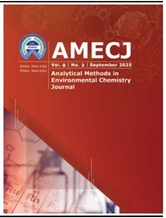
Anal. Methods Environ. Chem. J. 8 (3) (2025) 94-104

AMECJ

Research Article, Issue 3
Analytical Methods in Environmental Chemistry Journal
Journal home page: www.amecj.com/ir



Electrochemical analysis: Fabrication of bimetallic Pt/Sn nanocatalyst electrode with modified glassy carbon analysis and evaluation of methanol oxidation reaction

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ARTICLE INFO:
Received 17 May 2025
Revised form 19 Jul 2025
Accepted 14 Aug 2025
Available online 28 Sep 2025

Keywords:
Electrocatalyst,
Platinum,
Electrochemical analysis,
Polymer,
Tin,
Methanol oxidation

ABSTRACT

For the methanol oxidation reaction (MOR), the electrocatalytic activity of Platinum and Platinum/tin nanoparticles supported on Vulcan carbon and multi-walled carbon nanotubes (MWCNT) was examined. Pt/C, Pt-Sn/C, Pt/MWCNT, and Pt-Sn/MWCNT catalysts were created via the precipitation reduction process with sodium borohydride. Cyclic voltammetry, field emission scanning electron microscopy (FESEM), and X-ray diffraction (XRD) were employed to investigate the electrocatalysts. In comparison to other electrocatalysts, the Pt-Sn/MWCNT electrocatalyst with the Pt/Sn atomic ratio (3:1) demonstrated a lower peak potential during cyclic voltammetry and a greater forward peak current density (0.3 mA) in the methanol oxidation process (MOR) (0.55 V). The impact of applying a thin polyaniline layer to the glassy carbon (GC) electrode surface was next examined. In aqueous acidic environments, a thin coating of polyaniline and platinum-tin electrocatalytic particles supported by multiwall carbon nanotubes (GC/polyaniline/Pt-Sn/MWCNT) (1.2 mA) increased methanol oxidation. Platinum and platinum-tin nanoparticles on multi-walled carbon nanotubes and Vulkan carbon have been effectively produced by a deposition-reduction method with sodium borohydride.

1. Introduction

Developing innovative materials capable of addressing complex challenges in renewable energy conversion, generation, and storage is essential for securing an alternative to environmentally harmful fossil fuels. Fuel cells are clean, efficient, and suitable for renewable energy sources and carriers for energy security and sustainability. Recently, methanol electro-oxidation has garnered scholarly interest due to the potential of direct methanol fuel cells (DMFCs) as significant power sources for the future [1]. Compared to hydrogen, low-temperature direct methanol fuel cells (DMFCs) are very attractive as energy sources for mobile, portable, and transportation applications due to their ease of fuel management and safe storage [2,3]. In addition to electrocatalytic approaches, other methods for determining organic materials have also been widely employed. These include various extraction techniques and analytical

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https://doi.org/10.24200/amecj.v8.i03.1038

