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$PtCoNb_2O_5$/graphene as electrocatalyst towards oxygen reduction reaction in alkaline and acidic media

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The aim of the study was to prepare and to investigate the catalytic activity of the Nb_2O_5/graphene-supported platinum–cobalt catalyst for the oxygen reduction reaction (ORR) in alkaline and acidic media. Electrocatalytic properties of the prepared catalyst towards the oxygen reduction reaction were characterized by using cyclic voltammetry (CV) and rotating disk electrode (RDE) linear sweep voltammetry (LSV).

It has been found that the $PtCoNb_2O_5$/graphene catalyst shows more positive onset potentials, as well as higher current in the mixed-kinetic-diffusion region towards the oxygen reduction reaction in both alkaline and acidic solutions as compared with those for the bare Pt/GR catalyst.

Keywords: platinum–cobalt, niobium(V) oxide, electrocatalysts, oxygen reduction

INTRODUCTION

In this era there is crucial development of technologies that creates sustainable, highly efficient and, of course, green energy resources because of energy crisis and global warming [1]. So, this is the reason why fuel cells are one of the best alternative energy sources – they can convert chemical energy into electrical energy and do not contaminate the environment. Fuel cells are promising energy conversion devices that directly generate energy without an intermediate mechanical link.

One of the three basic components of a fuel cell is a cathode, where the oxygen reduction reaction takes place. Up to now, the most popular and most commonly used catalysts for the oxygen reduction reaction are platinum and platinum based materials. They are known as the most active catalysts for the ORR, although a high cost of platinum is their scarcity [2]. That is the main reason why lowering the amount of noble metals in catalysts is the general interest of researches. Carbon and graphene based materials as catalysts are known for many advantages, such as light weight, high electrical conductivity and low cost materials [2].

Pt alloys for fuel cells have been studied since 1970. The reason was the increase of platinum activity [3]. In addition to this, Pt-based alloys with metals, such as Ni, Fe, Co, Cr, Cu etc., often demonstrate higher electrocatalytic activity towards the ORR than pure platinum [4–8]. The increase of catalysts activity in the ORR is explained by the decrease of the distance between platinum surface atoms, and the increase of d-electron vacancy of the platinum surface [3]. Enhancement of ORR kinetics is the result of a significant decrease of the Gibbs free energy for the electron transfer steps in the ORR, caused by the d-orbital coupling of two metals in the binary catalyst [9]. A. Schenk et al. compared the behaviour of PtCo/C and Pt_3Co/C catalysts and found that the PtCo catalyst exhibited higher activity in the oxygen reduction reaction [10]. Alloys of Pt with 3d transition metals attract more attention, because they decrease the amount of Pt and increase the electrocatalytic activity in a fuel cell element.

It was proved that metal oxides, such as TiO_2, Nb_2O_5, CeO_2, V_2O_5, Mn_xO_y etc., can be relatively inexpensive supplementary

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materials for the use for Pt catalysts in fuel cells [11–14]. Studies have shown that the addition of a second metal, such as Ce, Nb, etc., upgrades the activity of CO oxidation. It is based on the size of particles, metal and support metal interface, morphology and electronic effects [15]. It is known that Nb_2O_5 interacts with the other metals in the catalyst, has a feature to reduce and has oxygen storage capacity, exhibits well chemical stability, it is not harmful and can be applied in industry [16].

In our previous study it was identified that the Nb_2O_5/graphene-supported platinum–cobalt catalyst shows enhanced electrocatalytic activity towards the oxygen reduction reaction in an acidic solution (by using a stationary state electrode) [17] as well as towards oxidation of alcohols [18].

This paper reports the experimental characterization of Nb_2O_5/graphene-supported platinum–cobalt catalysts (denoted $PtCoNb_2O_5$/GR) synthesized by using a fast and simple microwave irradiation method. The composition, morphology and structure of the synthesized catalysts were determined by means of inductively coupled plasma optical emission spectroscopy (ICP-OES), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD). Electrocatalytic properties of the prepared catalyst were characterized towards the oxygen reduction reaction in a 0.5 M H_2SO_4 and 0.1 M NaOH solution by using a rotating disk electrode. For comparison, the graphene supported platinum catalyst (denoted as Pt/GR) was synthesized and characterized in the same manner.

EXPERIMENTAL

Chemicals

H_2PtCl_6 (99.9%), $CoCl_2 \cdot 6H_2O$ (98%) and Nb_2O_5 powder (99.9%) were purchased from Alfa-Aesar and Sigma-Aldrich. Graphene (GR) (purity 97%, specific surface area 60 $m^2\ g^{-1}$) was purchased from Graphene Supermarket. Polyvinylidenefluoride N-metil-2-pirolidone, ethylene glycol (EG), acetone, H_2SO_4 (96%) and NaOH (98.8%) were purchased from Chempur. Oxygen (99.999%) was used for the saturation of H_2SO_4 solution. All chemicals were of analytical grade. Ultra-pure water with a resistivity of 18.2 $M\Omega\ cm^{-1}$ was used to prepare all the solutions.

