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Electrooxidation of ethanol and acetaldehyde using PtSn/C and $PtSnO_2$/C catalysts prepared by a modified alcohol-reduction process

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Abstract. Well-dispersed PtSn/C and $PtSnO_2$/C catalysts with an atomic ratio of Pt:Sn (3:1) were deposited onto carbon using a modified alcohol-reduction process for electrochemical oxidation of ethanol. These materials were characterized using Energy-Dispersive X-ray Spectroscopy (EDX) and X-Ray Diffraction (XRD). An electrochemical study in the ethanol and acid medium showed that the $PtSnO_2$/C catalyst had a better performance compared with the PtSn/C and Pt/C catalysts. The CV analysis indicated that the presence of SnO_2 increased the sensitivity of catalyst to oxidation of acetaldehyde because of its low onset potential for the electrooxidation of adsorbed ethanol and acetaldehyde compared to PtSn/C and Pt/C. Since the activation of C-C bond breaking results in the generation of higher current density (mass activity) at higher potentials, the $PtSnO_2$/C catalyst has been found favourable for the overall oxidation of ethanol.

1. Introduction

Low molecular weight alcohols are extensively suggested as possible fuel for low temperature fuel cells in portable applications. Among the alcohols, methanol and ethanol have been widely considered in Direct Alcohols Fuel Cells (DAFCs). DEFCs have attracted great attention as a power source in many applications because of their high efficiency, nontoxic and environmentally friendly characteristics, and straightforwardness in structure [1-3].

The best binary alloyed catalyst for Ethanol Oxidation Reaction (EOR) in an acid environment is Pt-Sn [4]. The catalytic activity of a partly alloyed Pt-Sn catalyst and that of a quasi non-alloyed $PtSnO_x$ catalyst were compared. In the analysis of chronoamperometry results of DEFC, the $PtSnO_x$ catalyst displayed higher catalytic activity for ethanol electrooxidation than the Pt-Sn alloy. Jiang et al. inferred that the unchanged lattice parameter of Pt in the PtSnOx catalyst is favourable to ethanol adsorption and the presence of tin oxide, a part of Pt nanoparticles, could simply provide oxygen species to remove the CO-like species of ethanolic residues to free Pt active sites [5]. In another study, $PtSnO_2$ and PtSn alloy catalyst were synthesized and investigation of the products demonstrated that the addition of SnO_2 improved the total oxidation to CO_2 [6]. SnO_2 is recognized for potentially being able to provide OH-species to oxidize, toughly, bound intermediates such as CO [7,8].

Ethanol oxidation involves three key products: acetaldehyde as a main product, acetic acid, and carbon dioxide. The mechanism of oxidation of acetaldehyde shows that acetic acid and CO_2 are the main products, though the type and content of yields depend on catalyst material, electrode potential, temperature, and acetaldehyde concentration [9,10]. Electrooxidation of acetaldehyde was studied on Pt and Rh electrodes in an acid solution by Differential Elec-

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trochemical Mass Spectroscopy (DEMS) techniques. CO_2 and acetic acid were detected as electrooxidation yields and methane and ethane were formed during the reduction of adsorbed species. Ethane was detected only on the Pt electrode [11]. FTIR spectroscopy and High Performance Liquid Chromatography (HPLC) techniques were applied to investigate the electrooxidation of acetaldehyde on Pt electrodes. The studies have shown that acetic acid is the main product and formic acid and CO_2 are detected in small amounts [12].

The oxidation of acetaldehyde on $PtSnO_2/C$ has been studied rarely. In this work, we synthesized SnO_2 and made a binary catalyst by adding a tin oxide to Pt; its performance was then compared with PtSn/C and Pt/C. An assembly of techniques, such as Field Emission Scanning Electron Microscopy (FESEM), X-Ray Diffraction (XRD), and Induced Coupled Plasma (ICP), were employed to characterize the physical properties of the synthesized catalysts. Then, the electrocatalyst activity in ethanol and acetaldehyde solution on synthesized PtSn/C, $PtSnO_2/C$, and Pt/C catalysts was investigated.

