization of the test methods and to the certainty of the strength capacity of the supercapacitor after their resistance has been identified. Therefore, in this work, graphene oxide (GO) and polyaniline (PANI) nanocomposite supercapacitor electrode was fabricated and the performance was investigated by means of cyclic voltammetry and galvanostatic charge-discharge analyses. GO was synthesized using improve Hummers method and PANI using oxidative polymerization chemical synthesis. Three different electrode's compositions were prepared using PANI/GO nanocomposite and labelled as PGO30, PGO50 and PGO70. This article will conclude the electrochemical performance of the electrode. From the results, it was found that PGO50 electrode (50% PANI/50% GO) has the best calculated capacitance with 19.71 F/g compared to the other composite electrodes. This may be attributed from the good electrical conductivity distribution of PANI and graphene oxide. The findings of the work may significantly drive the future of supercapacitor electrode from nanocomposite related materials.

Keywords:
PANI/GO nanocomposite; supercapacitor electrode; cyclic voltammetry; charge-discharge

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https://doi.org/10.37934/mjcsm.3.1.1426

1. Introduction

Supercapacitors are attractive in delivering better power efficiency than traditional capacitors and batteries due to their long cycles, protection, good stability, and environmental friendliness [1]. It also has great features, such as being capable of storing 10 - 100 times more energy per unit volume than electrolyte condensers, being able to tolerate and handle charging much quicker than batteries, and tolerating much more charging and discharge cycles than rechargeable batteries. There are two major types of supercapacitors; electric double layer capacitor (EDLC) and pseudocapacitor. EDLC retains electrical energy through the electrostatic adsorption and desorption of ions in the electrolyte, forming sheets on both electrodes at the electrode-electrolyte interface. Low cost carbon porous materials are normally utilized as DL electrode materials because of their high specific surface area and phenomenal mechanical and chemical stabilities [2-3]. In contra, pseudocapacitors accumulate charges based on a rapid and reversible reduction and oxidation (redox) reaction of active material with the electrolyte on the surface [4]. Supercapacitor plays a significant part as a successful energy storage device to address the challenging problems of fossil fuel depletion and climate change that has a high power capacity and a long life span compared to the commercial batteries [5-6]. In addition, because it meets the need for a wide range of energy application, it is also used as portable devices, power backup systems, and electric vehicles. In dealing with the disadvantages of the low energy density of the supercapacitors, the development of electrode/active materials is needed to boost energy storage efficiency.

Polyaniline (PANI), one of the extensively considered conductive polymers for electrochemical applications, has certain drawbacks, such as reduced solubility, process efficiency and decreased stability due to structural conformation, shrinkage, and cracking [7]. Due to its distinct electronic and electrochemical properties in the reduced and oxidized states, PANI is quite desirable among the numerous conductive polymers [8]. PANI provides advantage like its rapid switching between redox states with a high degree of electrochemical reversibility [9]. PANI has typically been used as positive electrode materials for asymmetric supercapacitors and a broad range of different capacitance values have been recorded, e.g. from 10 to over 1,000 of F/g.

Graphene oxide (GO) has been popular for the supercapacitor application due to its unique structure, low weight, good mechanical properties. The GO framework includes a huge range of functional groups (carboxyl, hydroxyl, epoxy group) [10]. This made GO a suitable candidate for the reinforcement of electrode materials due to the specific functional groups which provide good dispersion and hydrophilic behavior. Furthermore, GO also has great total surface area, huge chemical potential, great chemical stability, and rich drape morphology. The cyclic stability and conductivity of the electrode increased with GO incorporation. On the other hand, a single-atomic layered material can be produced by powerful graphite oxidation, which is cheap and abundant. GO is an oxidized graphene form with inferior electrical conductivity, but they undergone processes for properties enhancement. Hummers and Offeman eased the procedures by utilization of potassium permanganate ($KMnO_4$) as an oxidizer instead of potassium chlorate ($KClO_3$); not popular because it produces harmful chlorine dioxide (ClO_2). Hummers is often the most preferred method [11] accounting for its simplicity and adaptability.

