

Performance of Reduced Graphene Oxide/Iron(III) Oxide/Silica Dioxide (rGO/ Fe₃O₄/SiO₂) as a Potential Oxygen Reduction Electrocatalyst in Fuel Cell (Prestasi Grafin Oksida Terturun/Ferum(III) Oksida/Silika Dioksida (rGO/Fe₃O₄/SiO₂) yang Berpotensi sebagai Elektromangkin Penurunan Oksigen dalam Sel Bahan Api)

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ABSTRACT

Synthesis of the nanocomposite comprises reduced graphene oxide, iron (III) oxide and silica dioxide nanocomposites which were denoted as rGO/Fe₃O₄/SiO₂. The acquired nanocomposite was determined to be a substitute for platinum electrode in oxygen reduction reaction (ORR) to catalyze reaction, as usage of platinum causes disadvantages in production. The nanocomposite was analyzed physically and electrochemically to ensure the quality of the synthesized compound. Fourier transform-infrared spectroscopy (FTIR) shows the presences of functional groups such as O-H hydroxyl group, C=C, C=O and existence of silica peak in the spectra of rGO/Fe₃O₄/SiO₂, where the data is also supported by SEM-EDS. Raman Spectrophotometer shows the structural change of three different graphene related materials as modification took place and X-Ray Diffraction (XRD) analysis confirms the reduction of GO into rGO, where the crystalline structure decreased significantly approximately about 10 nm. This data supported with Brunauer-Emmett-Teller (BET) analysis through surface area examination. The compound of rGO/Fe₃O₄/SiO₂ was drop-casted onto glassy carbon electrode (GCE) for modification into rGO/Fe₃O₄/SiO₂/GCE to carry out electrochemical analysis where Cyclic Voltammetry (CV) shows current response by modified electrode is greater than bare GCE while Electron Impedance Spectroscopy (EIS) of same modified electrode affirms the sample underwent reversible process with stable and rapid electron transfers with minimal resistance charge transfer (R_{CT}). The study of ORR was carried out and observed a good electrochemical response of the nanocomposite when purged with oxygen gas.

Keywords: Graphene; magnetite; oxygen reduction reaction; silica dioxide

ABSTRAK

Nanokomposit disintesis dengan grafin oksida terturun, ferum (III) oksida dan silika dioksida yang dinamakan rGO/Fe₃O₄/SiO₂ sebagai pengganti untuk elektrod platinum tindak balas penurunan oksigen (ORR) sebagai pemangkin atas sebab kekurangan semasa reaksi. Pencirian fizikal dan elektrokimia dibuat untuk memastikan kualiti nanokomposit dijamin. Spektroskopi transformasi Fourier inframerah (FTIR) menunjukkan kehadiran kumpulan berfungsi di nanokomposit rGO/Fe₃O₄/SiO₂ seperti kumpulan hidroksi O-H, C=C, C=O dan silika. Data ini disokong oleh analisis mikroskop pengimbasan elektron (SEM). Spektroskopi Raman menunjukkan perubahan struktur untuk komposit berkenaan grafin, manakala difraksi sinar-X (XRD) menunjukkan pengecilan struktur kristal lebih kurang 10 nm, yang disokong oleh analisis penentuan kawasan permukaan Brunauer-Emmett-Teller (BET). Elektrod karbon berkaca (GCE) diubah suai menggunakan rGO/Fe₃O₄/SiO₂ dengan kaedah salutan titisan bagi pencirian elektrokimia. Kitaran voltametri (CV) dan spektroskopi elektrokimia impedan (EIS) membuktikan nanokomposit melalui proses terbalik dan stabil berserta pertukaran elektron yang pantas, dengan rintangan yang minimum. Objektif utama penyelidikan, ORR dijalankan dan didapati tindak balas elektrokimia yang bagus apabila diterapkan dengan gas oksigen, yang membuktikan nanokomposit yang disintesis dengan kos rendah boleh menggantikan platinum untuk tindak balas elektrokimia.

Kata kunci: Grafin; magnetit; silika dioksida; tindak balas penurunan oksigen

INTRODUCTION

Fuel cells received great attention over the past decades as a promising and economical renewable energy with higher energy conversion efficiency. It is a recommended technology where electrical energy was produced through conversion of chemicals. Oxygen reduction reaction (ORR) is a prominent reaction in fuel cell system, where a direct reduction of through four electron transfer occurred from O_2 to produce H_2O occurs (Muhamad & Yusoff 2018). The typical cathode electrode in the system, platinum, outputs sluggish kinetics reaction despite being the optimal material amongst other materials due to overpotential. To add on, the catalytic activity of platinum is low due to contamination of CO gases during reaction. To overcome this, Kumar et al. (2017) suggested the use of bifunctional catalyst in fuel cells to enhance the electrocatalytic activity and facilitate electron transfer in slow reactions.

