Morphological Study of Synthesized RGO/ Pt Nanocomposites via Facile Chemical Reduction Method

(Kajian Morfologi Komposit Nano RGO/ Pt yang Disintesis melalui Kaedah Penurunan Kimia secara Mudah)

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ABSTRACT

Reduced graphene oxide nanosheet (RGO)/Pt nanocomposite have been successfully prepared through a facile chemical reduction method. The reduction of Pt precursor was carried out using sodium borohydride as the efficient chemical reductant. The morphology of RGO/Pt nanocomposite was investigated using high resolution transmission electron microscopy (HRTEM) and field emission scanning electron microscopy (FESEM). HRTEM analysis showed that platinum nanoparticles were homogenously distributed onto the surface of RGO. The electrochemical study proved that Pt nanoparticles were successfully incorporated onto RGO. Therefore, it can be concluded that the proposed method could provide well-dispersed of Pt nanoparticles onto RGO to form RGO/ Pt nanocomposite.

Keywords: Chemical reduction; electrochemical; morphology; reduced graphene oxide nanosheet; RGO/ Pt nanocomposite

ABSTRAK

Komposit nano RGO/Pt telah berjaya disediakan melalui kaedah penurunan kimia secara mudah. Penurunan bahan pemula Pt telah dijalankan dengan menggunakan natrium borohidrida sebagai bahan penurun kimia yang efisyen. Morfologi komposit nano RGO/Pt telah dikaji menggunakan HRTEM dan FESEM. Analisis HRTEM menunjukkan bahawa zarah nano platinum telah diagihkan secara sekata ke atas permukaan RGO. Kajian secara elektrokimia membuktikan bahawa zarah nano Pt telah berjaya diperbadankan ke atas RGO. Oleh itu, dapat disimpulkan bahawa kaedah yang dicadangkan ini boleh menyediakan penyebaran zarah nano Pt yang baik ke atas RGO untuk membentuk komposit nano RGO/Pt.

Kata kunci: Elektrokimia; kepingan grafena oksida yang diturunkan; komposit nano RGO/Pt; morfologi; penurunan kimia

INTRODUCTION

A green power source of direct methanol fuel cell (DMFC) has generated tremendous for portable electronic devices and power electric vehicles (Ma et al. 2013). The chemical reaction occurs at the anode in DMFCs comprises methanol oxidation over platinum (Pt) as noble metal catalysts. Pt can be considered as an efficient catalyst for methanol oxidation reaction which shows remarkable catalytic activity among other pure metals (Gao et al. 2013). However, insufficient catalytic activity, poor durability and high cost of Pt catalyst has prevent its application in the field of DMFCs. The emerging of carbonaceous materials as highly conductive support for Pt catalyst such as mesoporous carbon (Wen et al. 2008), multi-walled carbon nanotubes (Hull et al. 2006), single-walled carbon nanotubes (Kongkanand et al. 2006) and carbon nanofibers (Steigerwalt et al. 2001) have become an alternative anode catalyst in DMFC. Therefore, many efforts have been focused on the dispersion of Pt catalyst and the property of carbon support to enhance the catalytic activity (Lin et al. 2005).

Recently, graphene is widely used as the catalyst support in fuel cells due to the extraordinary mechanical strength (Lee et al. 2008), excellent electrical conductivity (Service 2009) and thermal conductivity (Balandin et al. 2008). It is one of the carbonaceous materials with that arranged in the two-dimensional and rigid honeycomb lattice structure comprising of sp^2 carbon atom (Choi et al. 2012). In particular, graphene has large surface area to volume ratio and suitable for the electrochemical reaction. This enables graphene to be considered as the promising candidate to be used as Pt catalyst support (Liang et al. 2013). Dispersion of Pt nanoparticles on graphene are widely investigated by many researchers. The methods to deposit the Pt nanoparticles onto graphene are extensively studied. In previous research, Pt nanoparticles and graphene were synthesized separately whereby the complex was formed by adding the polymer and surfactant (Ji et al. 2012).