GO is an active component of PANI nanocomposites regardless of the way that it contains a much lower electrical conductivity, due to the dynamic utilitarian bunches that connected chemically with PANI chains. Building the chemical interactions between PANI and the functional groups formed on the surface can naturally improve the specific capacitance and reduce the load transfer resistance. GO would help increases PANI dispersion, and thus will greatly boost PANI efficiency and result in much higher performance. PANI/GO nanocomposites often exhibit strong electrochemical efficiency, high specific potential and long cycle stability. Therefore, this made PANI/GO is a suitable material for supercapacitor electrode. Several types of research analyzed the surface of GO and found extremely deficient zones, presumably because of the presence of oxygen and relatively undamaged areas [12-14]. The graphene-like honeycomb lattice in GO is conserved, i.e. the carbon atoms attached to the functional groups are slightly moved, but the estimation of the unit cell in GO remains comparable to that of graphene [15]. The functional group may influence the thermal and electrical conductivities of GO. Graphene and GO expanded fascination of scientists rapidly in different fields whereby numerous potential applications are in progress and proposed the usage of graphene and GO, for example, sensors, batteries and supercapacitors [16-17].

Basically, this work is focusing on the synthesis of GO by improved Hummers method [18], synthesis of PANI by chemical oxidation method [19], fabricating the supercapacitor electrode and analyzing the electrochemical performance of the electrode. Graphene is an excellent material for hosting conductive polymers due to its high electrical conductivity and surface properties [20], and PANI can improve other carbon materials' electrochemical ability. The synergistic effect between the two components can result in a substantial increase in efficiency. The nanocomposite electrodes were fabricated using the conventional slurry technique where PANI/GO, carbon black, and polytetrafluoroethylene (PTFE) binder with a weight ratio of 80, 10, 10 wt. %, respectively are deposited on top of the Ni foam current collector. Cyclic voltammetry and galvanostatic charge-discharge techniques were used to analyze the electrochemical performance of PANI/GO nanocomposite supercapacitor electrode.

2. Methodology

Figure 1 shows the methodology flow chart of the work. Main idea is to synthesize GO and PANI, then followed by the nanocomposite preparation. Table 1 lists the materials needed to synthesize the GO and PANI. For GO synthesis, a modified Hummer's method was applied. First, a mixture of graphite powder (3 g) was dispersed in 70 ml H_2SO_4 and the mixture was stirred at 5 °C. $KMnO_4$ (9 g) was added to the suspension with slowly controlled rate. Then the mixture was heated to 35 and 85 °C and stirred again for 1 h. Deionized (DI) water (100 ml) was gradually added during the heating process to dilute the mixture until the color shifted to brown. Eventually, the solution was poured into 30 ml of hydrogen peroxide (H_2O_2), and the color changed to bright yellow. After filtering the solution, the solids were washed with 5 % HCl, ethanol and DI water to neutralize solution's pH. GO was obtained as loose brown particles after an overnight drying under vacuum.

PANI was prepared by a chemical synthesis, namely oxidize polymerization. First, aniline (2 ml) was added to the 50 ml HCl solution and stirred for 40 mins and at the same time ammonium persulfate (APS; 2.5 g) was added to another 50 ml HCl and stirred for 40 mins. Aniline solution is moved into an ice bath and APS solution was slowly added into the aniline solution drop by drop. After that, the solution was stirred in an ice bath for 5 h. The beaker was then removed from the ice bath and left cooled at room temperature. Finally, the solution was filtered to obtain the precipitate and dried it in an oven at 60 ^{o}C for 24 h.

Briefly, to prepare the PANI/GO nanocomposite electrode, carbon black and polytetrafluoroethylene (PTFE) binder were mixed from a weight ratio of 80:10:10 (wt.%) in alcohol under sonication to form homogenous slurry. Then, the slurry was coated onto Ni Foam with a surface area of 1.0 cm^2 using a brush then the Ni foam was dried in oven at 60 ˚C for 8 h to remove the solvent and water moistures. The three samples of the PANI/GO nanocomposite electrode with different weight compositions are fabricated and labelled as PGO30 (70% PANI-30% GO), PGO50 (50% PANI-50% GO), and PGO70 (30% PANI-70% GO). The weight of the electrode for PGO30, PGO50 and PGO70 was measured using electronic weighing scale. The average mass of all PGO nanocomposite electrodes is 4.80 mg and this was value used to calculate the specific gravimetric capacitance. The electrodes were assembled in a stainless steel battery jig and the electrochemical performance was studied using two-electrode cell configuration (Figure 2). A 6 M potassium hydroxide (KOH) aqueous solution was employed as electrolyte for the supercapacitor. The cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) tests were performed using potentiostat/galvanostat (Wonatech; WMPG1000S) at room temperature. In addition, the morphology of the synthesized PANI and GO was studied by using scanning electron microscope (SEM; Hitachi SU3500).