To date, variety of compounds have been proposed through previous studies to find inexpensive and impressive activity electrocatalyst as a substitute to platinum in the system (Rameshkumar et al. 2015). Amongst all materials, graphene-based compounds been extensively researched as possible substitutes for its unique properties (Lee et al. 2015). Graphene have properties such as sp^2 hybridization, larger surface area and good conductor of electricity (Deng et al. 2008; Liu et al. 2013; Yang et al. 2009). In low overpotential condition, graphene assist electron transfer between molecule to an electrode through its carbon edges electrochemically (Kui et al. 2012). Chemical reduction of graphene oxide makes the substance to exist as a compound in between graphene and graphene oxide, in other words having the compound defect-less while retaining its unique properties (Gupta et al. 2017). The step is also important to reduce the surface area of GO from macropore to mesopore. Macropore material are not suitable for electrochemistry as agglomeration occurs. Reducing graphene into rGO by removing functional groups makes the material stronger, increases active sites, enlisting the material into mesopore region and restores its electronic properties (Schöche et al. 2017).

Nanoparticles of metal elements with magnetic force; commonly iron oxides (Fe_3O_4) chosen for suitable substitution studies for its simple synthesis and economical method. Previous studies using rGO combined with Fe_3O_4 as a bifunctional electrocatalyst unleashed excellent performance of the material as the cathode catalyst for ORR reactions (Yusoff et al. 2020). Iron oxide possesses strong magnetic properties which makes it useful in various applications (Krishna et al.

2016). The magnetism aids the electron transfer especially in O_2 reduction in fuel cells as oxygen has paramagnetic properties. However, Fe_3O_4 compound alone could not be the electrocatalyst as it need to go through many processes and surface modification before being introduced into application, however, do not ensure the capability to perform better as platinum (Zhu et al. 2016). Hence, combination of these two nanoparticles aids the research with enhanced stability of Fe_3O_4 facilitated by conductive properties of carbon with greater biocompatibility (Hassan et al. 2017).

The strength of combined nanoparticles can be further boosted through addition of another material. This material acts as a protective layer of the nanoparticles while increasing the surface area a little. Choice of apt element with the nanoparticle is important to ensure the excellency of characteristics and longevity of the electrocatalyst while holding better stability in terms of thermal and conditioning. Silica dioxide (SiO_2) in this research formed an upgraded electrocatalyst with good bond on the graphene sheets. Silica was proven to be low in acidity, which can be a good carrier of iron particles (Olcese et al. 2012). Alam et al. (2009) quoted that complications such as agglomeration of magnetic induced nanoparticles could be overcome by fabricating nanoparticles with porous material support, such as silica. With this, agglomeration could be inhibited through the increase in surface area and pore diameter as well as improved stability. Low electronegativity of silica atoms driven the material to react vigorously in oxygen saturated atmosphere (Chen et al. 2013).

In this paper, successfully synthesized novel nanocomposite rGO/ Fe_3O_4 / SiO_2 as cathode catalyst for fuel cell was discussed. The significance of this study was the capability of nanocomposite to substitute platinum and yield the optimal level of production, while reducing the pollutions and economically inexpensive. The nanocomposite has been applied into ORR and showed better responses towards the reduction of oxygen.

MATERIALS AND METHODS

REAGENTS AND CHEMICALS

Graphite powder was used for synthesis of GO through oxidation process based on the modified Hummers' method (Hummers & Offeman 1958). Ferrous chloride and ferric chloride were used in production of magnetic nanoparticle while tetraethyl orthosilicate (TeOs) for SiO_2 particles. For diluting and washing the substances, deionized water of $18.2\text{ m}\Omega\text{cm}$ was used. All chemicals were purchased from Sigma Aldrich, USA.