Guo et al. (2010) produced rapidly a new Pt nanoparticle ensemble-on-graphene hybrid nanosheet through a microwave-assisted heating by adding poly (methacrylic acid sodium salt). Besides, the dispersion of Pt nanoparticles on graphene functionalized with poly (diallyldimethylammonium chloride) has been reported by Luo et al. (2012). The resulting composite

shows remarkable electrochemical activity due to the homogenous dispersion of small size Pt nanoparticles. However, the multi-steps procedures may reduce the catalytic activity capability of Pt. Finding the most cleanly and facile method for the Pt nanoparticles formation which can be incorporated into graphene is still remains a great challenge.

Sodium borohydride has the potential to reduce Pt ion to form Pt nanoparticles (Van Rheenen et al. 1987) that can deposit on the graphene. It is recommended to eliminate the stepwise reduction procedures by using excessive sodium borohydride. In addition, the remaining oxygen-containing functional groups existed on the graphene could provide active sites to anchor the Pt nanoparticles (Kou et al. 2009). In this study, RGO/ Pt nanocomposite was fabricated via simple chemical reduction process in which $PtCl_4^{\,2-}$ ions were reduced into Pt nanoparticles in the solution by using powerful chemical reductant, sodium borohydride. The produced RGO/ Pt nanocomposite has the high potential as enhanced catalyst which can be implemented in the DMFC system.

EXPERIMENTAL DETAILS

CHEMICALS

Sodium nitrate ($NaNO_3$, Riedel-de Haën), graphite powder (Superior Graphite Co. Ltd.), sodium oxalate ($Na_2C_2O_4$) (Fisher brand), hydrogen peroxide (H_2O_2, 35%) (RCI Labscan), Hydrochloric acid (HCl, 37 %) (RCI Labscan), Potassium Permanganate ($KMnO_4$, COMAK), Nafion solution (5 wt. %, Sigma Aldrich), concentrated sulfuric acid (H_2SO_4, 95-97%), (QRëC), chloroplatinic acid hydrate ($H_2PtCl_6.\times H_2O$, Sigma Aldrich), sodium borohydride ($NaBH_4$, Sigma Aldrich), sodium hydroxide (NaOH, Sigma Aldrich) and ethanol (QRëC). All the chemicals were used without further purification.

PREPARATION OF GRAPHENE OXIDE NANOSHEETS (GO)

GO was produced through the modified Hummers' method which involved oxidation of graphite (Hanifah et al. 2015b). 3 g of $NaNO_3$ was added into 140 mL of concentrated H_2SO_4 in an ice bath. Then, 3 g graphite was slowly added into the above mixture followed by 15 g of $KMnO_4$. The temperature was maintained less than 20°C while vigorous stirring for the duration of 2 h. Later, temperature was increased and fixed at 35°C. The mixture was stirred for extra 12 h to form brown paste. After that, 250 mL of deionized water was added slowly into the mixture and stirring was continued for 30 min. 20 mL of H_2O_2 and 800 mL of deionized water were added into the hot mixture. The process of stirring was continued until the mixture turn into yellowish colour. The resulting graphite oxide precipitate was subjected to ultrasound irradiation for 1 h by a sonicator to exfoliate the graphite oxide into a GO. The GO was then filtered, washed with 1 M HCl and deionized water until the pH nearly neutral. Finally, the GO was dried overnight in oven at 80°C.

PREPARATION OF REDUCED GRAPHENE OXIDE NANOSHEETS (RGO)

RGO was produced through the chemical reduction reaction of exfoliated GO based on the previous work (Hanifah et al. 2015a). Initially, the sonication process was conducted to disperse 200 mg of GO in 200 mL of deionized water for 3 h until dark-brown GO aqueous colloid was obtained. Then, 100 mL of the GO suspension was mixed throughout with 40 mL of $Na_2C_2O_4$ solution. The resulting mixture was maintained at 95°C for 2 h. Lastly, the produced RGO were centrifuged, washed with deionized water for several times and dried in oven at 80°C.