Table 1
Materials/chemicals used to synthesize GO and PANI

Material/Chemical	Quantity	Remark
Graphite powder	3.0 g	For GO
Potassium permanganate ($KMnO_4$)	9.0 g	
Phosphoric Acid (H_3PO_4)	7.78 ml	
Sulfuric Acid (H_2SO_4)	70 ml	
Hydrogen Peroxide (H_2O_2)	30 ml	
Ammonium persulfate ($(NH_4)_2S_2O_8$)	2.5 g	For PANI
Hydrochloric Acid (HCl)	100 ml	
Aniline	2 ml	

3. Results and discussion

3.1 The Morphology of the PANI and GO

The structures of the PANI and GO were investigated by scanning electron microscopy (SEM). The SEM images of GO is shown in Figure 3 (a). It is observed that the GO produced from strong oxidizing agent ($KMnO_4$) was crumpled in shape and nearly transparent at the edge of GO layers. While a much rougher surface with wrinkles is observed in inner sheet due to the exfoliated graphite flakes into graphene oxide [21-22]. Figure 3 (b) displays the SEM image of PANI. It was found that by chemical oxidative polymerization of aniline, formation fiber-like PANI was appeared with aggregated of nano-fibrous structure [23- 24]. The PANI consisting of nanofibers and the range of fibers from hundreds of nanometer to several micrometers in length, where the composition of the networks tends to be more compact and comparatively small. The creation of nanofibers is due to the self-assembly of individual polymers or oligomer through the interaction between aniline oligomers, the hydrogen bonding and Vander Walls forces that produce polymer nanofibers.

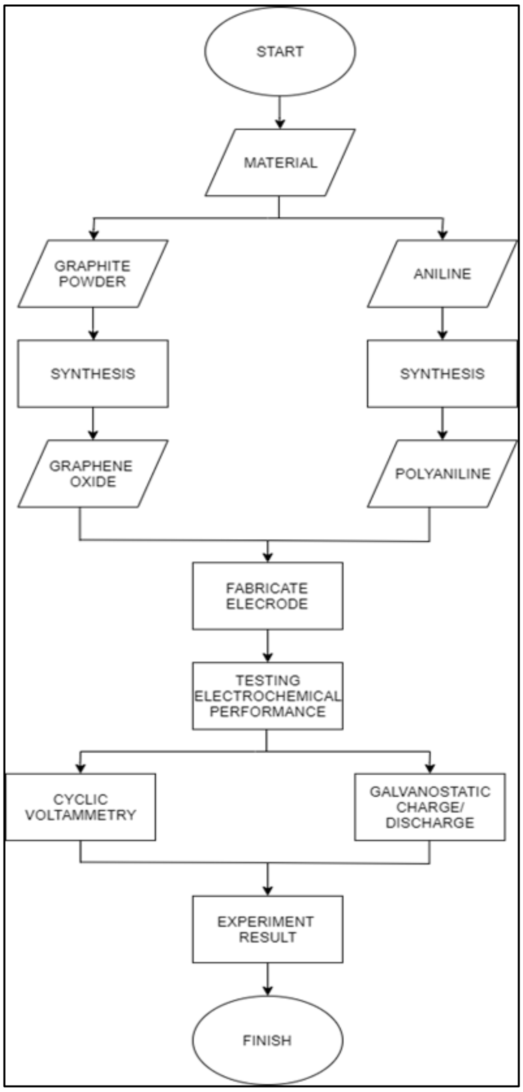

Fig. 1. Flow chart of the project's methodology

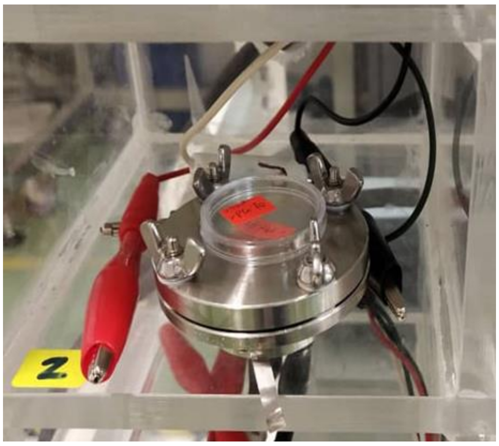

Fig. 2. PANI/GO nanocomposite electrodes were assembled in a battery jig for electrochemical testing