SYNTHESIS OF NANOPARTICLE $rGO/Fe_3O_4/SiO_2$

Production of $rGO/Fe_3O_4/SiO_2$ nanocomposite was done through facile chemical method. GO was first synthesized by adding approximately 1.0 g graphite powder with sodium nitrate ($NaNO_3$) into concentrated sulfuric acid (H_2SO_4) in volumetric flask under ice bath. Potassium permanganate ($KMnO_4$) was then added gradually into the solution and diluted with 45 mL of deionized water. The solution was filtered after 3 h and dried under vacuum at room temperature. Using the GO powder obtained, rGO/Fe_3O_4 was done through one-pot synthesis where around 0.9 g of GO was added into 450 mL of deionized water. Aqueous solution of ferric chloride (Fe^{3+}) and ferrous chloride (Fe^{2+}) was prepared in a molar ratio of 2:1 in another different beaker and gradually added into GO solution at room temperature. The solution was then heated at 90 °C with 10 mL of hydrazine hydrate (N_2H_4) with constant stirring for 4 h. Hydrazine hydrate was added for reduction of GO into rGO by eliminating excess O-H functional groups in the compound. The mixture was then cooled to room temperature and filtered using a magnet. The precipitate was washed with deionized water several times to neutralize the pH and dried at 70 °C for 12 h. The powder then added into a beaker diluted in ratio of 1 g for 100 mL with added 3 mL of tetraethyl orthosilicate (TEOs). The solution was stirred for 24 h and filtered using a magnet.

INSTRUMENTAL CHARACTERIZATION

Physical characterization was carried out using FTIR to identify the functional groups in nanocomposite, Raman spectrophotometer to observe structural changes in graphene materials, XRD analysis to determine the crystalline structure, BET to observe the surface and SEM-EDX to identify the morphology of nanocomposite.

Electrochemical characterization was carried out to acquire information regarding catalytic activity of the synthesized nanocomposite in reduction process through ORR. To add on, cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) was done using potentiostat galvanostat to observe the electrochemical efficiency and electron mass transfer between electrolyte and electrode surface, respectively.

RESULTS AND DISCUSSION

PHYSICAL CHARACTERIZATION

FTIR as portrayed in Figure 1(a) identified the functional group existed in the synthesized nanocomposite. The weakened hydroxyl (O-H) functional group was found at peak 3400 cm^{-1} attributed from adsorbed water by graphene oxide sheets during Hummers' method. Presence of other peaks (Papiya et al. 2017). The C=C stretching peak was identified at 1625 cm^{-1} while peak of Fe and Si were observed at 580 cm^{-1} and 1100 cm^{-1} , respectively. Appearances of expected functional groups in $rGO/Fe_3O_4/SiO_2$ indicated the nanocomposite managed to be synthesized as desired.

Raman spectrophotometer in Figure 1(b) shows two distinct peaks for the test; D band at 1351 cm^{-1} of sp^3 vibration and G band at of 1570 cm^{-1} for vibration of sp^2 carbon hybridization (Yusoff et al. 2020). Presence of G band proposed the elongation of sp^2 C-C bonds in the nanocomposite. Intensity ratio was studied through I_D/I_G formula and found to be 0.87 which proved the graphitic structure of the nanocomposite (Zhang et al. 2000). Peak 410 cm^{-1} in spectrum of $rGO/Fe_3O_4/SiO_2$ marked the existence of silica crystalline in the nanocomposite. Figure 1 shows the results of analysis for FTIR, Raman and XRD. These findings supported data of FTIR and were assisted in further analyses.

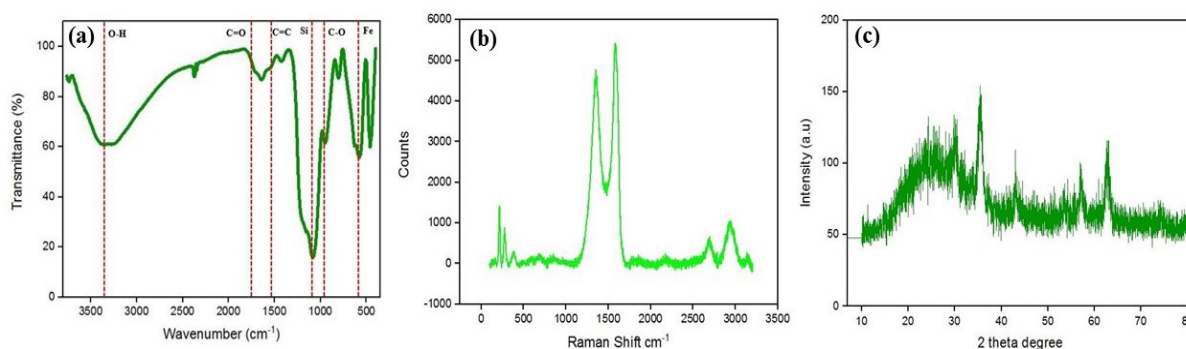


FIGURE 1. (a) FTIR, (b) RAMAN, and (c) XRD of $rGO/Fe_3O_4/SiO_2$

The data of XRD analysis as portrayed in Figure 1(c), observed a total of five peaks: at 25, 31, 43, 57, and 64°. The intensity of rGO/Fe₃O₄/SiO₂ increased proportional to the presence of silica. The average crystalline size of the nanocomposite and interlayer *d*-spacing was found to be 8.84 and 0.348 nm, respectively, using Scherrer Equation (1) and Bragg's Law (2).