FABRICATION OF RGO/PT NANOCOMPOSITE

The RGO/Pt nanocomposite was fabricated via chemical reduction process according to our previous report (Hanifah et al. 2016). Typically, 2 mL of $H_2PtCl_6.\times H_2O$ solution (0.1 M) was added into 30 mL of RGO solution (1.0 mg/mL). Then, the suspension was sonicated for 30 min to obtain the homogenous distribution of Pt nanoparticles. A certain amount of NaOH solution (1M) was added into the above solution. After that, under vigorous stirring, 200 mL of sodium borohydride (1.0 mg/mL) was added drop wise into the solution and the temperature was fixed at 85°C for 3 h. The resulting homogenous solution was then centrifuged at 4000 rpm and further purified with distilled water for several times.

CHARACTERIZATION

The morphology and the microstructure of the samples were characterized by field emission scanning electron microscopy (FESEM), transmission electron microscopy and high-resolution transmission electron microscopy (HRTEM, FEI Tecnai G2) by using voltage of 120 and 200 kV, respectively. The samples for both TEM analysis were prepared by dispersing the samples in ethanol ultrasonically for 10-15 min and subsequently, a drop of the suspension was placed on a carbon-coated copper grid and dry in a few minutes. The analysis of chemical composition can be performed using EDX analysis. XRD patterns of sample was obtained on a high resolution X-ray Diffractometer System (Bruker D8 Advance) with 2θ range between 20° and 90° by using CuKα (λ = 1.54 Å). The Raman spectra were recorded with a RENISHAW 1000 confocal Raman microprobe using argon ion laser frequency of 514 nm as an excitation source. Electrochemical measurement was conducted on an electrochemical work station (e-corder 410) for the cyclic voltammetry (CV). Ag/AgCl electrode and platinum wire were applied as the reference electrode and the counter electrode, respectively. A thin layer of RGO/ Pt nanocomposite was covered onto a glassy carbon electrode with diameter of 3 mm and act as the working electrode.

RESULTS AND DISCUSSION

MORPHOLOGICAL ANALYSIS OF SYNTHESIZED RGO

The surface morphology of GO and RGO were identified using TEM and HRTEM, respectively. Figure 1(a) and 1(b) shows the surface morphology of scrolled GO and RGO, respectively. From the figure, it can be seen that the interlayer coherence of GO was not damaged (McAllister et al. 2007). The wrinkled paper-like structure was observed and arranged in transparent layer of RGO which is very stable under electron beam (Amamath et al. 2011). The distinct layers number could be given by RGO folder edges (Hanifah et al. 2015a; Liu et al. 2013). HRTEM image of the RGO edge with high magnification are demonstrated in Figure 1(c). The most tansparent regions of RGO are expected to have a single layer or a few layer as showed by the arrow.

MORPHOLOGICAL ANALYSIS OF RGO/PT NANOCOMPOSITE

Figure 2 exhibits HRTEM results of produced RGO/Pt nanocomposite. Figure 2(a), 2(b), 2(c) and 2(d) shows the various magnification, whereby the Pt nanoparticles appear as black spots onto the RGO. These deposited of Pt nanoparticles are distributed evenly onto the basal planes and at the edge of RGO surface. It also proved that the sodium borohydride could reduce the Pt ions to form Pt nanoparticles, which strongly deposited on the RGO surface.

From the HRTEM images, some of the wrinkles surface with light shaded are actually corresponded to RGO. The average of Pt nanoparticles size was approximately 4.47 nm based on Scherrer equation.

DISPERSION OF PT NANOPARTICLES ONTO RGO

Figure 3 exhibits the typical SEM and FESEM images of RGO and RGO/Pt nanocomposites, respectively. The FESEM was conducted without Pt coating toward the samples to prevent the interference from the coated Pt. Figure 3(a) and 3(b) shows the surface morphology of RGO and RGO/Pt nanocomposite with same magnification. The Pt nanoparticles were embedded and dispersed uniformly onto the surface of RGO. The Pt nanoparticles were anchored in the wrinkled RGO indicating strong distribution and deposition of Pt nanoparticles onto RGO. The result is tally with the observation done by HRTEM as discussed, previously. In addition, the Pt nanoparticles can be effectively supported by the wrinkled nature of RGO in a large surface area. The Pt nanoparticles shape is spherical with irregular size as shown in Figure 3(c).