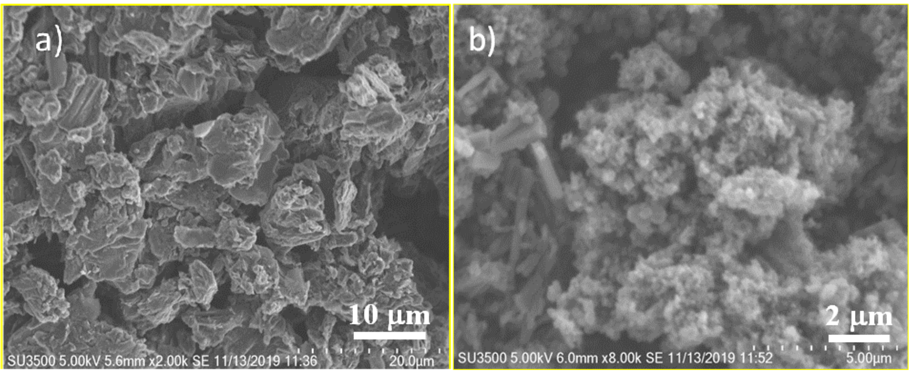

Fig. 3. SEM images of (a) GO and (b) PANI

PGO nanocomposites have finer particle grains of the surface morphology (Figure 4). The particulate amount of PGO70 nanocomposite is less than PGO30 and PGO50. It was noticed that the larger the polyaniline fraction of a substance, the greater the particle dimensions. All PGO nanocomposites samples also seems to be agglomerated. Furthermore, it indicates that GO pores and galleries are planted with polymers. Therefore, the independent step GO and PANI in the PGO nanocomposite is difficult to discern. Also, the nanofiber of the polymer was not clear on the GO. Other than that, on the top and edge of crumpled GO boards, the PANI scales were thinly powdered. At the same time, the PGO nanocomposite was much thicker and the surface much rougher than the original GO, mainly because the overlap level had decreased significantly.

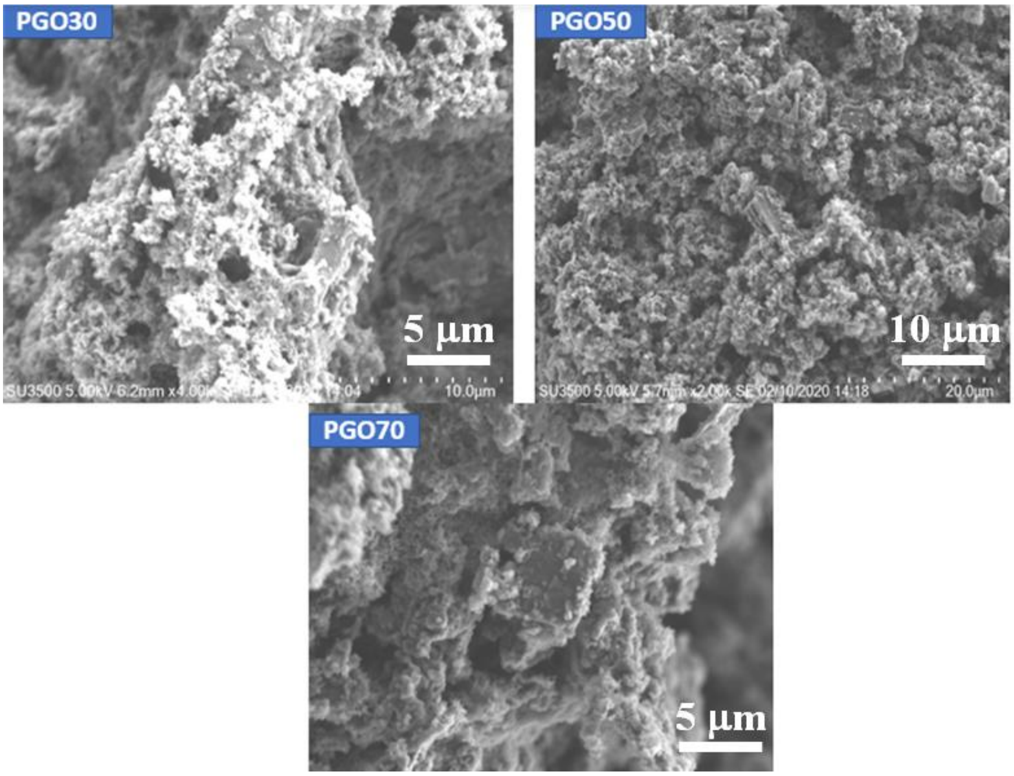

Fig. 4. SEM images of PGO nanocomposites

3.2 Cyclic Voltammetry Analysis of PANI/GO Nanocomposite Electrode

The performance of the PANI/GO nanocomposite electrode was first analysed through CV technique. CV provides information about the stability of the complexed transition metal oxidation state, the reversibility of electron transfer reactions, and the reactivity information [25]. Its specific gravimetric capacitance (C_{sp}) was calculated from the results obtained by using Eq. 1:

$$C_{sp} = \frac{\int_{E_1}^{E_2} i(E)dE}{2(E2-E1)mv'} \tag{1}$$

where E1 and E2 are cut-off potentials during CV, i(E) is current (A), $\int_{E_1}^{E_2} i(E)dE$ is the total voltammetric charge obtained by integration of positive and negative sweeps during CV, (E2-E1) is the potential window (V), m is the average mass of active material in gram (g) and v is the CV scan rate (V/s). The calculated C_{sp}s from CV curves of PANI/GO nanocomposite electrodes at different scan rates are tabulated in Table 2. The CV curves of all nanocomposite electrodes were plotted with different CV scan rates of 5, 10, 30, 50, 70, 90 mV/s with the potential range of 0.0 - 1.0 V.

The CV curves of PGO30 electrode were plotted and shown in Figure 5 (a). All curves showed the tendency of redox peak appearance and reflect a mixture of both double layer and pseudocapacitance. Due to the PANI's redox transition between leucoemeraldine base states and emeraldine salt, a further transformation from emeraldine to pernigraniline can be suggested [26]. Redox peaks are originated from the faradaic reactions occurring between electrodes' surface and electrolyte ions [27]. The highest capacitance of PGO30 electrode is 13.85 F/g at 5 mV/s. Next, the CV curves of PGO50 electrode were plotted and shown in Figure 5 (b). The highest C_{sp} of PGO50 electrode is 19.71 F/g at 5 mV/s. This is the highest recorded C_{sp} of all samples. This may be attributed from the good electrical conductivity distribution of PANI and graphene oxide. Graphene sheets act as an electronic conductive channel in the composite, and is beneficial for the fast transfer of ions and electrons throughout the electrode surface.

Finally, the CV curves of PGO70 electrode are shown in Figure 5 (c). When the PANI load is too small the agglomeration of the GO may be enhanced. The highest specific capacitance of PGO70 electrode is 9.65 F/g at 5 mV/s. In all electrodes, the specific capacitance was found to decrease with the increasing scan rates. This is due to the fact that the ion concentration on the electrode-electrolyte surfaces is rapidly increasing and the diffusion rates of the electrolyte ions from the interface to electrode still not enough to satisfy the electrochemical reactions. In addition, Figure 6 depicts the C_{sp} as a function of CV scan rate for PANI/GO nanocomposite electrodes. The trend and the values of the capacitance are almost similar for all electrodes.

Table 2
Calculated C_{sp} of CV curves of PANI/GO nanocomposite electrodes at different scan rates

CV scan rate (mV/s)	C_{sp} (F/g) PGO30	PGO50	PGO70
5	13.85	19.71	9.65
10	7.06	10.10	2.70
30	2.82	5.29	1.51
50	2.50	4.24	0.96
70	0.69	0.74	0.65
90	0.64	0.53	0.48