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

$$d = \frac{\lambda}{\beta \sin\theta} \quad (2)$$

The crystalline size confirmed that the nanocomposite categorized in mesopore region of nanoparticle classification while interlayer spacing indicated the aggregation of composites is well controlled (Marcano et al. 2010). The calculated value supported obtained results of BET as follows.

Results of BET analysis was tabulated in Table 1. The nanocomposites comparison showed the surface area for rGO/Fe₃O₄/SiO₂ is greater compared to its pioneer composite rGO/Fe₃O₄ where the introduction of silica dioxide increased the surface area. The rGO/Fe₃O₄/SiO₂ possessed a significant 151.04 m²/g of surface area compared to rGO/Fe₃O₄ with 124.41 m²/g of surface area. GO possessed highest surface area of 304.43 m²/g, however, categorized as macropores with pore volume of 1.43 cm³/g which is about 143 nm when the Barrett-Joyner-Halenda (BJH) pore volume were studied. The rest of synthesized nanoparticles falls into mesopore region in the range of 0.1 to 0.3 cm³/g indicated the nanocomposite was less porous. In electrochemistry, macropores were not recommended as disruption of electron transfer at electrode surface might occur (Alam et al. 2009; Iwan et al. 2017).

TABLE 1. Surface area and pore volume of synthesized composites

Sample	BET surface area (m ² /g)	BJH pore volume (cm ³ /g)
GO	304.43	1.43
rGO/Fe ₃ O ₄	124.41	0.26
rGO/Fe ₃ O ₄ /SiO ₂	151.04	0.16

SEM was used to analyze surface morphology of the nanocomposite synthesized as portrayed in Figure 2(a). Composite rGO/Fe₃O₄/SiO₂ confirmed the well dispersed of Fe₃O₄ and SiO₂ on the nanocomposite indicating the compound was easily grafted on defected-sites of rGO due to less agglomeration. TEM in Figure 2(b) exhibited the transparency of rGO/Fe₃O₄/SiO₂ nanocomposite was

lower and filled with darker spots which explained the dispersy of Fe₃O₄ and SiO₂ onto sheet of rGO (Krishna et al. 2016). EDX in Figure 2(c) confirmed the existence of all compounds proposed for this nanocomposite with its mass and atomic composition tabulated in Table 2. The findings supported the fact of increased surface area as discussed in BET earlier.

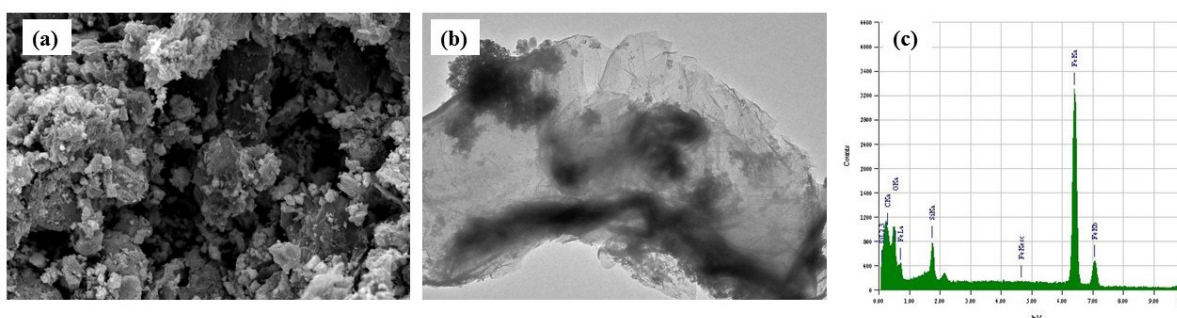


FIGURE 2. (a) SEM, (b) TEM, and (c) EDX of rGO/Fe₃O₄/SiO₂

TABLE 2. EDX data of mass and atomic composition of rGO/Fe₃O₄/SiO₂

Element	Mass (%)	Atomic (%)
C	12.95	13.13
O	4.06	10.07
Fe	76.56	68.39
Si	6.43	8.41