2. Materials and method

2.1. Materials

Ethylene glycol, H_2SO_4, $H_2PtCl_6.6H_2O$, and $SnCl_2.2H_2O$ as metal sources, isopropyl alcohol (IPA), ethanol, and acetaldehyde were all purchased as analytically pure reagents from Merck.

2.2. Catalyst synthesis

PtSn/C alloy nanoparticles were deposited on Vulcan XC-72R by using the modified polyol method. The steps involved in the preparation were:

i) Incorporation of a predetermined amount of carbon, i.e. Vulcan, to 15 cc of ethylene glycol, then sonicating the entire mixture for 30 min;

ii) Addition of the required precursors, i.e. $H_2PtCl_6.6H_2O$ and $SnCl_2.2.H_2O$, to the composite mixture in the stoichiometric amount required and sonication for 30 min;

iii) Addition of 45 cc of ethylene glycol and sonication again for 30 min;

iv) Increasing the temperature to 120°C, and then keeping it constant for 16 h to allow the nucleation and growth of the alloy metal on the carbon support;

v) Cooling the suspension down to room temperature;

vi) Separation of the solid by filtration, and subsequent rinsing with water and drying the solid at 80°C for 24 h in an oven.

In the case of $PtSnO_2/C$, first the stoichiometric amount required of $SnCl_2.2.H_2O$ was added to 50 cc ethylene glycol and heated at 195°C under an oxygen atmosphere till a light yellow coloured solution was obtained. The solution was cooled to room temperature and the required amount of $H_2PtCl_6.6H_2O$ was added to the mixture; then, the above preparation steps after "iii" were repeated for the mixture. Atomic ratios of catalysts were Pt:Sn (3:1) with 10% of metal loading.

2.3. Preparation of catalyst electrodes

The Glassy Carbon (GC) electrode ($d = 1$ mm) was polished with an aluminium oxide paste as a working electrode. A specific amount of prepared catalyst powder was mixed to 1 cc of IPA:H_2O (2:1) and 0.1764 mg/ml Nafion suspension as a binder. Then, the mixture was sonicated for 20 min to achieve a homogeneous ink-like solution. The ink was embedded onto the surface of GC by Hamilton pipette and dried in the air at 70°C.

2.4. Electrochemical characterization

For the electrochemical characterization, a conventional three-electrode cell was used. The working electrode was a glassy carbon covered by a thin layer of catalyst detachment in a Nafion polymer electrolyte film. A Pt foil of 1 cm^2 geometric area was used as the counter electrode and a saturated calomel electrode as the reference electrode. The electrochemical characterization was recorded in a 1 M C_2H_5OH in 0.5 M H_2SO_4 solution and 0.25 M C_2H_4O in 0.5 M H_2SO_4 solution at room temperature. Most of the electrochemical experiments were carried out at room temperature. The real active electrode area was determined from the CV in acidic media at 50 mV s^{-1} in a 0.5 M H_2SO_4 solution.

2.5. Physical characterization

The samples were characterized with a Field Emission Scanning Electron Microscope (FE-SEM) ZEISS SIGMA VP (Germany). X-Ray powder Diffraction (XRD) measurements were performed using a Philips X'pert diffractometer with mono chromated Co-Kα (1.78897 Å) radiation.

3. Results and discussion

3.1. Physical measurements

PtSn/C and $PtSnO_2/C$ electrocatalysts were prepared using ethylene glycol as solvent and reducing agent in the presence of carbon Vulcan. The atomic ratio of Pt:Sn obtained was similar to the nominal atomic ratios used in the preparation procedure from ICP, the Energy Dispersive X-ray spectrometry (EDX) is shown in Table 1.

Figure 1 represents the EDX of $PtSnO_2/C$, where three peaks of intense Pt signals demonstrate the

Table 1. Characterization results of carbon supported catalysts.

Electrocatalyst	Nominal atomic ratio		Atomic ratio, ICP		Atomic ratio, EDX		Particle size (nm), XRD
	Pt	**Sn**	**Pt**	**Sn**	**Pt**	**Sn**	
PtSn/C	75	25	73	27	-	-	4.5
$PtSnO_2$/C	75	25	74	26	77	23	4.6
Pt/C	100	0	-	-	-	-	2.6

Table 2. Comparative performance data of $PtSnO_2$/C, PtSn/C, and Pt/C electrodes determined from Figures 3 and 5.