STRUCTURAL ANALYSIS OF RGO/PT NANOCOMPOSITE

The determination of the crystallinity of RGO/Pt nanocomposite can be conducted by XRD analysis. Our previous report showed that the RGO/Pt nanocomposite
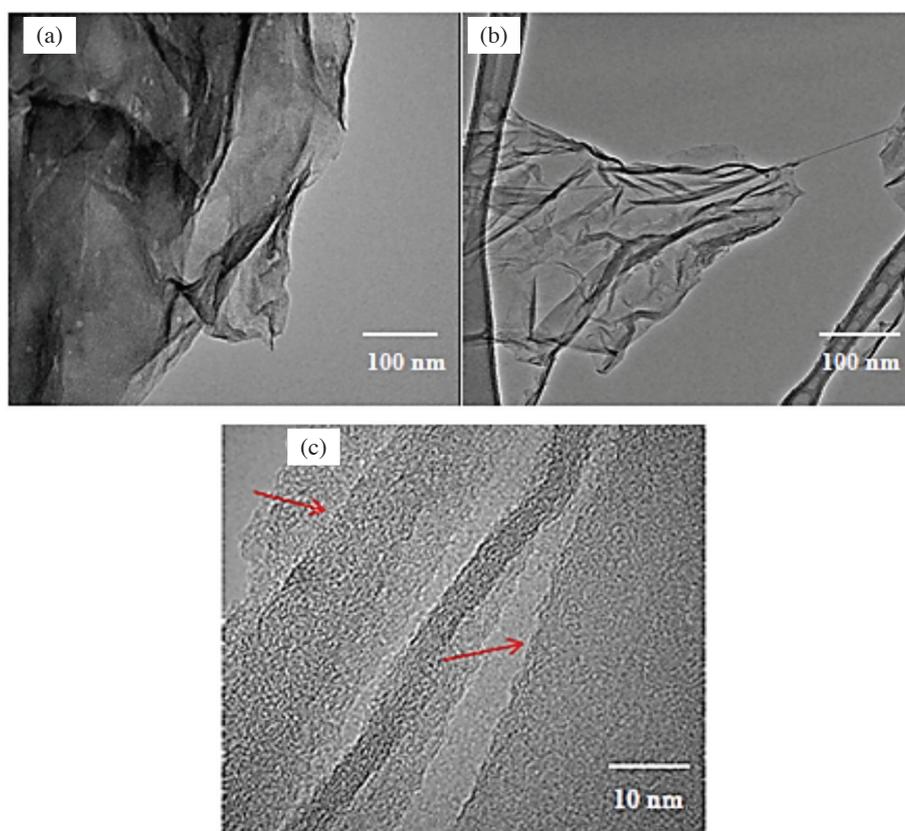