ELECTROCHEMICAL CHARACTERIZATION

Electrochemical efficiency of the synthesized nanocomposite was studied through CV and EIS with 5.0 mM K₄[Fe(CN)₆] as electrolyte. GCE was used as bare electrode before being modified by depositing nanocomposites such as GO, rGO/Fe₃O₄ and rGO/Fe₃O₄/SiO₂ through drop-cast method. The scan rate used for cyclic voltammetry analysis was 100 mV. The cyclic voltammogram in Figure 3(a) shows rGO/Fe₃O₄/SiO₂/GCE possessed significant redox responses compared to bare GCE and other electrocatalyst-deposited electrode, pointing the drop cast facilitated diffusion of ions onto the electrode surface. Electrode rGO/Fe₃O₄/SiO₂/GCE exhibited outstanding redox current with broadest peak possessing I_{pa} and I_{pc} values of 101.2 and 88.4 μ A, respectively, compared to bare GCE (37.4 and 38.6 μ A) and rGO/Fe₃O₄/GCE with values of 87.1 and 61.7 μ A. Peak potential separation ($\Delta E_p = E_{\text{Anodic peak}} - E_{\text{Cathodic peak}}$) of rGO/Fe₃O₄/SiO₂/GCE was at $\Delta E_p = 100$ mV as tabulated in Table 3, suggesting that the electron transfer is rapid and stable with the reversible process (Nicholson 1965). The effect of scan rate assisted to identify the reaction kinetics of rGO/Fe₃O₄/SiO₂/GCE in range of 50 to 200 mV/s. Figure 3(b) observed the redox increased progressively proportional

to current potential. The square root of scan rate obtained based on the range applied earlier. Regression of both oxidation and reduction plots were merely equal to 1 (0.994 and 0.996 for anode and cathode, respectively), and the shift proved the redox reaction of rGO/Fe₃O₄/SiO₂/GCE was quasi reversible (Wipf et al. 1988).

EIS experimented the electron mass transfer between the electrolyte and electrode surface. The charge transfer resistance (R_{ct}) which symbolizes the kinetic resistance of charge transfer of redox reaction decreased (1173.24 Ω) as the bare GCE electrode was deposited with electrocatalyst from GO to rGO/Fe₃O₄/SiO₂ on GCE. R_{ct} portrayed as semicircle in Nyquist plot in Figure 3(c). Modified electrode rGO/Fe₃O₄/SiO₂/GCE possessed incomplete and discrete semicircle compared to bare GCE electrode, hence proved that charge transfer resistance was greater in bare GCE compared to electrocatalyst deposited electrodes (Muhamad & Yusoff 2018). Lower R_{ct} linked with conductivity capability and smoother charge transfer in the electrolyte medium. Apparent electron transfer rate constant, K_{app} was calculated for rGO/Fe₃O₄/SiO₂/GCE and obtained rate constant of 9.71×10^{-4} cm s⁻¹, indicating higher electron transfer rate process strengthen the points discussed earlier (Yusoff et al. 2013).

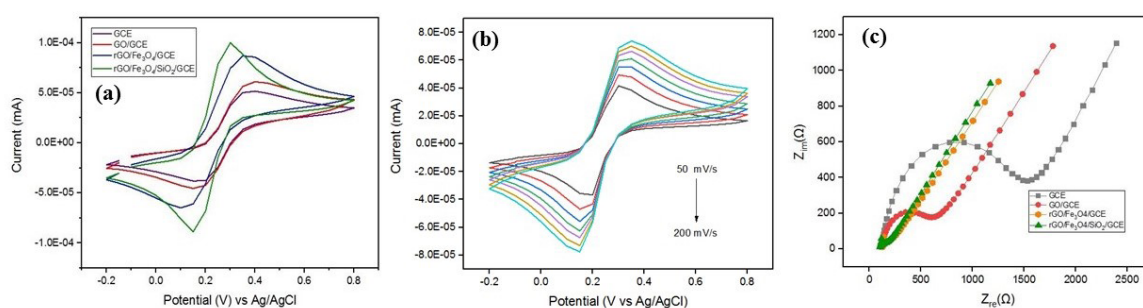


FIGURE 3. (a) CV of bare GCE and nanocomposite modified electrode, (b) effect of scan rate of rGO/Fe₃O₄/SiO₂/GCE, and (c) EIS of bare GCE and modified electrode

