# Microwave-assisted synthesis of platinum-tungsten/ graphene catalysts

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Department of Catalysis, Center for Physical Sciences and Technology, Saulėtekio Ave. 3, LT-10257 Vilnius, Lithuania In this study a rapid microwave heating method was used to prepare the graphene supported platinum–tungsten catalysts with different Pt:W molar ratios (denoted as PtW/GR). Transmission Electron Microscopy and Inductively Coupled Plasma Optical Emission Spectroscopy were used for the characterization of prepared catalysts. The electrocatalytic activity of the PtW/GR catalysts with respect to the oxidation of borohydride in an alkaline medium was investigated by means of cyclic voltammetry and chrono-techniques.

It has been determined that the graphene supported platinum–tungsten catalysts with the Pt:W molar ratios equal to 1.6:1, 2.3:1 and 7.1:1 were prepared. The prepared PtW/GR catalysts with different Pt:W molar ratios show an enhanced electrocatalytic activity towards the oxidation of  $H_2$  generated by catalytic hydrolysis of  $BH_4^-$  and direct oxidation of  $BH_4^-$  compared with the bare Pt/GR and W/GR catalysts.

Keywords: platinum, tungsten, graphene, sodium borohydride, oxidation

## INTRODUCTION

Fuel cells are promising environmentally clean energy sources, and their research and development are very intense all over the world [1,2]. The greatest efforts in the world are concentrated in developing of novel, more effective catalysts for direct methanol fuel cells (DMFCs), direct borohydride fuel cells (DBFCs), etc. [3, 4]. It is well known that alloying of Pt with transition metals, such as Ni, Co, Cu, etc., allows creating cheaper catalysts for fuel cells. It has been determined that electrocatalytic activity of the Pt-M based catalysts towards borohydride oxidation [5–11], methanol oxidation [12–18] and oxygen reduction [17–28] is higher as compared with that at pure metals. The Pt-based catalysts with other metals such as W and Mo have been investigated as electrocatalysts towards methanol oxidation and oxygen reduction [29–32].

Recently graphene has been used as a new carbon support for low-temperature fuel cell catalysts [33, 34]. In this study we investigated the graphene supported platinum-tungsten nanoparticles catalysts (denoted as PtW/GR) with different Pt:W molar ratios for their borohydride oxidation performance. The PtW/GR catalysts were prepared by means of microwave synthesis. The catalysts obtained were characterized by Transmission Electron Microscopy (TEM) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). The activity of catalysts was investigated with respect to the electro-oxidation of borohydride in an alkaline medium by means of cyclic voltammetry (CV) and chrono-techniques.

## **EXPERIMENTAL**

## Chemicals

H<sub>2</sub>PtCl<sub>6</sub> (Sigma-Aldrich), Na<sub>2</sub>WO<sub>4</sub> · 2H<sub>2</sub>O (Sigma-Aldrich), ethylene glycol (EG) (Chempur), citric acid (Sigma-Aldrich), graphene powder (purity of 97%, specific surface area 60 m<sup>2</sup>/g, Graphene-Supermarket), Nafion (5 wt.%, D521, 1100 EW, Ion Power Inc.) were used. All chemicals were of analytical grade and used without further purification. Ultrapure water with a resistivity of 18.2 MΩ cm<sup>-1</sup> was used to prepare all the solutions.

## **Fabrication of catalysts**

The graphene supported PtW catalysts with different Pt:W molar ratios were prepared by means of microwave heating of  $H_2PtCl_6$  and  $Na_2WO_4 \cdot 2H_2O$  in EG solutions. Briefly, 0.25 ml of 0.0974 M  $H_2PtCl_6$ , required amounts of  $Na_2WO_4 \cdot 2H_2O$  (dissolved in a minimal amount of 10 mM citric acid solution) and 0.3 g graphene powder were mixed with 18.5 ml of EG and sonicated for 30 min. The synthesis was carried out in a microwave reactor Monowave 300 (Anton Paar) at a temperature of 150 °C for 5 min. For comparison, the bare Pt/GR and W/GR

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were prepared in the same manner. After 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 prepared catalysts 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 sample were first sonicated in 1 ml of ethanol for 1 h and then deposited on a Ni grid covered with a continuous carbon film. The composition of the PtW/GR catalysts was estimated using an ICP optical emission spectrometer Optima 7000 DV (Perkin Elmer).