Preparation of catalysts

By carrying out our experimental studies, we have synthesized the $PtCoNb_2O_5$/GR catalyst, that showed enhanced catalytic activity in both oxidation and reduction reactions for fuel cells. The $PtCoNb_2O_5$/GR catalyst was prepared by means of microwave synthesis using the polyol method [18]. A typical preparation consists of the following steps: 1) dry niobium(V) oxide/graphene powder was mixed with graphene powder (mass ratio being 1:1) in a 2-propanol solution and sonicated for 30 min, with further desiccation of the mixture; 2) 20 ml of an ethylene glycol solution contains 0.25 ml of 0.096 M H_2PtCl_6 and 0.6 ml of the calculated concentration of $CoCl_2$ solution was prepared. The pH of the solution was adjusted to 11.7 by adding dropwise a 1 M NaOH solution, then 100 mg of the as-prepared Nb_2O_5/GR powder was added to the mixture and sonicated for 30 min. For the microwave irradiation a Monowave 300 microwave reactor (Anton Paar) was used. The reduction of PtCo nanoparticles was carried out at a temperature of 170°C for 30 min. For comparison, the graphene supported Pt catalyst was also prepared in the same manner by heating of an EG solution with Pt(IV) salt at 170 °C for 30 s. With the aim to have approximately a similar size of Pt particles in the created $PtCoNb_2O_5$/GR and Pt/GR catalysts, the duration of Pt/GR preparation was shortened to 30 s. After the preparation, the synthesized catalysts were washed with acetone, ultra-pure water, then filtered and dried in a vacuum oven at 80°C for 2 h.

Characterization of catalysts

The Pt and Co metal loadings were estimated by means of inductively coupled plasma optical emission spectroscopy (ICP-OES). The ICP optical emission spectra were recorded using an ICP optical emission spectrometer Optima 7000 DV (Perkin Elmer).

The shape and size of catalyst particles were examined using a transmission electron microscope Tecnai G2 F20 X-TWIN equipped with an EDAX spectrometer with an r-TEM detector. For microscopic examinations, 10 mg of the sample was first sonicated in 1 ml of ethanol for 1 h and then deposited on the Cu grid covered with a continuous carbon film.

The phases and lattice parameters of the synthesized catalysts were characterized by powder X-ray diffraction using a D8 diffractometer (Bruker AXS, Germany, 2003) with Cu Kα radiation using a Ni/graphite monochromator. A step-scan mode was used in the 2-theta range from 20 to 90° with a step length of 0.02° and a counting time of 5 s per step.

Electrochemical measurements

All electrochemical measurements were assessed in a conventional 80 ml three-electrode cell using a rotating disc electrode and the AUTOLAB electrochemical workstation. The working electrode was a glassy carbon RDE with a geometric area of 0.07 cm^2. A Pt sheet was used as a counter electrode and an Ag/AgCl/KCl electrode was used as a reference. The catalyst layer was obtained according to the following steps: at first, the required amounts of the investigated catalysts were dispersed ultrasonically for 1 hour in a 2% polyvinylidenefluoride N-metil-2-pirolidone (PVDF) solution. Then 3 μl of the prepared suspension mixture was pipetted onto the polished surface of a glassy carbon electrode and dried in an oven at 80°C for 4 h.

The cyclic voltammetry (CV) measurements were carried out in a 0.5 M H_2SO_4 solution. The potential was cycled in a range of 0–1.5 V with a scan rate of 50 $mV\ s^{-1}$.

For the ORR measurement in acidic media a 0.5 M H_2SO_4 solution was used. In alkaline conditions an electrolyte was a 0.1 M NaOH solution. For the ORR experiments the electrolytes were purged with O_2 for 10 min prior to each ORR test. The RDE linear sweep voltammetry curves were recorded from 1 to 0.1 V in the cathodic direction at 5 mV s^{-1}, varying the rotation speed from 0 to 2000 rpm.

Koutecky–Levich (K–L) equations (1–3) were used for calculation of the electron transfer number per oxygen molecule (*n*) in ORR:

$$j^{-1} = j_k^{-1} + j_d^{-1}, \tag{1}$$

$$j_d = 0.62\mathrm{nFD}^{2/3}\,\mathrm{Co}_2\mathrm{V}^{-1/6}\omega^{1/2}, \tag{2}$$

$$j^{-1} = j_k^{-1} + w^{-1/2} \times \mathrm{A}. \tag{3}$$

Here, j, j_k and j_d are the measured current density, kinetic current density and the diffusion limiting current density (mA cm^{-2}), respectively; n is the number of electrons transferred in the reaction; F is the Faraday constant; D is the diffusion coefficient of the reactant; Co_2 is the concentration of the reactant in the bulk electrolyte; V is the kinetic viscosity of the electrolyte; w is the rotation rate; A is the slope of the linear plot of j^{-1} vs $w^{-1/2}$ (K–L plot) [22, 23].

All measurements were carried out at a temperature of 25°C. The electrode potential is quoted vs the reversible hydrogen electrode (RHE). The presented current densities are normalized with respect to the geometric area (0.07 cm^2) of the working electrode.