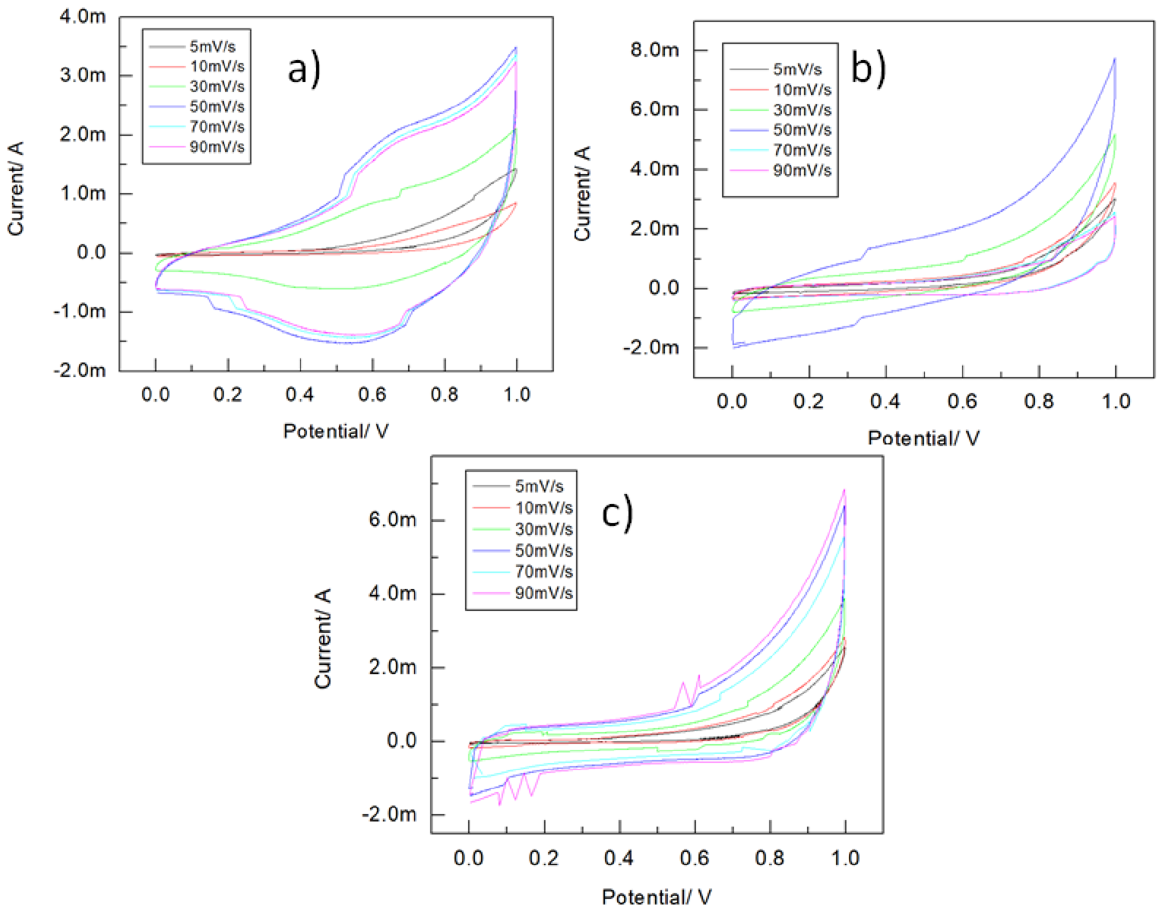

Fig. 5. CV curves of PANI/GO nanocomposite electrodes. a) PGO30, b) PGO50 and c) PGO70

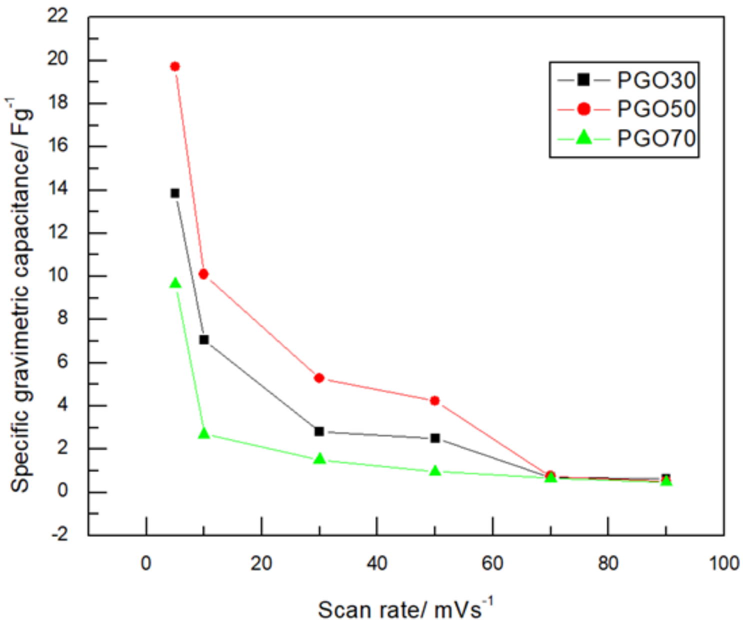

Fig. 6. Specific gravimetric capacitance as a function of CV scan rate for PANI/GO nanocomposite electrodes

3.3 Charge-discharge Analysis of PANI/GO Nanocomposite Electrode

Meanwhile, galvanostatic charge-discharge (GCD) is also a complementary method for assessing particular capacity under controlled current conditions. Discharge curves were also used to calculate the C_{sp} using Eq. 2:

$$C_{sp} = \frac{I \times \Delta t}{\Delta V \times m} \tag{2}$$

where I is the applied current, Δt is the CD discharge time, ΔV is the potential, and m is the average mass of the active material in the electrode. The calculated C_{sp}s from GCD curves of PANI/GO nanocomposite electrodes at different currents are tabulated in Table 3. The GCD curves of all PGO nanocomposite electrodes were plotted with different currents of 0.1, 0.3, 0.5, 0.7, 0.9, 1.0 mA with the potential range of 0.0 - 0.8 V (note that this is differed with the CV analysis).