methods [4], if the amounts of heavy metals are more than the acceptable amounts (mentioned by WHO, such as immobilization on graphene oxide [5], functionalized MWCNTs [6], nano-carbon structures (such as Carbon Quantum Dots synthesized from olive stones) [7,8], and task-specific ionic liquids [9,10], as well as approaches for heavy metals determination in environmental and human samples. Methanol possesses distinct advantages as a viable fuel alternative, including high energy density, minimal environmental contamination, favorable oxidation kinetics relative to other alcohols at moderate temperatures, and complete conversion to CO_2, which results in maximum electron transfer. Consequently, methanol oxidation has been extensively researched over the past few decades [11]. Electrode materials play a crucial role in the electrocatalytic oxidation of methanol. Adsorption, oxidation, and repulsion of adsorbed intermediates are all handled by the methanol oxidation reaction (MOR), which is a somewhat sluggish process in and of itself [12]. Two primary issues affect the effectiveness of DMFs: inadequate anodic kinetic properties for the extended oxidation of methanol and the poisoning of anodic electrocatalysts resulting from intermediate reactions during the methanol oxidation half-cell reaction in a DMFC. Both are limitations that must be resolved before the extensive implementation of these fuel cells as energy delivery systems. Platinum serves as the most effective catalyst for alcohol oxidation; yet, its surface is swiftly compromised by the irreversible adsorption of CO-like intermediates resulting from the dissociative adsorption of alcohols [13]. Furthermore, platinum is a scarce metal and exceedingly costly, necessitating a reduction in the cost-to-efficiency ratio. Therefore, increasing the electrocatalytic activity for methanol oxidation and methanol tolerance while also lowering the cost of these electrocatalysts is a primary goal in fuel cell catalyst research [14]. Alloying Pt metal with other transition metals, for example, Sn [15-18], Ti [19, 20], and Ru [21,22], is an efficient approach for improving the tolerance of catalysts. Numerous parameters, including fabrication techniques, supporting materials, particle size, dispersion, and surface conditions, influence the electrocatalytic activity of Pt nanoparticles in methanol reactions. The extensive dispersion of Pt nanoparticles on the support is crucial for its electrocatalytic performance [23]. Catalysts with high surface area are utilized to minimize metal loading while maintaining the catalysts' high activity [24]. Optimal support should also exhibit substantial electrical conductivity. The elevated electrical conductivity of the supports during methanol oxidation promotes swift electron transfer from the electrode to the external circuit, thereby improving catalytic performance [25]. MWCNTs are regarded as a compelling support material due to their strong electrical conductivity, substantial surface-to-volume ratio, and excellent thermal and chemical stability [26]. Conductive polymers serve as an effective supporting material in fuel cell technology by facilitating the uniform distribution of catalyst particles, a fundamental requirement for electrocatalytic reactions. These polymers are frequently employed as host matrices to integrate noble metal catalysts for potential uses in the electrooxidation of small molecules, including hydrogen, methanol, and formic acid [27]. Polyaniline is particularly noteworthy among conducting polymers due to its environmental resilience, adjustable electrical conductivity, and diverse redox characteristics. Polyaniline exhibits a unique interaction with Pt, which reduces CO adsorption on Pt particles —a major factor in the poisoning of the Pt catalyst [28]. This study investigates the performance of electrocatalysts prepared on carbon-based supports through chemical reduction with sodium borohydride, and subsequently tests them for methanol oxidation. The electrodes were modified with polyaniline and Pt-Sn/MWCNT particles. Results indicate that including MWCNT and PANI enhances electrocatalytic efficiency for the methanol oxidation reaction (MOR).

2. Experimental

2.1. Materials and apparatus

Hexachloroplatinic acid hexahydrate ($H_2PtCl_6·6H_2O$, CAS N.: 18497-13-7, Merck, Germany), tin(II) chloride dihydrate ($SnCl_2·2H_2O$, CAS N.:10025-69-1, Merck, Germany), sodium hydroxide (NaOH, CAS N.: 1310-73-2, Merck, Germany), sodium borohydride ($NaBH_4$, CAS N.: 16940-66-2, Merck, Germany) were procured from Merck. Carbon Vulkan XC72 (CAS N.: 133-86-4) and multiwall carbon nanotubes (MWCNTs, CAS N.: 308068-56-6) were acquired from Alfa Aesar (USA), while Nafion (Wt. 5%, CAS N.: 31175-20-9) was purchased from Aldrich (USA).

2.2. Preparation of electrocatalysts

Pt/C, Pt-Sn/C, Pt/MWCNT, Pt-Sn/MWCNT Catalysts with Pt/Sn atomic ratios of 3:1 were synthesized with $H_2PtCl6·6H2O$ and $SnCl_2·2H_2O$ as metal sources, together with carbon Vulkan XC72 and multiwall carbon nanotubes as supports. Using $NaBH_4$ (Merck) as a reducing agent, catalysts were created in the liquid phase using the deposition-reduction approach. The supports were suspended in deionized water under magnetic stirring for 30 minutes at 40°C. Measured quantities of $H_2PtCl_6·6H_2O$ (0.003 M) and $SnCl_2·2H_2O$ solutions (0.047 M) were included in the aforementioned combination. The black suspension was immersed in an ultrasonic bath for one hour. A freshly made 0.1M $NaBH_4$ solution (in 1M NaOH) was incrementally added to the aforementioned solution under vigorous agitation in four equal volumes. The portions were added every 30 minutes. After 2 hours of agitation, the mixture was held at room temperature for 12 hours, filtered, and rinsed with deionized water. The powder was dehydrated in a 70°C vacuum oven for 3 hours. Platinum catalysts were also made via $NaBH_4$ reduction.