Sample	Q_H (μC)	ECSA (m^2/g)	For ethanol oxidation (Figure 3)				For acetaldehyde oxidation (Figure 5)	
			MSA	E_{on} (V)	I_p (A)	I_f/I_b	I_p (A)	E_{on} (V)
Pt/C	406	61	1698	0.28	35.9	0.84	1.44	0.25
Pt-Sn/C	360.9	68	2975	0.18	40	0.85	1.96	0.14
Pt-SnO_2/C	348.3	65	4364	0.13	50	1.01	3.39	0.01

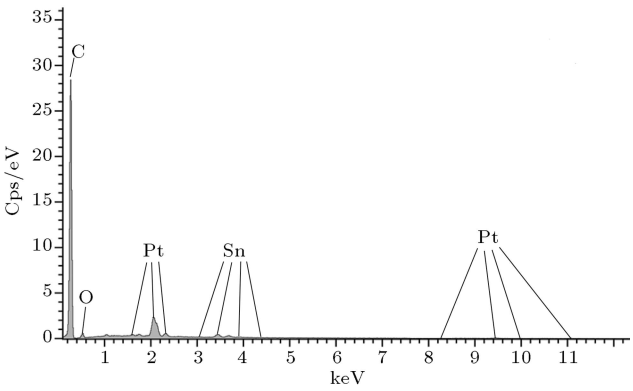

Figure 1. Energy dispersive X-ray spectra of $PtSnO_2$/C.

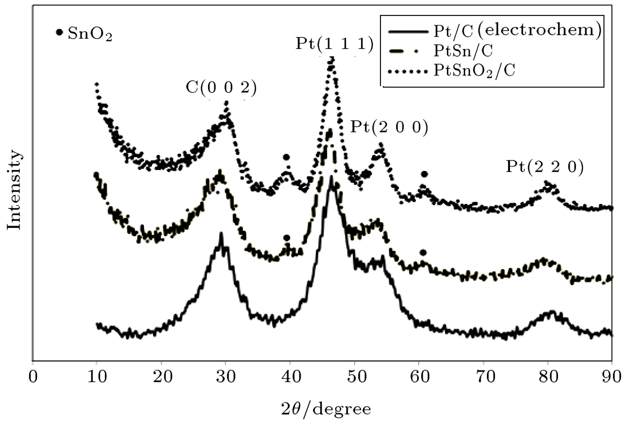

Figure 2. XRD patterns of $PtSnO_2$/C, PtSn/C, and Pt/C catalysts.

appropriate deposition of Pt on the support. Moreover, the catalyst shows an extra Sn peak, which confirms the predeposited SnO_2.

The XRD patterns were achieved at a scanning rate of 1°/min with a step size in the 2θ scan of 0.02° in the range $10-90°$ and demonstrated in Figure 2. All of the catalysts show three characteristics diffraction peaks corresponding to the (111), (200), and (220) crystalline faces of Pt. A broad peak at about 29° is attributed to the (0 0 2) planes of the Vulcan XC72 support material. Two small peaks for SnO_2 ($2\theta = 39°$ and 59°) were observed in PtSn/C and $PtSnO_2$/C [13]. The crystallite sizes of the catalysts were calculated and depicted in Table 1, according to the Scherrer formula [14].

3.2. Electrochemical measurements

Cyclic Voltammetry (CV) was applied to investigate the catalytic activity of electrocatalysts. According to the CVs in acid media, the electrochemically active surface area (ECSA) was calculated through integration of the anodic current corresponding to the hydrogen desorption reaction with the correction of the double layer region. The ECSA values are shown in Table 2 and can be determined from Eq. (1) [15]:

$$S_{\text{ECSA}}\ (\text{m}^2\text{g}^{-1}) = \frac{Q_H\ (\mu\text{C/cm}^2)}{210 \times L_{Pt}\ (\text{g/m}^2)}, \tag{1}$$