## **Electrochemical measurements**

Borohydride oxidation measurements were performed with a Zennium electrochemical workstation (ZAHNER-Elektrik GmbH & Co. KG) and three-electrode cell. The working electrode was a glassy carbon electrode with a geometric area of 0.07 cm<sup>2</sup> and with a thin layer of Nafion-impregnated catalyst cast, the Pt sheet was used as a counter electrode and Ag/AgCl as a reference electrode. The catalyst layer was obtained by mixing the required amounts of PtW/GR, W/GR or Pt/GR catalysts ultrasonically for 1 hour in a solution containing 0.25 µl of Nafion and 0.75 µl of deionized H<sub>2</sub>O to produce a homogenous slurry. The 5  $\mu$ l of the prepared suspension mixture was pipetted onto the polished surface of a glassy carbon electrode and dried in air for 12 h. The electrocatalytic activity of catalysts towards sodium borohydride oxidation was carried out by recording cyclic voltammograms (CVs) in a 0.05 M NaBH, + 1 M NaOH solution at a scan rate of 10 mV  $s^{-1}$  from -1.1 to 0.6 V at a temperature of 25 °C. The presented current densities are normalized with respect to the geometric area of catalysts. The electrochemically active surface areas (ESAs) of the prepared catalysts were determined from the CVs of the Pt/GR and PtW/GR catalysts recorded in a deaerated 0.5 M H<sub>2</sub>SO<sub>4</sub> solution at a scan rate of 50 mV s<sup>-1</sup> by calculating the charge associated with hydrogen adsorption (220 µC cm<sup>-2</sup>) [35]. All solutions were deaerated by argon for 15 min prior to measurements. The chronoamperometric curves for the investigated Pt/GR, W/GR and PtW/GR catalysts were recorded at a constant potential value of -0.9 and 0.1 V vs Ag/AgCl, respectively, for 30 min. Chronopotentiometric curves were recorded at a current density of 2 mA cm<sup>-2</sup> vs the geometric area of the investigated catalysts for 30 min.

## **RESULTS AND DISCUSSION**

The graphene supported PtW catalysts were prepared by microwave-assisted heating of Pt(IV) and W(VI) salts in ethylene glycol solutions. It should be noted that the reaction mixtures contain the constant amounts of Pt, whereas the amounts of W were different. In this case, the graphene supported PtW catalysts were prepared with different Pt:W molar ratios. The ICP-OES was used for determination of the Pt and W loadings in the synthesized catalysts. The data obtained are given in the Table. After the microwave heating of the reaction mixture at 150 °C for 5 min, the graphene supported PtW catalysts were prepared with the Pt:W molar ratios equal to 1.6:1, 2.3:1 and 7.1:1 (Table). The catalysts were denoted as PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR. The Pt loadings determined by means of ICP-OES analysis were 73 µg Pt cm<sup>-2</sup> in the Pt/GR, 24.7, 54.6, and 41.4  $\mu g$  Pt  $cm^{-2}$  in the synthesized PtW (1.6:1)/ GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR catalysts, respectively.

Table. Composition of catalysts by ICP-OES and electrochemically active surface areas determined from CV

Catalyst	M loading, μg cm <sup>-2</sup>		Pt:W molar	ESA,	ESA,
	Pt	W	ratio	cm <sup>2</sup>	<b>m<sup>2</sup> g</b> <sup>-1</sup>
PtW/GR	24.7	14.4	1.6:1	2.3	134.0
PtW/GR	54.6	21.9	2.3:1	3.2	84.5
PtW/GR	41.4	5.5	7.1:1	3.6	125.7
Pt/GR	73.0	_	1:0	5.2	102.5

Figure 1 presents the TEM views of the prepared Pt/GR (a), PtW(1.6:1)/GR (b), PtW(2.3:1)/GR (c) and PtW(7.1:1)/GR (d).