2.3. Physical characterization

Using a Cu anode on an X'Pert Pro device, the X-ray diffraction (XRD) patterns of the synthesized catalysts were obtained to analyze their crystalline structure. The operational voltage and current for XRD were set at 40 kV and 40 mA, respectively. The diffractograms were acquired at 2θ angles ranging from 5 ° to 80° with a step size of 0.026°. The field-emission scanning electron microscope (FESEM) was utilized for the microscopic analysis of the synthesized catalysts.

2.4. Electrochemical activity measurements

Electrochemical experiments were conducted at ambient temperature utilizing a potentiostat within a standard three-electrode test cell. An electrode made of glassy carbon served as the active electrode. In this setup, a platinum wire served as the reference electrode and a standard calomel electrode (SCE) as the counter electrode. All the potentials examined in this investigation were associated with SCE. The MOR activity was assessed via cyclic voltammetry between -1 and +1.2 V/SCE in 0.5 M CH_3OH with 0.1 M H_2SO_4 as the electrolyte at ambient temperature, utilizing a scan rate of 50 mV/s. Catalyst inks were prepared by dispersing 5 mg of catalyst powder in a mixture of Nafion, ethanol, and water, followed by sonication for approximately 30 minutes to achieve a homogeneous ink. Subsequently, 10 microliters of catalyst inks from each type of catalyst were applied as a thin layer over a freshly polished glassy carbon electrode using a pipette and dried at 60°C for 10 minutes.