where Q_H (μC cm^{-2}) is the charge corresponding to hydrogen desorption on the Pt surface, L_{Pt} (g m^{-2}) is the Pt loading in the working electrode, and 210 (μC/cm^2) represents the charge required to oxidize a monolayer of hydrogen on the Pt surface. The results show that PtSn/C has a higher ECSA compared to $PtSnO_2$/C and Pt/C. In $PtSnO_2$/C, some tin oxide particles cover the Pt nanoparticles resulting in ECSA reduction [16]. Then, the catalytic activities of carbon-supported $PtSnO_2$/C, PtSn/C, and Pt/C catalysts for ethanol electrooxidation were examined at ambient temperature by Cyclic Voltammetry (CV) at a scan rate of 50 mV s^{-1} in 1 M EtOH + 0.5 M H_2SO_4 (aq) solution (Figure 3). It was observed that the $PtSnO_2$/C catalyst exhibited significantly enhanced

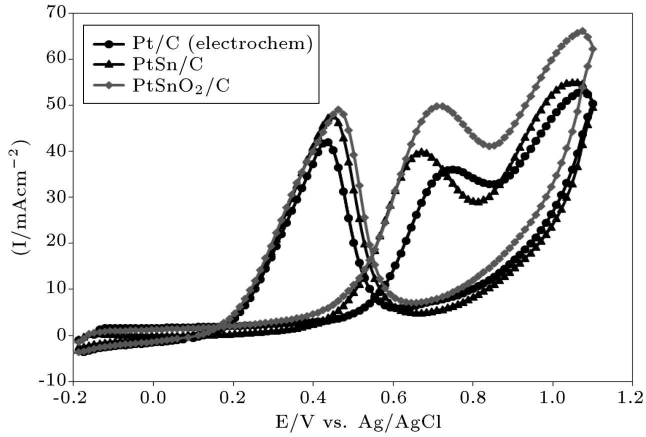

Figure 3. Cyclic voltamograms of the PtSnO$_2$/C, PtSn/C, and Pt/C in 1 M ethanol + 0.5 M H_2SO_4 solution at the 50th cycle with a scan rate of 50 mVs^{-1}.

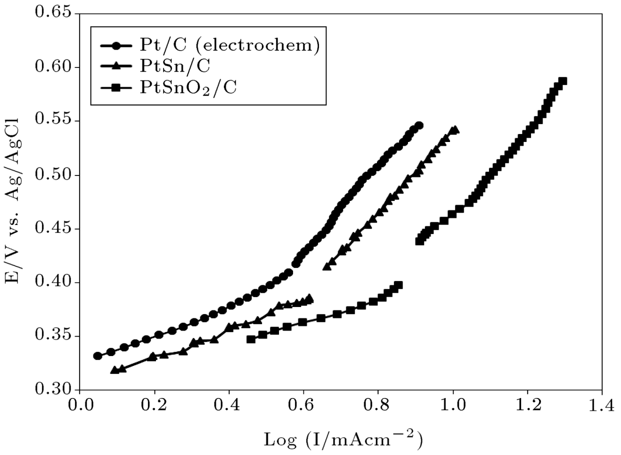

Figure 4. Tafel plots of the PtSnO$_2$/C, PtSn/C, and Pt/C in 1 M ethanol + 0.5 M H_2SO_4 at room temperature; the scan rate is set at 1 mVs^{-1}.

catalytic activities for ethanol oxidation compared to PtSn/C and Pt/C catalysts.

The current densities for ethanol oxidation on PtSnO$_2$/C were higher than those of PtSn/C and Pt/C. Three synergic reasons including the Pt surface area increase, the change in the "d" band level of the material, and the removal of adsorbed intermediates by a bi-functional mechanism should be mentioned to justify the obtained results [13]. Mass Specific Activity (MSA) was calculated using Eq. (2), according to the required charge density for ethanol oxidation reaction (Q_{EOR}) in Figure 3 [17]:

$$\mathrm{MSA}\ (\mathrm{mC\ mg}_{Pt}^{-1}) = \frac{Q_{\mathrm{EOR}}\ (\mathrm{mC/cm}^2)}{L_{Pt}\ (\mathrm{mg/cm}^2)}. \tag{2}$$

The significantly higher MSA for PtSnO$_2$/C with respect to the others is justified based on the three reasons mentioned above (Table 2).