RESULTS AND DISCUSSION

Due to the known PtCoNb$_2$O$_5$/GR electrocatalytic activity towards the oxygen reduction reaction in an acidic solution by using a stationary state electrode [17], in this paper we report on the electrocatalytic activity of the PtCoNb$_2$O$_5$/GR catalyst towards the oxygen reduction reaction in acidic and alkaline media by using a rotating disk electrode.

The PtCoNb$_2$O$_5$/GR and Pt/GR catalysts with the Pt nanoparticles in size of ca. 1.5 and 5 nm, respectively, were synthesized according to [18].

X-ray diffractograms for the studies of PtCoNb$_2$O$_5$/GR and Pt/GR catalysts are presented in Fig. 1. The pattern of the Pt/GR catalyst (Fig. 1a) shows peaks corresponding to Pt. The composition of the latter catalyst consists of small Pt (PDF 4-802) crystallites of ca. 4–5 nm in size. In the case of the PtCoNb$_2$O$_5$/GR catalyst the crystallites of Pt are very small (ca. 1–2 nm according to TEM [18]), so they were not detected by XRD (Fig. 1b). Niobium(V) oxide is predominant in a orthorhombic (PDF 27-1003) and a monoclinic (PDF 37-1468) form with particles of ca. 9 nm in size. The crystallite size of the as-prepared catalysts was determined using the Sherer's equation and values of the full width at half maximum of XRD peaks [19].

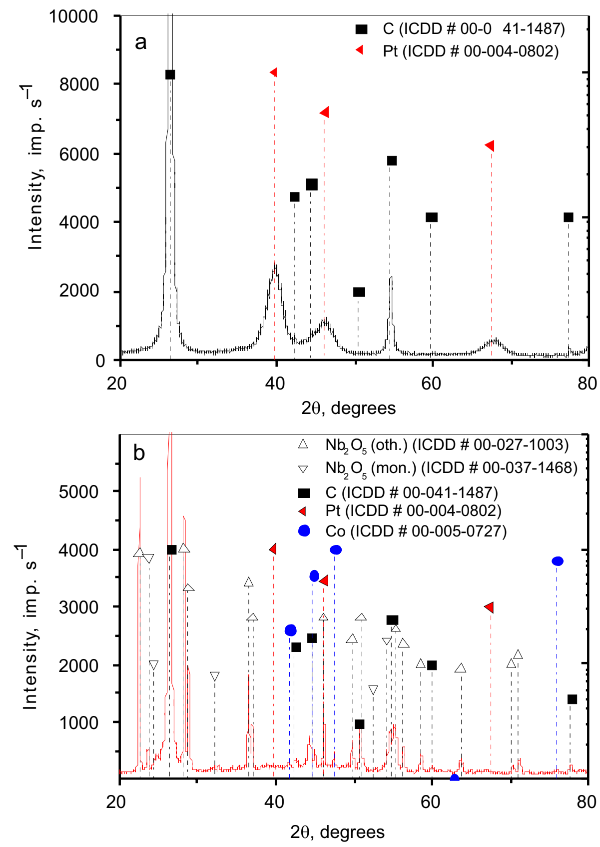

Fig. 1. XRD diffractograms of the Pt/GR (a) and PtCoNb$_2$0$_5$/GR (b) catalysts

The amount of platinum for each catalyst was determined by ICP-OES. The estimated Pt loading is equal to 0.171 and 0.086 mg Pt cm^{-2} in the synthesized PtCoNb$_2$0$_5$/GR and Pt/GR catalysts, respectively.

The electrochemically active surface area (ESA) of the catalyst is one of the important parameters to determine electrocatalytic properties of the investigated catalysts. ESA values of Pt in the synthesized catalysts were determined from the cyclic voltammograms recorded in an argon deaerated 0.5 M H_2SO_4 solution at a sweep rate of 50 mV s^{-1} (Fig. 2) and further calculation of the integrated charge of the hydrogen adsorption region (Q_H) in the cyclic voltammograms and calculated according to Eq. (4) [20]

$$\mathrm{ESA\ (cm^2)} = \mathrm{Q_H\ (\mu C)\ /\ 210\ (\mu C\ cm^{-2})}, \tag{4}$$

where 210 μC cm^{-2} is the charge required to oxidize a monolayer of hydrogen adsorbed on Pt.

In our case, the calculated ESA values were equal to 3.8 and 1.4 cm^2 for the PtCoNb$_2$O$_5$/GR and Pt/GR catalysts, respectively.