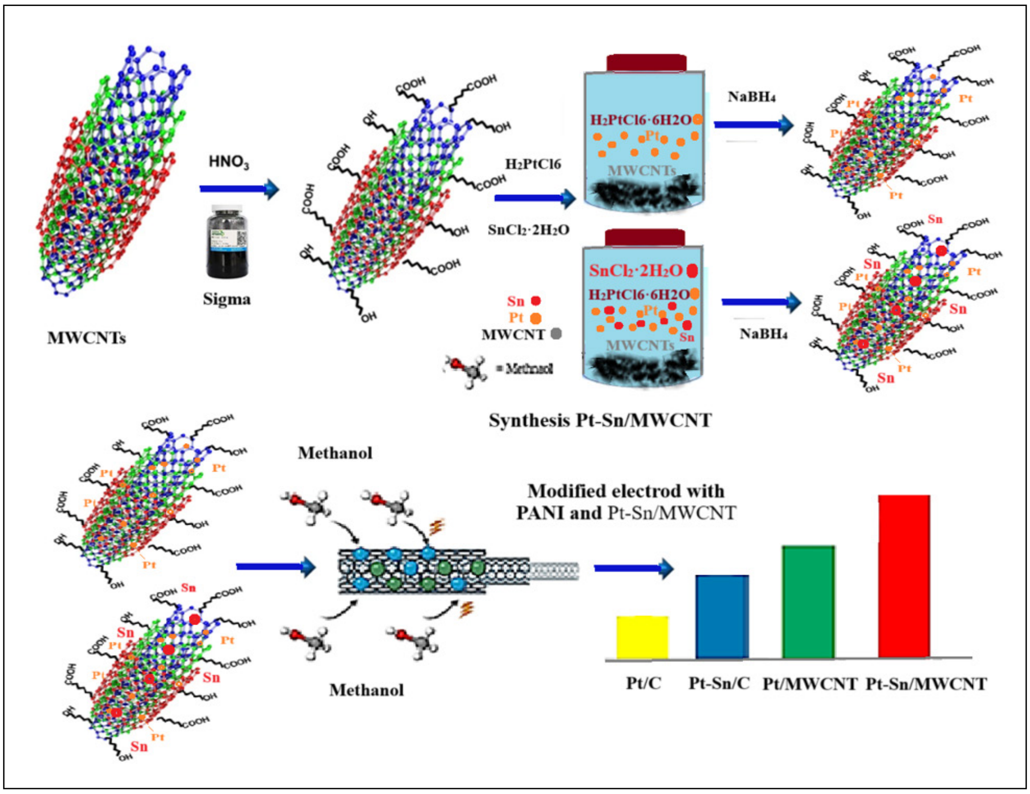
2.5. Electrode modification

The electrode was derivatized with Polyaniline (PANI) through electropolymerization of 0.04 M aniline in 0.5 M H_2SO_4. The electrode was cycled repeatedly from -0.2 to +0.9 V at 50 mV/s until the desired coverage of polymer was obtained (Schema 1).

3. Results and discussion

3.1. X-RAY diffractograms of the electrocatalysts

Figure 1 displays the X-ray diffractograms of the electrocatalysts. All of the PtSn catalysts' XRD patterns showed no peaks for Sn or its oxides. This indicates that Sn species occur in either an amorphous state or as diminutive crystallites [29]. Polycrystalline face-centered cubic (fcc) Pt is indicated by the three primary diffraction peaks of Pt (111), Pt (200), and Pt (220) at their corresponding diffraction positions, which are present in all samples. The (002) plane



Schema 1. Synthesis and Electrode modification with Polyaniline (PANI) and Pt-Sn/MWCNTS

of the carbon support material's hexagonal shape corresponds to the diffraction peak at 2θ ~ 25-26. [30]. Figure 1 indicates that the diffraction peaks of the binary electrocatalysts exhibit a shift to lower 2θ values for PtSn compared to the comparable peaks for Pt [31]. To find the average particle size, we used the broadening of the Pt (220) peak and applied Scherrer's equation. [32]. The size of the crystallites was calculated using the Scherrer formula, as shown in Equation 1.

$$\text{Crystallite size (D)} = \frac{k\lambda}{\beta \cos\theta} \qquad \text{(Eq.1)}$$

D stands for the size of the crystallite in nanometres, K denotes the form factor, which is 0.94, λ signifies the wavelength in angstroms, β indicates the full width at half maximum (FWHM) in radians, and θ refers to the diffraction angle in degrees.

The average particle size and lattice parameter are presented in Table 1, indicating the production of Pt-Sn particles measuring 5.9 to 7.3 nm. The measured fcc lattice parameter for Pt-Sn electrocatalysts exceeded that of Pt/C and Pt/MWCNT electrocatalysts, attributable to lattice expansion upon alloying, suggesting the incorporation of Sn into the fcc structure of Pt. The disparity in lattice parameters and the displacement of the (220) plane signify interactions between Pt and Sn [33].

3.2. FESEM images of the electrocatalysts

Figure 2 displays FESEM images of the Pt/C, Pt-Sn/C, Pt/MWCNT, and Pt-Sn/MWCNT catalysts, illustrating that the nanoparticles are equally distributed over the surfaces of the carbon supports. Photos taken using a field emission scanning electron microscope show that the catalyst is more durable when Sn is added, and that Sn close to Pt can produce oxygen-containing functional groups, which aid in the removal of CO intermediate molecules from the surface of Pt when electrooxidizing it with methanol. Methanol breakdown commonly occurs at platinum surface sites. In contrast, water decomposition transpires at tin surface sites, leading to the generation of oxygen-containing molecules that then react with carbon monoxide intermediates.