As shown in Figure 3, all catalysts show the same redox characteristics for ethanol oxidation; a distinctive oxidation current peak in the forward scan (I_f) and an oxidation peak in the backward scan (I_b). Our research shows that the ratio of I_f/I_b is dependent upon CO tolerance and more complete oxidation of ethanol or methanol to CO$_2$. The ratios of I_f/I_b values for all catalysts are given in Table 2. The higher ratio of I_f/I_b value for PtSnO$_2$/C implies a better oxidation of ethanol to CO$_2$, less accumulation of carbonaceous residues on the catalyst surface, and more CO tolerance than PtSn/C and Pt/C [18,19]. The higher ratio of I_f/I_b for PtSnO$_2$/C compared to the other catalysts indicates that the presence of SnO$_2$ in a catalyst increases the C-C bond breaking and reduces the CO poisoning. The peak current density (I_p) and onset potential (Eon) during ethanol oxidation on composite electrodes confirms the previous results as shown in Table 2. The peak current density on PtSnO$_2$/C is the highest at 1.4 times of Pt/C. According to Table 2, the onset potential on PtSn/C and PtSnO$_2$/C is about 100-150 mV less than Pt/C.

The kinetic data are extracted from the Tafel plots as shown in Figure 6. Tafel slopes of electrocatalysts are calculated from Tafel plots as shown in Figure 4. Every plot for each catalyst is fitted and separated into two linear sections according to the change of Tafel slopes. The second slopes are considerably higher than the first ones, demonstrating a possible change of reaction mechanism or at least a change of the rate-determining step at different potential ranges. All the Tafel slopes on the three catalysts were almost identical; so, this similarity indicates that the same reaction mechanism operated on the different catalysts. The addition of tin or tin oxide into Pt/C extremely improved the activities and shifted the onset potentials towards the negative potentials. The group of data in Table 3 indicates that the reaction starts up earlier on PtSnO$_2$/C. The current densities and charge transfer coefficient on this catalyst are higher than other catalysts (Table 3). The exchange current density can be calculated by extrapolating the Tafel line. As shown in Table 3, the exchange current density on PtSnO$_2$/C was more than one order of magnitude higher than that on Pt/C.

Figure 5 represents the voltammograms of acetaldehyde electrooxidation in a 0.25 M CH_3CHO + 0.5 M H_2SO_4 solution recorded on PtSnO$_2$/C, PtSn/C, and Pt/C catalysts. From the CV curves, the hydrogen adsorption/desorption is significantly depressed by the adsorption of acetaldehyde on the catalysts [20]. The highest mass activity and the lowest onset potential were apperceived on the PtSnO$_2$/C (Table 2). The improvements of acetaldehyde oxidation with the addition of SnO$_2$ into Pt are mainly attributed to the effective elimination of C2 species and inhibition of poison formation. Since the mentioned

Table 3. Tafel slopes, charge transfer coefficient, and exchange current density data for $PtSnO_2/C$, PtSn/C, and Pt/C electrodes extracted from Figure 4.

Sample	**Tofel slope for first linear region (mV/dec)**	**Tofel slope for second linear region (mV/dec)**	**Charge Transfer Coefficient**	**Exchange current density ($\mathbf{A\ cm^{-2}}$)**
Pt/C	150	393	0.39	6×10^{-6}
Pt-Sn/C	132	373	0.45	1×10^{-5}
Pt-SnO_2/C	104	368	0.56	5×10^{-5}

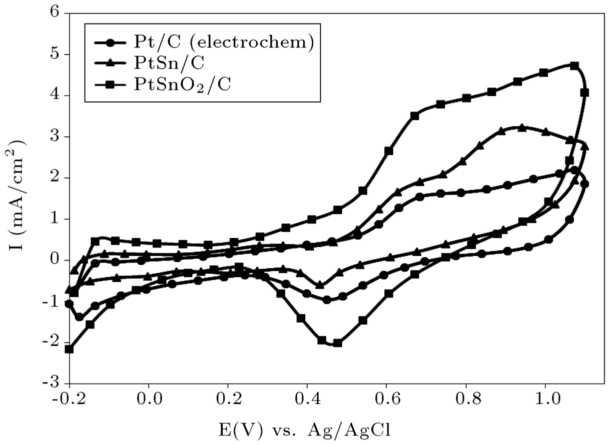

Figure 5. Cyclic voltamograms of the $PtSnO_2/C$, PtSn/C, and Pt/C in 0.25 M acetaldehyde + 0.5 M H_2SO_4 solution with a scan rate of 10 mVs^{-1}.

improvements do not involve C-C bond breaking, supplying oxygen containing SnO_2 species results in oxidation of acetaldehyde adsorbate (C2) compared to Sn.