The GCD curves PGO30 electrode were plotted and shown in Figure 7 (a). The charge curves of the electrode are symmetrical to their corresponding discharge, suggesting high reversibility of PGO30 electrode. PANI nanofibers cross-link to each other to form a uniformly distributed framework on the GO surface and thus provide a vastly accessible interface. The GO sheet-covered PANI system will include more active sites to facilitate redox reactions during the charge and discharge cycle. The GCD curves PGO50 electrode were plotted and shown in Figure 7 (b). The discharge curves of PANI/GO nanocomposites are also symmetric to their discharge counterpart with a slight curvature for internal resistance during the charging and discharging phases. The GCD curves also display a nonlinear relationship between voltage and time, showing a mixture of both pseudocapacitance and DL capacitance. The GCD curves PGO70 electrode are shown in Figure 7 (c). The curve of charge is asymmetric with the discharge counterpart. When the current density is increasing, the capacitance is slightly decreasing. The time taken for the electrode to complete one cycle also decreases from 30 s to 5 s. When the PANI content decreases, the charge discharge time also decreases, meaning that the nanocomposite's basic capacitance is higher when the PANI content is greater. Once the PANI content is low, the sample has large impedance or resistance and at the time of the charge discharge transition there is a major mutation, which results in unsatisfied results.

From the overall results, the capacitance values from GCD results are much lower than that of CV results. One of the reasons is that the applied voltage or potential during the GCD was set to only 0.8 V and it was 1.0 V during the CV analysis. This applied to the different measurement techniques which mean the variation of voltage per time scan rate for CV, whilst varying the current for the charge/discharge technique. Figure 8 plots the C_{sp} as a function of current density for PANI/GO nanocomposite electrodes during GCD analysis. It was found that PGO30 showed the highest calculated capacitance value. Unfortunately, this is contra to that of CV analysis results. It can be suggested that this could be due to the inconsistency preparation of the active materials which may affected the quality of the fabricated electrode during the coating process.

Table 3
Calculated C_{sp} of PANI/GO nanocomposite electrodes from GCD curves at different currents

Current (mA)	Current Density (A/g)	C_{sp} (mF/g)		
		PGO30	PGO50	PGO70
0.1	0.02	1070	310	650
0.3	0.06	1070	420	620
0.5	0.10	1100	450	620
0.7	0.15	1170	500	450
0.9	0.19	950	380	530
1.0	0.21	1000	290	330