According to the Pt loading of each catalyst, the specific ESA values (m^2 g^{-1}) were calculated according to Eq. (5) and equal

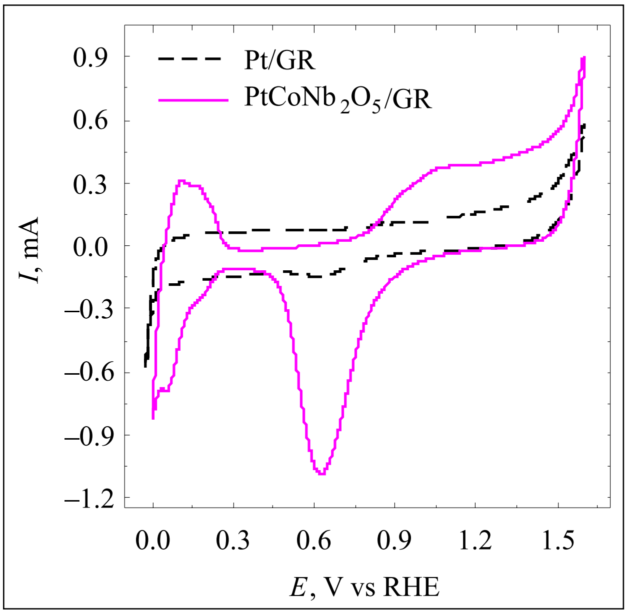

Fig. 2. Cyclic voltammograms of Pt/GR (a dashed line) and PtCoNb$_2$O$_5$/GR (a solid line) recorded in 0.5 M H_2SO_4 at 50 mV s^{-1}

32 and 23 m^2 g^{-1} platinum, respectively, for PtCoNb$_2$O$_5$/GR and Pt/GR:

$$\text{ESA } (m^2 g^{-1}) = Q_H/\text{Pt loading} \times 210. \quad (5)$$

To evaluate the ORR activity of PtCoNb$_2$O$_5$/GR and Pt/GR, LSV measurements were carried out in alkaline and acidic media in O_2-saturated 0.1 M NaOH and 0.5 M H_2SO_4 solutions. The reduction of oxygen was investigated for the mentioned catalysts electrochemically pretreated in 0.5 M H_2SO_4 at 50 mV s^{-1} for 30 min. For the LSV measurements the potential was scanned from ca. 1.0 V in the cathodic direction to 0.1 V vs RHE in acidic media and from ca. 1.0 to 0.4 V in alkaline media at a scan rate of 5 mV s^{-1}, varying the rotation speed from 0 to 2000 rpm. Figure 3 presents the ORR polarization curves on the PtCoNb$_2$O$_5$/GR and Pt/GR catalysts in acidic media (Fig. 3a, b) and in alkaline media (Fig. 3c, d). From Fig. 3 it is seen that in both alkaline and acidic media the ORR polarization curves have a typical view accordingly to the literature where it is described that at the potential between 0.9 and 0.7 V vs RHE

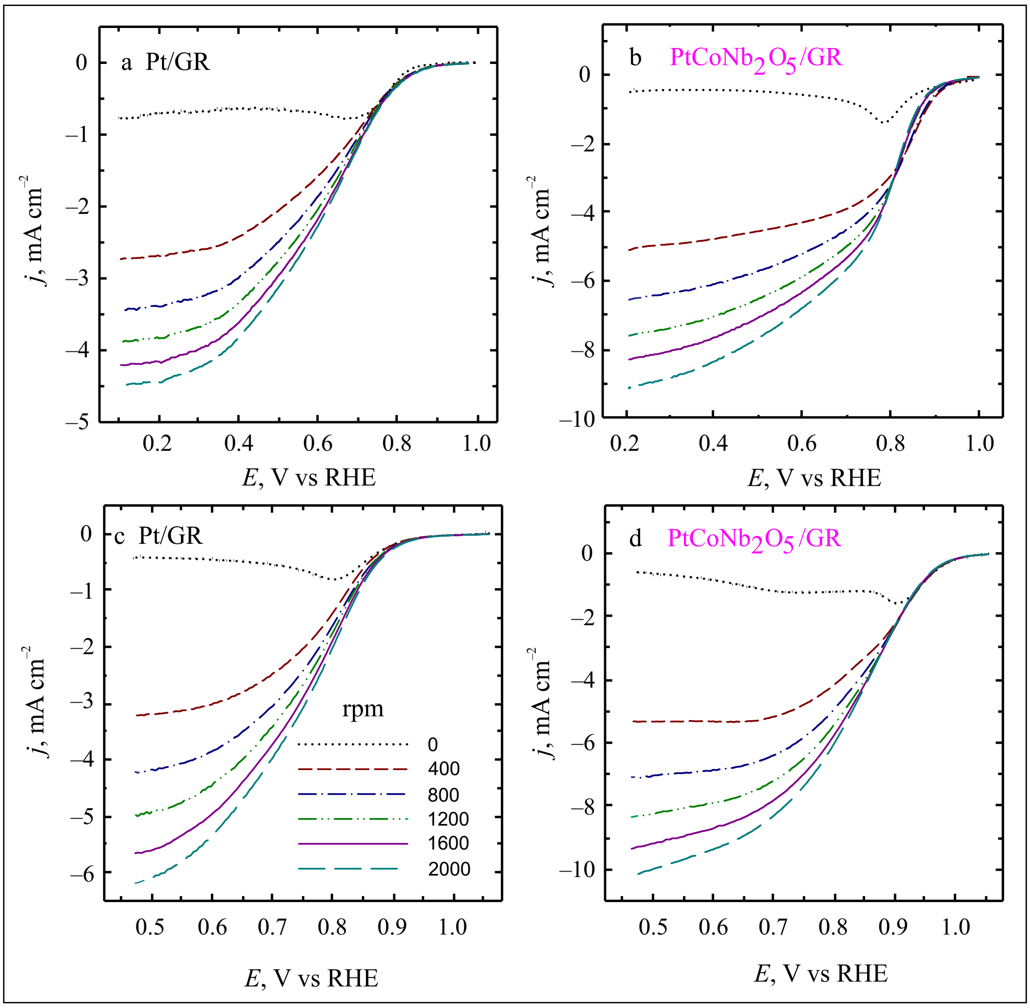

Fig. 3. Linear sweep voltammetry scans for Pt/GR and PtCoNb$_2$O$_5$/GR recorded at 5 mV s^{-1} in O_2-saturated 0.5 M H_2SO_4 solution (a, b) and O_2-saturated 0.1 M NaOH solution (c, d)