**Fig. 1.** TEM views of Pt/GR (a), PtW(1.6:1)/GR (b), PtW(2.3:1)/ GR (c), and PtW(7.1:1)/GR (d) prepared by microwave synthesis

As evident from the data in Fig. 1a, the Pt/GR catalyst with an average Pt nanoparticle size of ca. 3 nm was synthesized. In the case of the PtW/GR catalysts, the Pt nanoparticles were found to be ca. 5–20 nm (Fig. 1b–d). Pt nanoparticles were uniform and well dispersed on the surfaces of graphene.

The electrochemically active surface areas of Pt in the synthesized catalysts were determined from the cyclic voltammograms of the Pt/GR and PtW/GR catalysts recorded in a deaerated 0.5 M  $H_2SO_4$  solution at a sweep rate of 50 mV s<sup>-1</sup> by calculating the charge associated with hydrogen adsorption (220  $\mu$ C cm<sup>-2</sup>) (Fig. 2). The data obtained are also given



Fig. 2. CVs of the Pt/GR and PtW/GR catalysts recorded in 0.5 M H\_SO, at 50 mV s<sup>-1</sup>; 25 °C

in the Table. The ESA values are ca. 2.3, 1.6 and 1.4 times higher at the Pt/GR catalyst than those of the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR, respectively.

The activity of the graphene supported PtW catalysts having different Pt:W molar ratios with respect to the oxidation of sodium borohydride was investigated by cyclic voltammetry. Figure 3 presents CVs for the Pt/GR (a), W/GR (a), and PtW/ GR catalysts with the Pt:W molar ratios equal to 1.6:1 (b), 2.3:1 (c), and 7.1:1 (d) recorded in 0.05 M  $NaBH_4 + 1 M$ NaOH at a scan rate of 10 mV s<sup>-1</sup>. During long-term cycling, two anodic peaks A0 and A are seen in the CVs plots for Pt/ GR and different PtW/GR catalysts (Fig. 3). The mentioned catalysts show a typical electrochemical behaviour towards sodium borohydride oxidation as on a pure Pt electrode as described in Ref. [36]. The anodic peak A0 at lower potential values may be related with the oxidation of H, generated by catalytic hydrolysis of  $BH_{4}^{-}$ , and the peak **A** at more positive potential values may be corresponded to the direct oxidation of  $BH_{4}^{-}$  ions [36]. In the reverse potential sweep, the peak **B** observed at about -0.3 V can be assigned to the oxidation of absorbed intermediate oxidation products of BH<sub>3</sub>OH<sup>-</sup> on the partially oxidized Pt surface [36].

Figure 4 presents the comparison of positive-potential going scans of oxidation of  $BH_4^-$  ions recorded on Pt/GR, W/GR and PtW/GR catalysts with different Pt:W molar ratios in a 0.05 M NaBH<sub>4</sub> + 1 M NaOH solution at a potential scan rate of 10 mV s<sup>-1</sup>. The potential values of the anodic peak **A0** are shifted to the negative potential values at the PtW/GR catalysts having the Pt:W molar ratios equal to 2.3:1 and 7.1:1 as



**Fig. 3.** CVs for Pt/GR (a), W/GR (a), PtW(1.6:1)/ GR (b), PtW(2.3:1)/GR (c) and PtW(7.1:1)/ GR (d) recorded in 0.05 M NaBH<sub>4</sub> + 1 M NaOH at 10 mV s<sup>-1</sup>; 25 °C



Fig. 4. Positive-potential going scans of Pt/GR, W/GR and PtW/GR catalysts recorded in 0.05 M NaBH, + 1 M NaOH at 10 mV s<sup>-1</sup>; 25 °C

compared with those at Pt/GR and PtW/GR with the Pt:W molar ratio equal to 1.6:1, indicating a higher activity of those catalysts. The current densities of the peak **A0** recorded on the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR catalysts are ca. 1.3, 1.1 and 1.2 times higher than those on Pt/GR. The current densities under the peak **A** are also ca. 1.2–1.4 times higher as compared to those of Pt/GR.