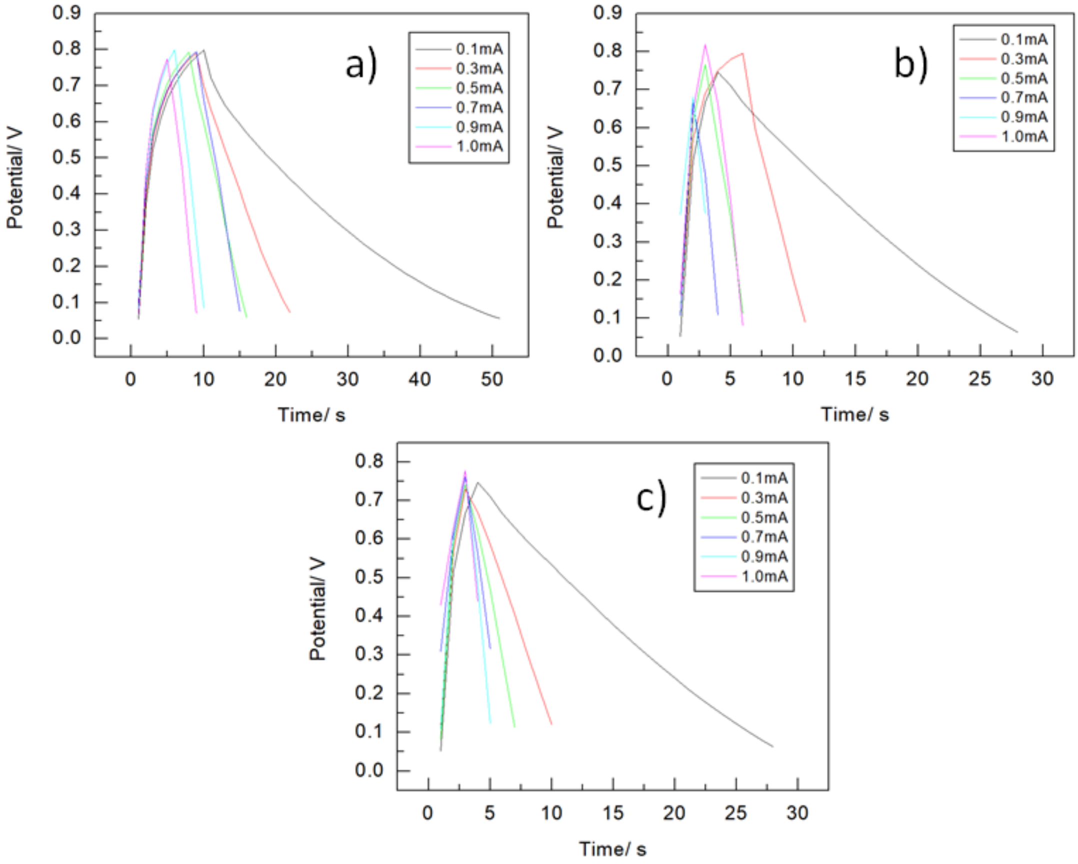

Fig. 7. GCD curves of PANI/GO nanocomposite electrodes, (a) PGO30, (b) PGO50 and (c) PGO70

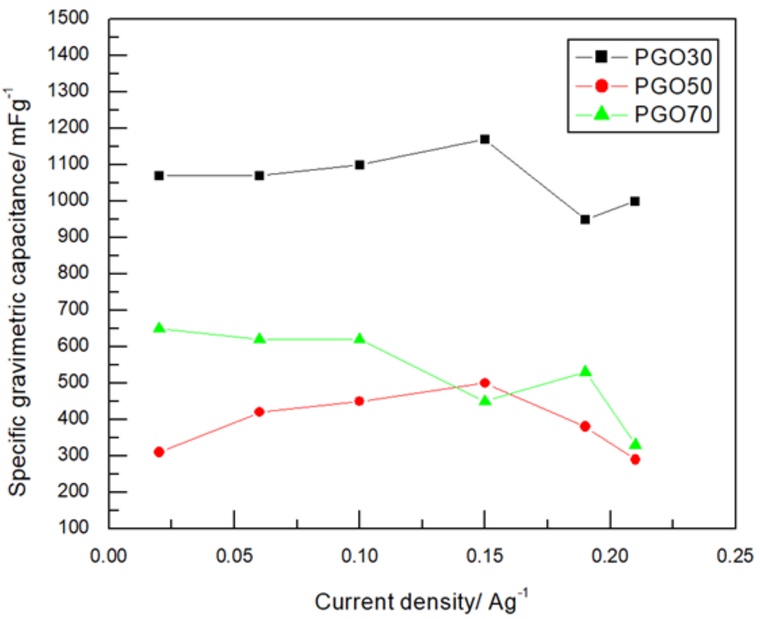

Fig. 8. Specific gravimetric capacitance as a function of current density for PANI/GO nanocomposite electrodes during GCD analysis

4. Conclusions

GO was prepared by improved Hummers method and PANI was successfully prepared by chemical oxidation method. From the CV analysis, it can be concluded that the highest percentage of PANI improved the performance of PANI/GO as electrode for supercapacitor application. The highest calculated specific capacitance from the CV analysis is the PGO50 electrode with 19.71 F/g at 5 mV/s scan rate. From the GCD analysis, it was found that the PGO30 nanocomposite electrode showed the best result due to the charge curve is asymmetric to its corresponding discharge counterpart, indicating its high reversibility. PANI is extensively studied as one of the conductive polymer for electrochemical applications because of its rapid charge transfer between redox states with a high degree of electrochemical reversibility. In short, attention is required for the important aspects (including mixing process, electrolyte usage, testing parameter, etc.) to consider in the future for the advancement of any type of nanocomposite electrode for supercapacitor.

Acknowledgement

Authors are grateful for the facilities and financial support from Universiti Teknikal Malaysia Melaka and Universiti Kuala Lumpur.

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