the ORR response is dominated by the kinetics of electrocatalysts [21], as well as the increase of ORR current densities with increasing of the rotating rate. It is seen that the onset of the ORR and the half-wave potential were significantly shifted to more positive potentials in the case of the $PtCoNb_2O_5$/GR electrode, indicating its higher catalytic activity for the reduction of oxygen, compared to that of Pt/GR (Fig. 3). The better ORR performance of $PtCoNb_2O_5$/GR could be attributed to the synergetic action between the metals present in the catalyst composition.

After the LSV measurements at the rotating speed from 400 to 2000 rpm, Koutecky–Levich plots were obtained at the potential from 0.4 to 0.8 V vs RHE. Figure 4 presents the K–L plots in O_2-saturated 0.5 M H_2SO_4 (Fig. 4a, b) and in O_2-saturated 0.1 M NaOH (Fig. 4c, d) solutions. In both cases the K–L curves show a linear relationship between j^{-1} and $w^{-1/2}$. According to the K–L equations (1–3) calculated the number of electrons transferred (n) varied from 3.5 to 4.2, indicating the 4 e^- transfer reaction and the direct reduction of O_2 to H_2O on the surfaces of the Pt/GR and $PtCoNb_2O_5$/GR catalysts.

Figure 5 presents the ORR polarization curves of the Pt/GR and $PtCoNb_2O_5$/GR catalysts recorded in O_2-saturated 0.5 M H_2SO_4 (Fig. 5a) and in O_2-saturated 0.1 M NaOH (Fig. 5b) solutions at 1600 rpm. As evident from Fig. 5, the $PtCoNb_2O_5$/GR catalyst shows higher onset potentials, as well as higher ORR current density in the mixed-kinetic-diffusion region (Fig. 5a, b) in both acidic and alkaline