TABLE 3. Square root of scan rate and peak height of rGO/Fe₃O₄/SiO₂/GCE

Scan rate (mV/s)	Square root of scan rate (v ^{1/2})	Peak current at anode (I _{pa})	Peak current at cathode (I _{pc})	R ² (I _{pa})	R ² (I _{pc})	ΔE
50	7.07	0.0416	-0.0367			100
75	8.66	0.0495	-0.0469			100
100	10.00	0.0552	-0.0558	0.994	0.996	100
125	11.18	0.0611	-0.0627			100
150	12.24	0.0664	-0.0675			100
175	13.23	0.0702	-0.0732			100
200	14.14	0.0739	-0.0776			100

OXYGEN REDUCTION REACTION (ORR) ANALYSIS

The study of ORR acquired from Figure 4(a) shows cyclic voltammogram of the modified electrode rGO/Fe₃O₄/SiO₂/GCE in ORR which was studied under condition of 0.1 M of potassium hydroxide (KOH) solution as electrolyte. The electrolyte was prepared in two sets and purged with N₂ and O₂ each for 10 min. It can be concluded that the fabrication of GCE with the nanocomposite increased the kinetic energy of the reaction as portrayed by potential peaks, indicated that the modified electrode

is viable and well adapted to the reaction process. The rGO/Fe₃O₄/SiO₂/GCE modified electrode showed better performances compared to other modified electrode. A positive shift was observed stipulating higher and better current density of the rGO/Fe₃O₄/SiO₂/GCE electrode compared to rGO/Fe₃O₄/GCE electrode (Lee & Kim 2017). The voltammogram of Figure 4(c) shows N₂ gas purged electrolyte showed no peak indicating no redox reaction occurred in that condition, while a peak was observed in O₂ purged electrolyte.

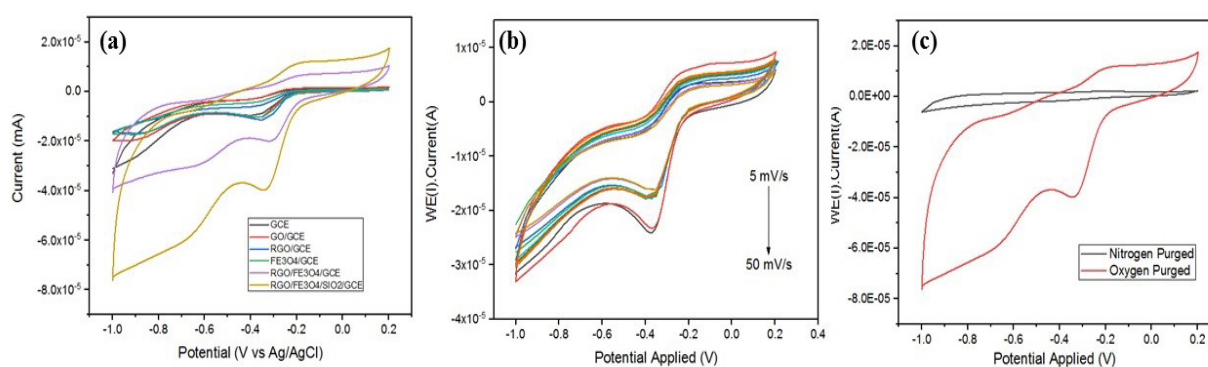


FIGURE 4. (a) ORR for bare GCE with nanocomposite modified GCE electrodes, (b) effect of scan rate of rGO/Fe₃O₄/SiO₂/GCE, and (c) comparison of O₂ and N₂ purged in electrolyte for rGO/Fe₃O₄/SiO₂/GCE

CONCLUSION

In conclusion, the novel synthesis of rGO/Fe₃O₄SiO₂ nanocomposite have been done successfully through inexpensive and facile chemical method. It was proven through acquired experimental results that the nanocomposite can be a promising electrocatalyst to substitute the platinum-based electrode in commercial usages. The surface area has been confirmed with BET and SEM, where 151.04 m²/g has been recorded with BET analysis, which was proven by SEM. With lower resistance at 1173.24 Ω, the electrochemical capacitance was at optimum level, hence proved the enhanced kinetics for ORR electrochemical reactions. ORR analysis shows positive shift hence proving better current density. Larger scale production of this nanocomposite could be better option towards renewable energy industries for sustainable economy and environment.

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