3.3. Cyclic voltammograms of electrocatalysts

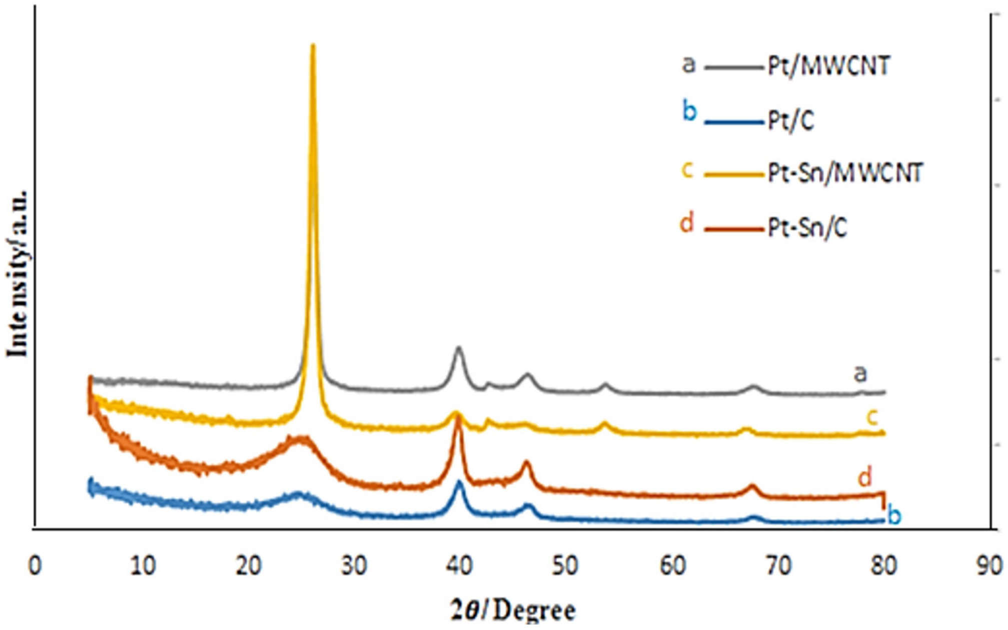


Fig. 1. XRD patterns of Pt/C, Pt-Sn/C, Pt/MWCNT, Pt-Sn/MWCNT

Table 1. Structural characteristic obtained from XRD

Catalyst	Particle size (nm)	Lattice parameter (nm)
Pt/C	9.55	0.391
Pt-Sn/C	7.34	0.392
Pt/MWCNT	6.3	0.391
Pt-Sn/MWCNT	5.95	0.394

Cyclic voltammetry tests were conducted at room temperature to examine the electrocatalytic activity of several catalysts in the oxidation of methanol. Figure 3 depicts the forward and reverse cyclic voltammetry profiles of MOR for Pt/C, Pt-Sn/C, Pt/MWCNT, and Pt-Sn/MWCNT in a 0.5 M CH_3OH + 0.1 M H_2SO_4 solution. To enhance the credibility of the results, the cyclic voltammogram data were repeated multiple times and shown after 100 scans. The amplitude of the forward anodic current peak indicates the efficacy of Pt catalysts in methanol oxidation. A standard method for measuring the catalytic activity of an electrocatalyst is by examining its peak potential, peak current density, and onset potential for oxidation. As illustrated in Figure 3, the produced Pt_ Sn/MWCNT catalyst exhibited a peak potential of around 0.55 V, which is lower than that of the Pt/C, Pt- Sn/C, and Pt/MWCNT electrocatalysts. The synthesized Pt-Sn/MWCNT exhibited the highest current density compared to other catalysts. This verifies the enhanced kinetics of methanol electro-oxidation in the presence of Sn and the influence of multiwall nanotubes as a support. Improved performance is dependent on the electrical conductivity and electron-transporting capabilities of the MWCNT support, which is enhanced when the MWCNTs are pretreated with concentrated HNO_3. The functionalization of the MWCNT surface with carboxylic acid groups provides an additional anchoring site for nanoparticles to adhere to the MWCNT surface. However, the addition of Tin (Sn) to a Pt-based alloy increased electrocatalytic activity. This is mainly because Sn is incorporated into the alloy system and its oxidized forms, which facilitates the process by lowering the oxidation potential [34].