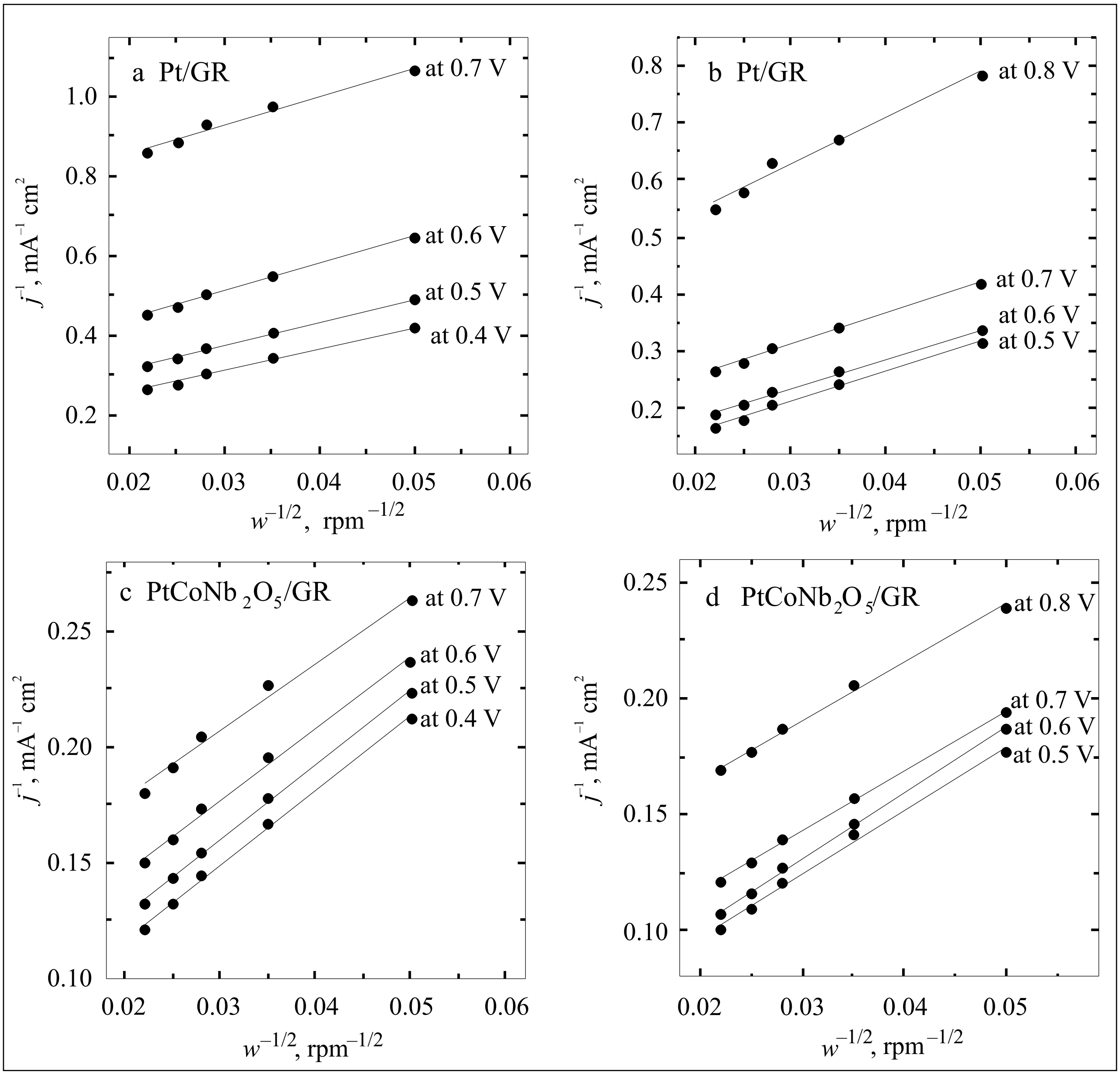

Fig. 4. Koutecky–Levich plots of the Pt/GR and $PtCoNb_2O_5$/GR catalysts on different potentials in O_2-saturated 0.5 M H_2SO_4 solution (a, c) and O_2-saturated 0.1 M NaOH solution (b, d)

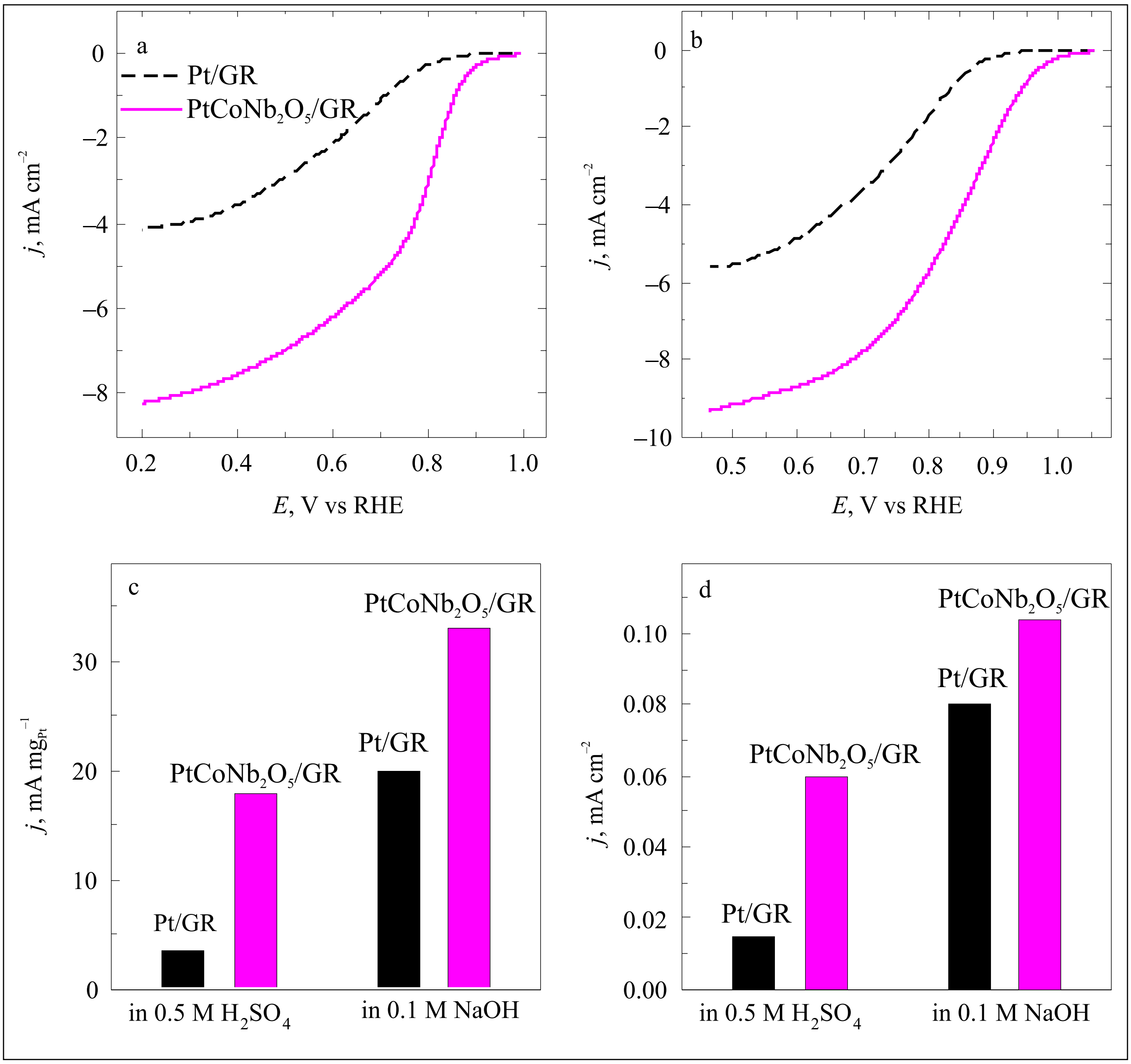

Fig. 5. Comparison of LSV curves at 1600 rpm recorded in O_2-saturated 0.5 M H_2SO_4 solution (a) and O_2-saturated 0.1 M NaOH solution (b); mass (c) and specific (d) activities at 0.8 V for the Pt/GR and $PtCoNb_2O_5$/GR catalysts