It should be noted that the prepared PtW/GR catalysts with different Pt:W molar ratios and bare Pt/GR significantly outperformed the bare W/GR catalyst. Since the borohydride oxidation current densities recorded at the W/GR catalyst (Fig. 4) are negligible as compared to those of different PtW/GR and Pt/GR catalysts, the enhanced electrocatalytic activity of the synthesized PtW/GR catalysts may be ascribed to the Pt electronic structure change due to the presence of W [37–39].

To evaluate the specific and mass activities of the prepared catalysts, the sodium borohydride oxidation current densities were normalized by the electrochemically active surface areas and Pt loadings for each catalyst. The data obtained are given in Fig. 5. As seen from the data in Fig. 5, the PtW/GR catalysts outperformed the bare Pt/GR catalyst. Assuming ca. 2.3, 1.6 and 1.4 times higher active surface areas of Pt/GR compared to those of the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR catalysts, respectively, surface area normalized borohydride oxidation current densities under the peaks **A0** and **A** are ca. 3.0, 1.8, 1.8 and 2.7, 2.0, 2.1, respectively, times higher on the latter catalysts (Fig. 5a, b). Mass activity of different PtW/GR catalysts is ca. 3.9, 1.5 and 2.2 (peak **A0**), and 3.5, 1.6 and 2.5 (peak **A**) times higher than that at Pt/GR (Fig. 5c, d).

Stability of catalysts for the oxidation of  $H_2$  generated by catalytic hydrolysis of  $BH_4^-$  (peak **A0**) and oxidation of  $BH_4^-$  ions (peak **A**) was investigated under chronoamperometric conditions. Figure 6 presents the chronoamperometric curves obtained at the Pt/GR, W/GR and PtW/GR catalysts



**Fig. 5.** Comparison of specific activity (a, b) and mass activity (c, d) of different PtW/GR catalysts under values of peak **AO** (a, c) and peak **A** (b, d). The catalysts are the same as in Fig. 4



**Fig. 6.** Chronoamperometric data of the Pt/GR and PtW/GR catalysts recorded in 0.05 M NaBH<sub>4</sub> + 1 M NaOH at -0.9 (a) and 0.1 (b) V, respectively, for 1 800 s

in 0.05 M NaBH<sub>4</sub> + 1 M NaOH at constant potential values of -0.9 (a) and 0.1 (b) V, respectively, for 1 800 s. All the catalysts show a current decay for both reactions. It is worth to note that at -0.9 V, the current densities obtained for all synthesized PtW/GR catalysts are much higher than those for Pt/GR and W/GR. The current densities of the peak **A0** recorded at the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR cata-

lysts are ca. 51, 65 and 62 times higher as compared to those at Pt/GR (Fig. 6a). The current densities of the peak **A** recorded at the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR catalysts at 0.1 V are ca. 1.3, 1.2 and 1.4 times greater than those at Pt/GR (Fig. 6b).

Specific and mass activities of catalysts obtained under chronoamperometric conditions are shown in Fig. 7. Surface



**Fig. 7.** Comparison of specific activity (a, b) and mass activity (c, d) of different PtW/GR catalysts recorded in 0.05 M NaBH<sub>4</sub> + 1 M NaOH at -0.9 (a, c) and 0.1 (b, d) V, respectively, at t = 1800 s. The catalysts are the same as in Fig. 6

area normalized borohydride oxidation current densities at -0.9 V are ca. 115, 106 and 90 and at 0.1 V are ca. 2.9, 2.0, 2.0 times greater on the PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR catalysts, respectively, as compared to those at Pt/GR (Fig. 7a, b), whereas the mass activity at -0.9 V is ca. 151, 87 and 110 and at 0.1 V is ca. 3.7, 1.6 and 2.4 times higher at PtW(1.6:1)/GR, PtW(2.3:1)/GR and PtW(7.1:1)/GR than those on Pt/GR (Fig. 7c, d).