The improved electrocatalytic activity and

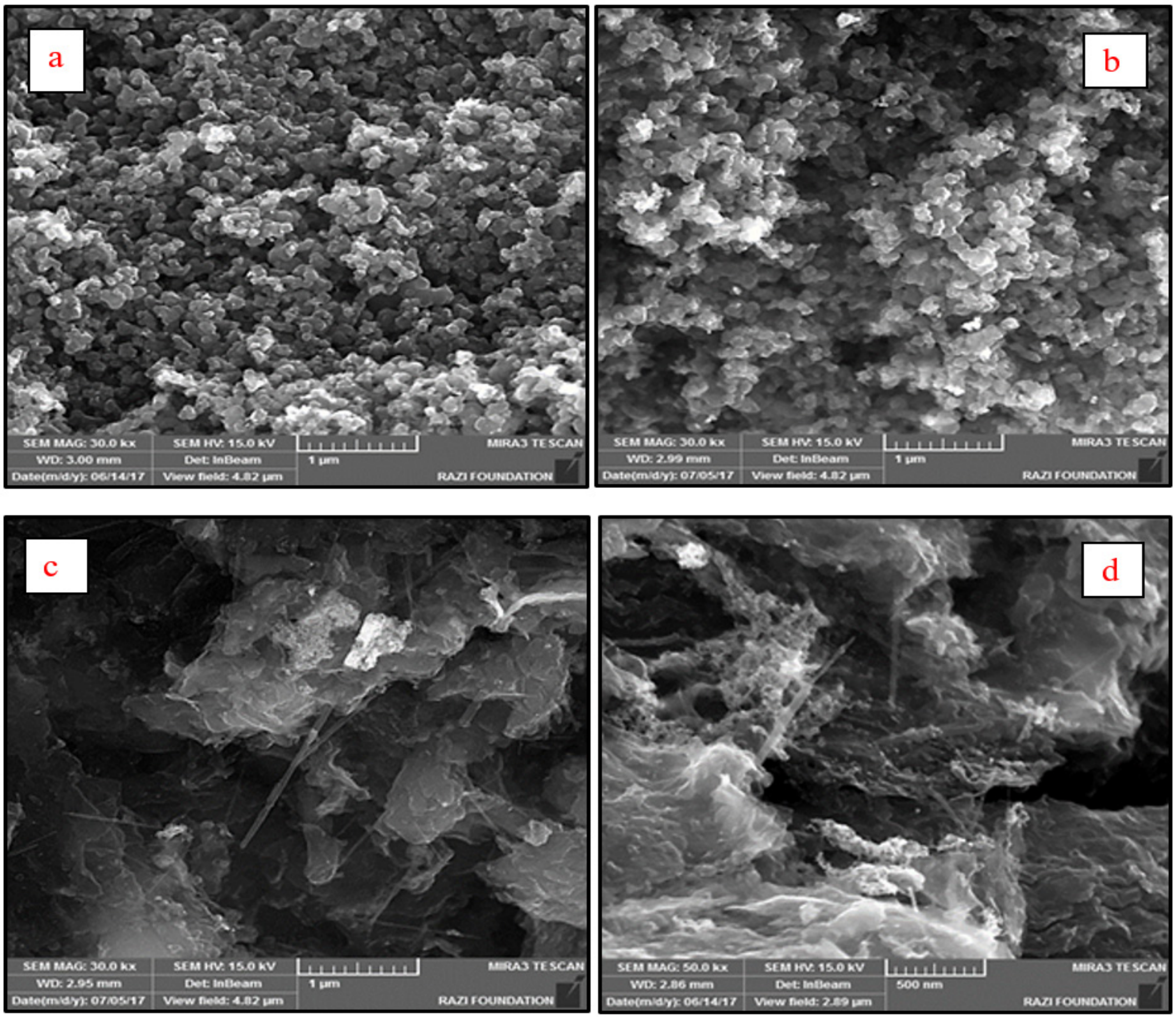


Fig. 2. The FESEM images of Pt/C (a), Pt-Sn/C (b), Pt-Sn/MWCNT (c), Pt/MWCNT(d).