media as compared with that at Pt/GR. With the aim of comparison, the oxygen reduction current densities at a potential value of 0.8 V were normalized by the Pt loadings and electrochemically active surface areas for each catalyst in acidic and alkaline media to represent the mass activity (mA mg_{Pt}^{-1}) (Fig. 5c) and the specific activity (mA cm^{-2}) (Fig. 5d), respectively. As seen from the data obtained at a potential value of 0.8 V vs RHE, the Pt-mass activity is ca. 5.0 and 1.6 times higher at $PtCoNb_2O_5$/GR in acidic and alkaline solutions, respectively, than that at Pt/GR (Fig. 5c). Oxygen reduction current densities normalized by the electrochemically active surface areas recorded at 0.8 V are ca. 4.0 and 1.3 times higher at the $PtCoNb_2O_5$/GR catalyst in acidic and alkaline solutions, respectively, than those at Pt/GR (Fig. 5d).

When comparing the influence of solution media on the activity of $PtCoNb_2O_5$/GR and Pt/GR catalysts towards ORR, it is clearly seen that the current densities, specific current density and mass activity at 0.8 V are ca. 2 and 5 times higher, respectively, in the O_2-saturated 0.1 M NaOH solution than those in acidic media.

With the aim of comparison, the ORR polarization curves of the $PtCoNb_2O_5$/GR catalyst containing 0.085 mg cm^{-2} of platinum and having a 1.25 cm^2 electrochemically active surface area were recorded in O_2-saturated 0.5 M H_2SO_4 (Fig. 6a) and in O_2-saturated 0.1 M NaOH (Fig. 6b) solutions at 1600 rpm. The same tendency is clearly seen at a potential value of 0.8 V vs RHE, the Pt-mass activity is ca. 3.3 and 1.8 times higher for $PtCoNb_2O_5$/GR in acidic and alkaline solutions, respectively, than that for Pt/GR (Fig. 6c). Oxygen reduction current