FIGURE 1. (a) TEM images of GO, (b) and (c) HRTEM images of RGO surface morphology and its edge, respectively
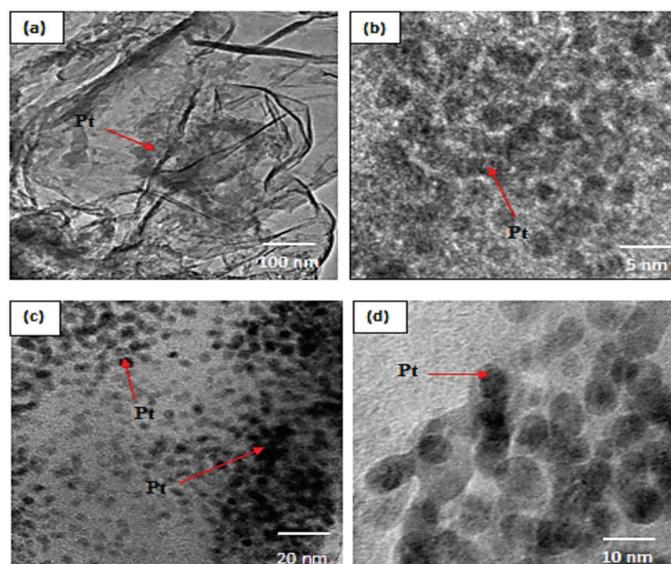
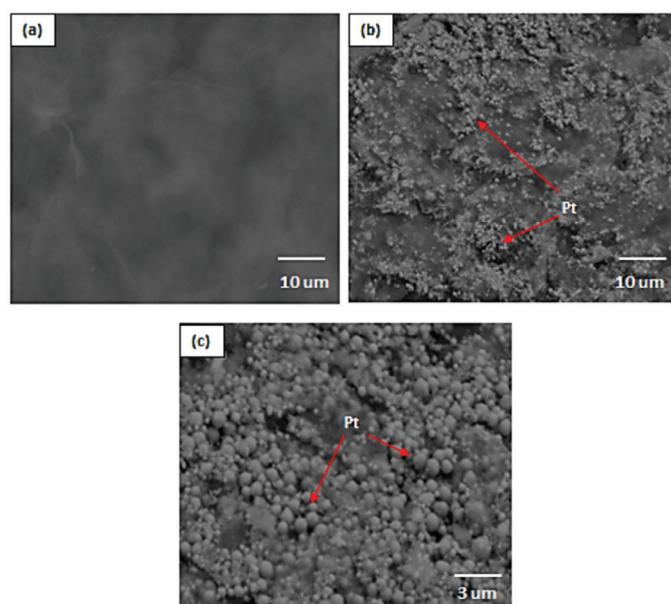FIGURE 2. HRTEM images of RGO/ Pt nanocomposite with different magnification

FIGURE 3. FESEM images of (a) RGO, (b) and (c) RGO/ Pt
nanocomposite with different magnification

had the broad diffraction peak at around 27.7° which was assigned to the hexagonal graphite structures C (002) (Hanifah et al. 2016). The typical peaks of the Pt nanoparticles-loaded RGO are summarized in Table 1. These results confirmed that the Pt precursor ($H_2PtCl_6 \cdot xH_2O$) had been reduced to Pt nanoparticles and successfully loaded onto the RGO without any impurity. The Pt (111) plane is dominating the orientation since it has the highest intensity than the others. Figure 4 shows the diffraction pattern of RGO/Pt nanocomposite.

TABLE 1. Fitting XRD data summary for RGO/ Pt nanocomposite catalyst

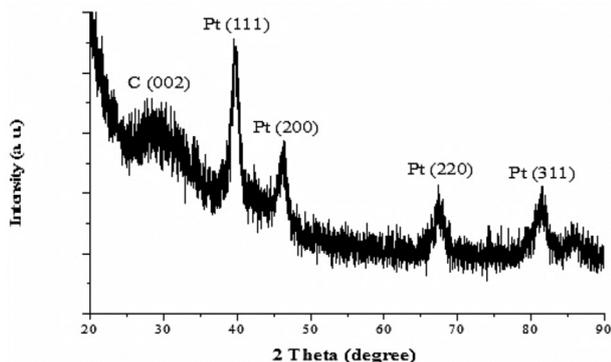| Pt plane	Pt (111)	Pt (200)	Pt (220)	Pt (311)
Angle (2θ)	39.6	46.1	67.4	81.4
d-value (nm)	0.227	0.197	0.139	0.118
Net intensity	65.5	21.7	16.3	13.3

FIGURE 4. XRD pattern of RGO/ Pt nanocomposite
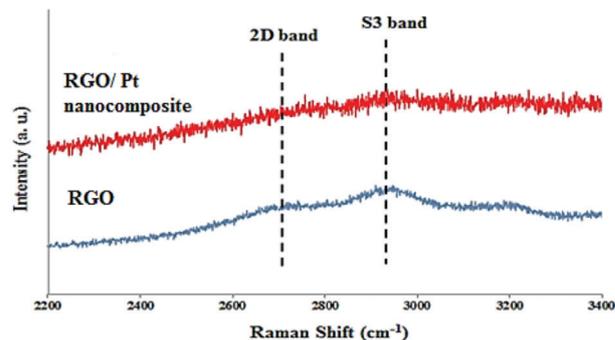(Hanifah et al. 2016)