Chronopotentiometry measurements were carried out to screen the catalyst performance under the conditions of constant current operation of the fuel cell anode. Following a rest period of 10 s at open circuit, a current density step of 2 mA cm<sup>-2</sup> was applied to the investigated catalysts for 1 800 s. Figure 8 shows the anode potentials (including the opencircuit values between -0.612, -0.920, -0.996, -1.028 and -1.027 V vs Ag/AgCl) for the catalysts operating at 2 mA cm<sup>-2</sup>. Notably, the open-circuit potential values are by ca. 0.3-0.4 V more negative than those of W/GR. After 1 800 s, the operating potentials for Pt/Gr and PtW/GR are by approximately 1.4–1.6 V more negative than those for W/GR, which demonstrates that the overpotentials for the oxidation of BH<sub>4</sub><sup>-</sup> ions on the Pt/GR and PtW/GR catalysts are lower than those for W/ GR. The difference between the steady-state operating anode potential and the one at open circuit was the smallest one on the PtW(1.6:1)/GR and was equal to ca. 0.007 V, followed by the PtW(2.3:1)/GR, Pt/GR and PtW(7.1:1)/GR catalyst, where they reached ca. 0.044, 0.126 and 0.164 V, respectively, and finally, W/GR where it reached ca. 1.245 V.



**Fig. 8.** Chronopotentiometric data of the Pt/GR, W/GR and PtW/GR catalysts recorded in 0.05 M NaBH<sub>4</sub> + 1 M NaOH at 2 mA cm<sup>-2</sup> for 1 800 s

## CONCLUSIONS

In this study the graphene supported PtW catalysts with different Pt:W molar ratios equal to 1.6:1, 2.3:1 and 7.1:1 were prepared by microwave-assisted heating of Pt(IV) and W(VI) salts in ethylene glycol solutions. The PtW/GR catalysts were synthesized with nanoparticles in size of ca. 5–20 nm.

It has been determined that the PtW/GR catalysts with different Pt:W molar ratios show an enhanced electrocatalytic activity towards the oxidation of  $H_2$  generated by the catalytic hydrolysis of  $BH_4^-$  and the direct oxidation of  $BH_4^-$  compared with Pt/GR and W/GR.

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## PLATINOS-VOLFRAMO / GRAFENO KATALIZATORIŲ FORMAVIMAS TAIKANT MIKROBANGŲ SINTEZĘ

#### Santrauka

Darbe platinos-volframo / grafeno katalizatoriai (PtW/GR) buvo formuojami taikant mikrobangų sintezę. PtW/GR katalizatoriai susintetinti esant skirtingiems Pt:W moliniams santykiams. Gauti katalizatoriai tirti taikant peršviečiamąją elektroninę mikroskopiją ir indukuotos plazmos optinės emisijos spektroskopiją. PtW/GR katalizatorių elektrokatalizinis aktyvumas natrio borhidrido oksidacijos reakcijai šarminėje terpėje tirtas taikant ciklinę voltamperometriją, chronoamperometriją ir chronopotenciometriją.

Nustatyta, kad taikant mikrobangų sintezę, buvo susintetinti PtW/ GR katalizatoriai, kuriuose Pt:W molinis santykis 1.6:1, 2.3:1 ir 7.1:1, o nusodintų metalo nanodalelių dydis – nuo 5 iki 20 nm. Šie katalizatoriai pasižymi didesniu elektrokataliziniu aktyvumu  $H_2$ , susidariusio natrio borhidrido katalizinės hidrolizės metu, oksidacijai ir tiesioginei natrio borhidrido oksidacijai, palyginti su grynaisiais Pt/GR ar W/GR katalizatoriais.