3.3. Electrochemical Impedance Spectroscopy (EIS) studies

Table 4. Charge transfer resistance (R_{ct}) of Pt/C, PtSn/C, and $PtSnO_2/C$ according to the fitting of corresponding Nyquist plots (Figure 6) by Z-view (ver. 2).

Electrocatalyst	**Pt/C**	**PtSn/C**	**$\mathbf{PtSnO_2/C}$**
R_{ct} (Ω cm^2)	190	62	44

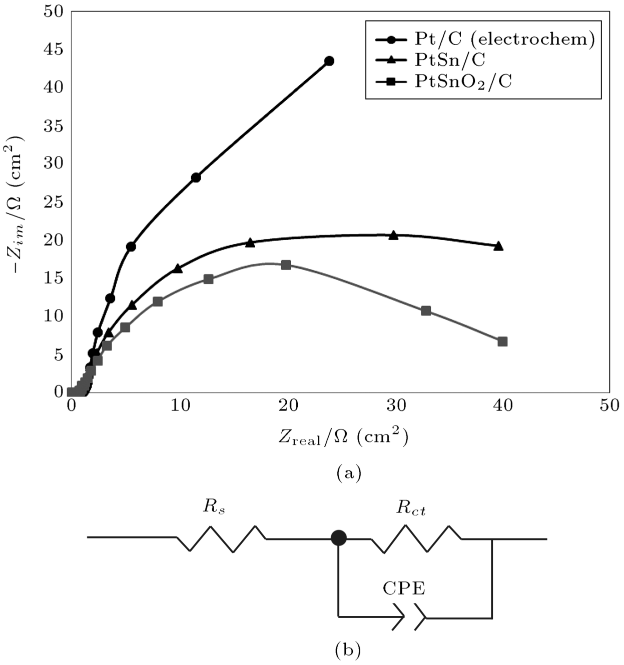

Figure 6. a) EIS Nyquist plots of Pt/C, PtSn/C, and $PtSnO_2/C$ in 1 M ethanol + 0.5 M H_2SO_4 electrolyte at 0.3 V vs. Ag/AgCl at room temperature. The frequencies ranged from 100 kHz to 100 mHz. b) Equivalent circuit for corresponding EIS plots, driven by Z-view software.

Figure 6(a) compares the EIS for EOR on Pt/C, PtSn/C, and $PtSnO_2/C$ catalysts at 0.3 V in 100 kHz-100 mHz frequency range with the amplitude of the sine wave being 5 mV. The equivalent circuit derived by Zview software to characterize the electrochemical behavior of the catalysts is shown in Figure 6(b) and the obtained charge transfer resistances (R_{ct}) are depicted in Table 4. The high frequency intercept on the real axis in the circuit is solution resistance (Rs). The CPE in parallel with R_{ct} indicates the double layer capacitance. At this potential, a large arc is seen in the spectrum of each catalyst. As shown in Table 4, the charge transfer resistance (R_{ct}) of the $PtSnO_2/C$ is much lower than PtSn/C and Pt/C. Supplying the oxygen containing species on SnO_2 for oxidation of adsorbed intermediate species, such as CHx or CO, the fresh Pt sites for ethanol adsorption are exposed [21].

4. Conclusion

Catalysts of $PtSnO_2/C$ and PtSn/C were prepared through a modified alcohol-reduction process. The EDX analysis showed that the atomic ratio of $PtSnO_2/C$ is close to the nominal value of Pt:Sn (3:1). The electrochemical measurements show that $PtSnO_2/C$ has a higher activity of ethanol and acetaldehyde oxidation than the other studied catalysts. XRD spectra indicate that the size of the synthesized nanoparticles on the supports is about 4-5 nm. The kinetic data was calculated and the obtained results showed that $PtSnO_2/C$ was an appropriate promising material in DEFCs.

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