FIGURE 5. Second-order Raman spectra of RGO
and RGO/Pt nanocomposite

The defect structure of RGO/ Pt nanocomposite and RGO can be analyzed by Raman spectrophotometer. The second-order of Raman spectra for RGO and RGO/ Pt nanocomposite are depicted in Figure 5. The 2D peak is very important characteristic in the Raman spectrum because it can clearly distinguish between single layer,

bilayer and a few layers of RGO via its position and shape. The 2D peaks of RGO and RGO/Pt nanocomposite occurred at 2692 and 2704 cm^{-1}, respectively, as shown in Figure 5. It was suggesting a red-shift after reduction process while their S3 peaks located similarly at 2933 cm^{-1}. This is the evidence that the analyzed region presence of a few layers
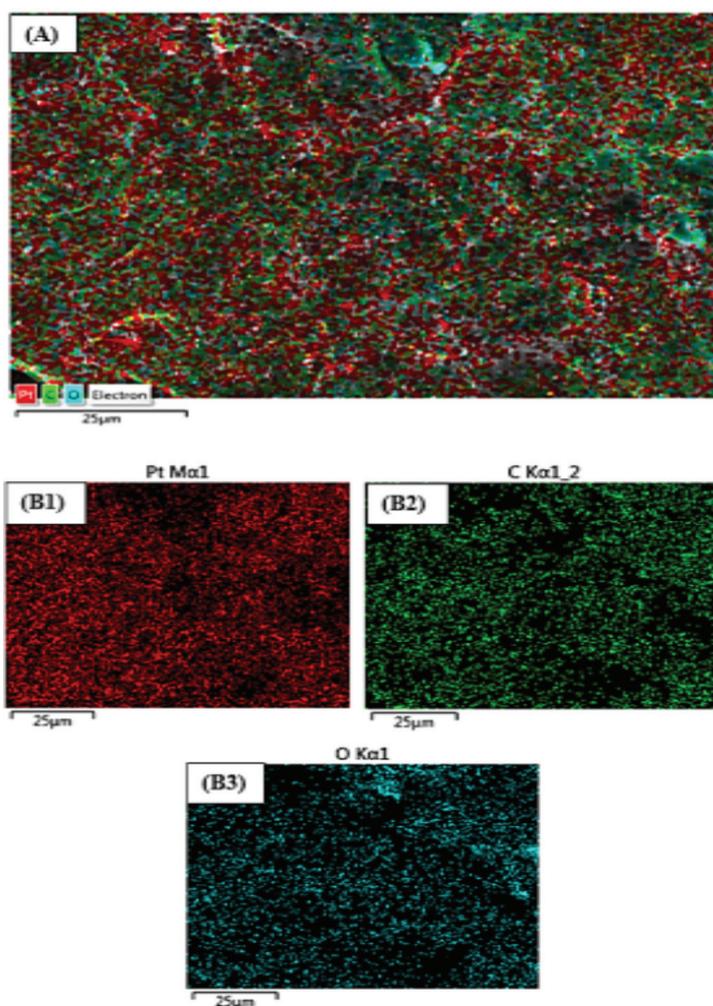

FIGURE 6(a). FESEM-EDX mapping images of over-lapping Pt-C-O (A), Pt (B1),
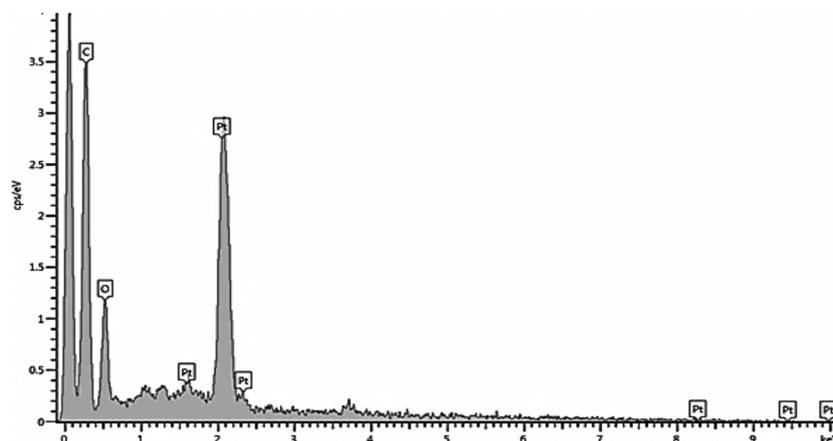C (B2) and O elements (B3), respectively