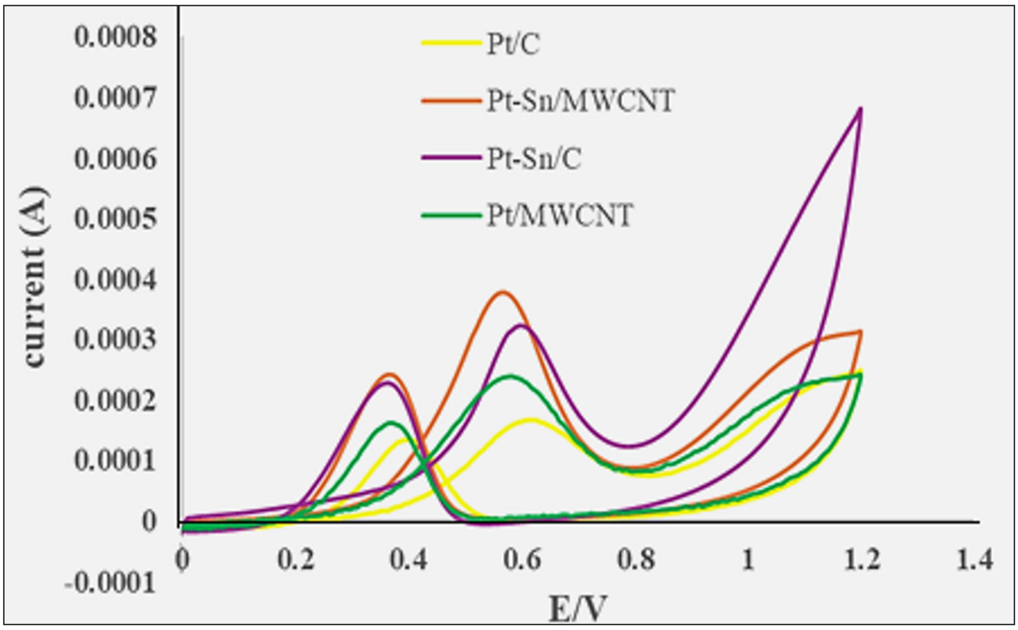


Fig. 3. CV$_s$ of Pt/C, Pt-Sn/C, Pt/ MWCNT , Pt-Sn/MWCNT in 0.5M CH_3OH and 0.1 H_2SO_4 (after 100 scans)

longevity of Pt/Sn catalysts for methanol and CO oxidation can be attributed to the bifunctional and ligand effects of the electrocatalyst, wherein methanol preferentially binds to Pt atoms and undergoes dehydrogenation to generate CO and intermediates, the primary poisoning agents on the Pt atoms (Eq.2). Because Sn or Sn oxides have a larger affinity for oxygen-containing species, water on these sites dissociates at lower potentials than on Pt sites (Eq.3a) and (Eq.3b), forming an oxygenated surface represented by the OHad species. This process facilitates the oxidation of adsorbed CO to CO2, hence mitigating CO poisoning (Eq.4a) and (Eq.4b), which enhances CO tolerance. Furthermore, Sn alters the surface chemical characteristics of Pt, resulting in diminished Pt-CO binding strength and so alleviating CO poisoning. Both processes enhance the catalytic activity and longevity of the Pt-Sn catalyst throughout the methanol oxidation process[35].

$$Pt + CH_3OH_{ad} \rightarrow Pt\_CO_{ad} + 4H^+ + 4e^- \qquad (Eq.2)$$

$$Sn + H_2O \rightarrow Sn\_OH_{ad} + H^+ + e^- \qquad (Eq.3a)$$

$$SnO_2 + H_2O \rightarrow SnO_2\_OH_{ad} + H^+ + e^- \qquad (Eq.3b)$$

$$Pt\text{-}CO_{ad} + Sn\_OH_{ad} \rightarrow CO_2 + H^+ + e^- \qquad (Eq.4a)$$

$$Pt\_CO_{ad} + SnO_2\_OH_{ad} \rightarrow CO_2 + H^+ + e^- \qquad (Eq.4b)$$

By dividing the forward anodic peak current (If) by the backward anodic peak current (Ib), the catalyst's tolerance to surface-accumulated incompletely oxidized species can be described, as is well known [35-39]. A larger ratio suggests that the toxic species are being removed from the catalyst surface more effectively. In Table 2, the ratio of If/Ib for Pt_Sn/MWCNT is only 1.6 m A, which is higher than that of other catalysts. Figure 4 displays the cyclic voltammogram of the GC/PANI/Pt-Sn/MWCNT in a 0.5 M CH3OH + 0.1 M H2SO4 solution. According to the findings, a multiwall carbon nanotube supported on a thin layer of polyaniline and platinum-tin electrocatalytic particles significantly improves the oxidation