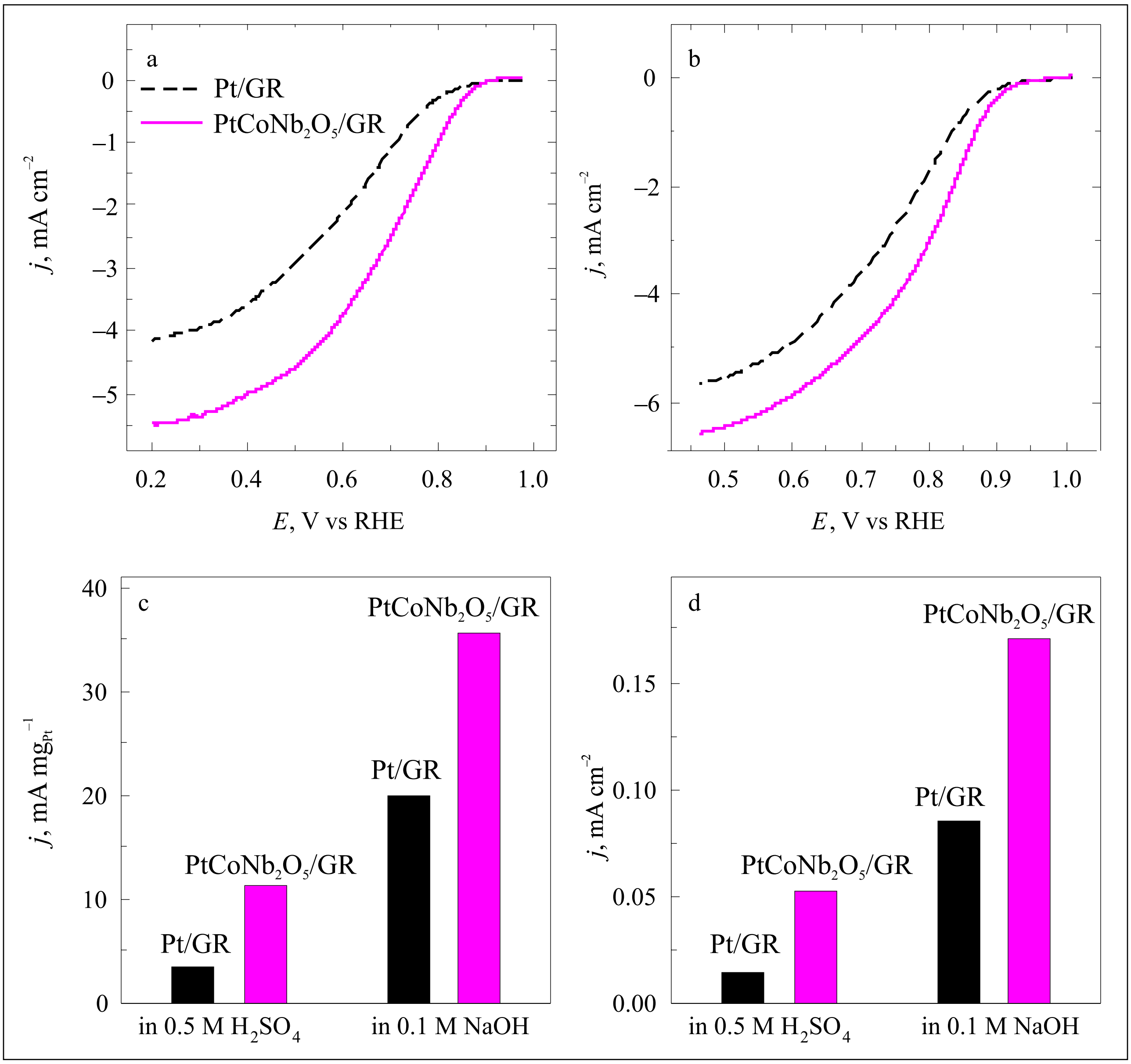

Fig. 6. Comparison of LSV curves at 1600 rpm recorded in O_2-saturated 0.5 M H_2SO_4 solution (a) and O_2-saturated 0.1 M NaOH solution (b); mass (c) and specific (d) activities at 0.8 V for the Pt/GR and $PtCoNb_2O_5$/GR catalysts with the same amount of Pt

densities normalized by the electrochemically active surface areas recorded at 0.8 V are ca. 3.5 and 2.0 times higher for the $PtCoNb_2O_5$/GR catalyst in acidic and alkaline solutions, respectively, than those for Pt/GR (Fig. 6d). The same trend is observed in terms of the influence of the medium of solution. The values of current densities, specific current density and mass activity at 0.8 V are ca. 3 and 5 times higher for the $PtCoNb_2O_5$/GR and Pt/GR catalysts, respectively, in the O_2-saturated 0.1 M NaOH solution than those in acidic media.

CONCLUSIONS

The oxygen reduction reaction catalyzed by the $PtCoNb_2O_5$/GR and Pt/GR catalysts was studied in alkaline and acidic solutions. Both investigated catalysts show better catalytic activities towards ORR in alkaline media. The $PtCoNb_2O_5$/graphene catalyst shows higher onset potentials, as well as higher current densities towards the oxygen reduction reaction in both alkaline and acidic solutions as compared with those of the bare Pt/graphene catalyst.

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PtCoNb$_2$O$_5$ / GRAFENO KATALIZATORIAUS PANAUDOJIMAS DEGUONIES REDUKCIJOS REAKCIJAI ŠARMINIAME IR RŪGŠTINIAME TIRPALUOSE

Santrauka

Mikrobangų sintezės metodu suformuotuose PtCoNb$_2$O$_5$ / grafeno ir Pt / grafeno katalizatoriuose apskaičiuotos Pt įkrovos bei išmatuotos elektrochemiškai aktyvaus paviršiaus ploto vertės atitinkamai lygios 0,171 ir 0,086 mg cm^{-2} bei 3,8 ir 1,4 cm^2. Nusodintų Pt nanodalelių dydis minėtuose katalizatoriuose yra 1–5 nm.

Elektrokatalizinis PtCoNb$_2$O$_5$ / grafeno ir Pt / grafeno katalizatorių aktyvumas deguonies redukcijos reakcijai tirtas naudojant sukamo disko elektrodą O$_2$ prisotintuose 0,5 M H$_2$SO$_4$ ir 0,1 M NaOH tirpaluose, sukimosi greitį keičiant nuo 400 iki 2000 apsisukimų per minutę.

Nustatyta, kad naudojant PtCoNb$_2$O$_5$ / grafeno katalizatorių tiek šarminiame, tiek rūgštiniame tirpaluose deguonies redukcija prasideda teigiamesniuose, t. y. 0,98–1,0 V potencialuose, o naudojant Pt / grafeno katalizatorių deguonies redukcija prasideda esant 0,88–0,93 V potencialui. Deguonies redukcijos srovės tankio vertės, apskaičiuotos pagal nusodintos Pt įkrovą ir aktyvų paviršiaus plotą esant 0,8 V potencialo vertei ir naudojant PtCoNb$_2$O$_5$ / grafeno katalizatorių, yra apie penkis kartus didesnės O$_2$ prisotintame 0,5 M H$_2$SO$_4$ tirpale ir apie 1,5 karto didesnės O$_2$ prisotintame 0,1 M NaOH tirpale, palyginus su Pt / grafeno katalizatoriumi. Abu tirti katalizatoriai pasižymi 3–5 kartais didesniu elektrokataliziniu aktyvumu O$_2$ prisotintame 0,1 M NaOH tirpale, palyginus su rūgštiniu tirpalu.