FIGURE 6(b). EDX analysis of RGO/Pt nanocomposite

of RGO which is consistent with the results of HRTEM. The intensity ratio of the 2D peak to the S3 peak for RGO and RGO/Pt nanocomposite were estimated to be 0.82 and 0.86, respectively. The different of this intensity ratio was indicating a slightly increased structural destruction of RGO and RGO/ Pt nanocomposite catalyst (Xu et al. 2014).

ELEMENTAL ANALYSIS OF RGO/ PT NANOCOMPOSITE

The elemental mapping for RGO/Pt nanocomposite in Figure 6(a) confirmed that the Pt nanoparticles were homogenously distributed and successfully formed onto RGO. The large surface area of RGO give the high uniform dispersion of Pt nanoparticles (Figure 6(a) (B1)). From EDX mapping image for over-lapping of Pt-C-O elementals (Figure 6(a) (A)), Pt nanoparticles were observed to have high tendency to be deposited onto RGO since the existence of remaining oxygen containing functional groups available in RGO that anchor the Pt nanoparticles (Figure 6(a) (B2) and Figure 6(a) (B3)). Figure 6(b) shows the EDX spectrum of RGO/Pt nanocomposite which confirmed that the Pt nanoparticles were successfully formed and deposited on RGO. The elements of C and O were also identified successfully instead of Pt elements. The C peak is attributed to the supporting film of copper mesh and RGO. The existence of O peak is due to presence of the oxygen functional groups which is present in RGO due to incomplete chemical reduction from GO to RGO.

ELECTROCHEMICAL PROPERTY OF RGO/ PT NANOCOMPOSITE

The electrochemical property of the prepared RGO/Pt nanocomposite has been performed in 0.5 M of sulfuric acid and evaluated by cyclic voltammogram (CV). Figure 7 shows the obvious hydrogen desorption peak in the potential range from -0.2 to 0.1V (vs SCE). This indicates that the Pt nanoparticles have been deposited successfully onto the RGO. The peaks in the potential ranges above 0.2 V are attributed to the oxidation of surface metal and the reduction of oxide formed.
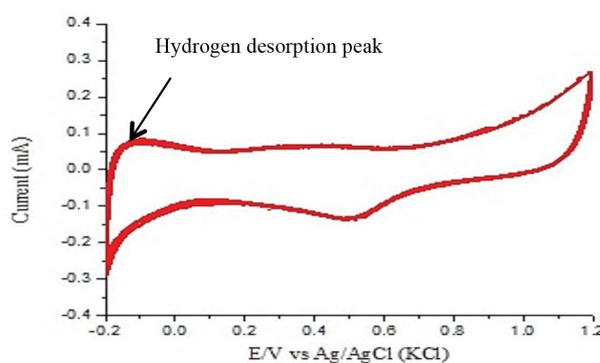

FIGURE 7. Cyclic voltammogram of RGO/ Pt nanocomposite

CONCLUSION

A simple, rapid and low cost RGO/Pt nanocomposite was fabricated using sodium borohydride as the strong reducing agent. The surface morphology of RGO/ Pt nanocomposite was performed by the HRTEM and FESEM which demonstrated the uniform dispersion of Pt nanoparticles onto RGO. The synthesized RGO/ Pt nanocomposite is valuable for scientific research in the future since it has highest potential to be used as anode catalyst in direct methanol fuel cells (DMFCs). The future work will be focused on the performance of DMFC by using RGO/Pt nanocomposite as an anode catalyst.

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