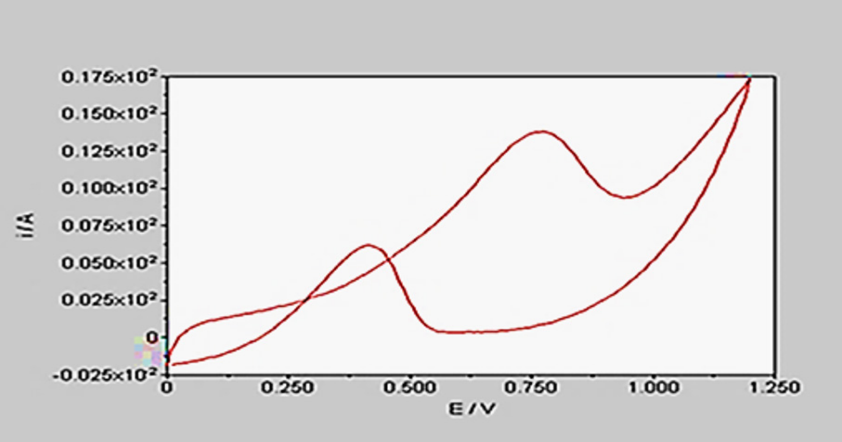


Fig. 4. CV of GC/PANI/ Pt-Sn/MWCNT in 0.5M CH_3OH & 0.1 M H_2SO_4, scan rate of 50 mVs^{-1} (after 100 scans)

Table 2. Comparing the CV_s of synthesized electrocatalysts

Catalyst	I_f (mA)	I_b (mA)	E_f (V)	I_f/I_b (mA)
Pt and C	0.16	0.125	0.62	1.28
Pt-Sn and C	0.3	0.225	0.59	1.33
Pt/MWCNT	0.23	0.160	0.57	1.40
Pt-Sn/MWCNT	0.37	0.230	0.55	1.60

of methanol in acidic water. This study indicates that the forward peak current density of the GC/PANI/Pt-Sn/MWCNT is 1/2 mA, surpassing that of platinum and platinum-tin electrocatalysts based on multiwall carbon nanotubes and Vulkan carbon. The activity for methanol oxidation was significantly increased by alloying platinum with tin, adding polyaniline, and adding multiwall carbon nanotubes. This catalytic improvement can be attributed to the following factors: The augmentation of the lattice parameter due to PtSn alloys facilitated the adsorption and dissociation of methanol, hence enhancing the activity of methanol oxidation at reduced potentials. (ii) The generation of OH groups via water activation on Sn species occurs at lower overpotentials compared to Pt, which is crucial for the oxidative elimination of adsorbed CO; consequently, the oxidation of adsorbed CO on the PtSn electrocatalyst can occur at reduced potentials or increased current densities relative to pure Pt [12], [40].

4. Conclusion

Platinum and platinum-tin nanoparticles on multi-walled carbon nanotubes and Vulkan carbon have been effectively produced by a deposition-reduction method with sodium borohydride. The findings indicate that GC/PANI/Pt-Sn/MWCNT is a superior electrocatalyst for methanol oxidation relative to the other catalysts examined in this study. Sn species enhance the kinetics of OH ads synthesis and increase the electrode's tolerance to poisoning species, particularly CO ads. Both a mesoporous network that allows catalyst particles to access and a conductive substance that enhances electron transport between nanotubes and catalyst particles are provided by the nanotubular materials. Porous polyaniline with a high surface area enhances catalytic activity for methanol oxidation.

5. Acknowledgment

We thank and appreciate the Faculty of Chemical Engineering, School of Engineering, University of Tehran, Tehran, Iran.

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