

Article

Efficient Electro-oxidation of 2-propanol at Platinum and Gold-modified Palladium Nanocatalysts

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Abstract: This study aims at investigating the catalytic performance of Pd, Pd/Pt, and Pd/Au nanocatalysts toward the 2-propanol electro-oxidation reaction (2POR) in an alkaline medium. The catalyst components (Pd, Pt, and Au) were sequentially electrodeposited onto a glassy carbon (GC) electrode surface and further characterized using electrochemical (cyclic voltammetry (CV)) and materials (Field-emission scanning electron microscopy (FE-SEM) coupled with energy dispersive X-ray (EDX)) characterization methods. The Pd/Au/GC catalyst showed the highest catalytic activity in terms of the highest oxidation current (0.386 mA) and the highest stability in terms of the highest obtained current after 1800 s of continuous electrolysis. This behaviour was attributed to the enhancement in the charge transfer kinetics where the Pd/Au/GC catalysts acquired the lowest charge transfer resistance (R_{ct} , 1.85 k Ω) during the 2POR.

Keywords: Fuel cells; 2-Propanol electro-oxidation; Pd; Poisoning; Electrocatalyst

1. Introduction

The energy dilemma is undeniably one of the most pressing concerns confronting modern society, as it plays a critical role in global and domestic economic development and security [1,2]. Due to the overwhelming increase in global population, coupled with the continuous economic growth seen in multiple developed and developing nations, an increase in global energy demand and consumption is unavoidable [3,4]. To address these concerns, cost-effective, clean, and renewable energy sources that could be used as viable substitutes for fossil fuels are urgently needed [5-11]. Energy storage and conversion devices have received worldwide interest as a key step toward the use of renewable energy resources as primary energy sources, and it has become necessary to thoroughly examine the various promising energy conversion and storage solutions, such as fuel cells (FCs) [12-18], and embark on their implementation [4,15]. In this regard, the direct alcohol fuel cells, such as the direct 2-propanol FCs (D2PFCs), appeared as excellent candidates [19,20].

In fact, 2-propanol is an excellent solvent and is involved in many industry processes such as cosmetics and pharmaceutical industries. Being twice as effective as ethanol, 2-propanol is the widely used disinfectant within pharmaceuticals, hospitals, cleanrooms, and electronics or medical device manufacturing [21]. It's also used as a gasoline additive to keep carburetors from freezing up in internal combustion vehicles [22]. The higher gravimetric energy density (8.6 kWh/kg) compared with that of methanol (6.1 kWh/kg) and ethanol (8.0 kWh/kg) is one more advantage for 2-propanol as a liquid fuel [23].

Recently, interests have been more dedicated to alkaline than acidic D2PFCs because of their higher performance which is likely resulted from the faster kinetics of the alcohol electrooxidation and the oxygen electroreduction in alkaline medium [24-26]. As the alkaline 2POR proceeds at low overpotential via the acetone production pathway ($\text{CH}_3\text{CHOHCH}_3 + 2\text{OH}^- \rightarrow \text{CH}_3\text{COCH}_3 + 2\text{H}_2\text{O} + 2\text{e}^-$) [26-28], several noble metals, including Pt, Pd, and Au, that exhibited previously excellent catalytic properties for several vital processes, have been recommended for 2POR [5,12,29-33]. However,

the obtained oxidation currents (I_p) decayed dramatically at low overpotentials which implies the rapid poisoning of the catalyst surface from the oxidation products [26,34,35]. Acetone was the suspicious poisoning species, as previously proposed for 2POR at Pd surfaces in alkaline medium [36].

This research addresses the impact of modifying a nanoparticle-based Pd surface with Pt and Au nanoparticles to test their potential to mitigate the activity deterioration of the Pd substrates during 2POR due to acetone poisoning. Three different catalysts (Pd/GC, Pd/Pt/GC, and Pd/Au/GC) will be prepared, characterized, and catalytically evaluated toward 2POR. The relationship between the concentration of electrolyte medium (NaOH) and the potential scan rate on the I_p will be obtained and the mechanism of enhancement toward the 2POR will be discussed.

2. Experimental

A cleaned GC ($d = 5.0$ mm), an Ag/AgCl/NaCl (3M), a spiral Pt wire were served, respectively, as the working, reference, and counter electrodes. The cleaning of the GC electrode was carried out through the conventional cleaning procedures described previously [37,38].

All chemicals in the current study were of high purity and were used without any prior purification. The electrodepositions of Pd, Pt, and Au were all carried out in 0.1 M Na_2SO_4 solutions containing, respectively, 2.0 mM $\text{Pd}(\text{CH}_3\text{COO})_2$, 2.0 mM $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$, 2.0 mM $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ at 0.1 V, allowing the passage of 10 mC.

All electrochemical experiments were conducted at room temperature (25 ± 1 °C) in a two compartments three electrodes glass cell using a Bio-Logic SAS potentiostat (model SP-150) operated with EC-Lab software.

The surface morphology and composition of the proposed catalysts were obtained using a Zeiss Ultra 60 field emission scanning electron microscope (FE-SEM) equipped with an energy-dispersive X-ray spectroscopy (EDX).

3. Results and discussion

3.1. Electrochemical and Materials characterization

Figure 1 shows the typical behaviour of Pd-based catalysts in alkaline media where the Pd oxidation to PdO which extended from -0.2 to 0.6 V was coupled with the subsequent reduction of PdO to Pd again at -0.4 V. Along with that, the hydrogen adsorption/desorption ($H_{\text{ads/des}}$) region was observed between -0.6 and -0.9 V. This behaviour was observed for all proposed catalysts (Fig. 1a-d). A deeper inspection of Fig. 1 reveals several other important observations:

- Compared to the bulk Pd catalyst (Fig. 1a), a larger surface area (SA) was obtained at the nanoparticles-modified catalysts (Fig. 1b-d). The SA was calculated to be 0.08, 0.52, 0.59, and 0.83 cm^2 for the Pd, Pd/GC, Pd/Pt/GC, and Pd/Au/GC catalysts, respectively, based on the charge associated with the PdO reduction peak using a reference value of $420 \mu\text{C cm}^{-2}$ [38,39]. This trend appeared again in the $H_{\text{ads/des}}$ region because of the large surface area offered by nanoparticles.
- The Pd/Pt/GC (Fig. 1c) and the Pd/Au/GC (Fig. 1d) catalysts acquired a broader PdO reduction peak compared to that obtained at the Pd/GC catalyst (Fig. 1a). This highlighted the role of adding the Pt and Au surface modifiers in providing diverse Pd-Pd and Pd-O bonding and/or facets' reconstruction for the Pd surface.
- The large $H_{\text{ads/des}}$ peaks at the Pd/Pt/GC catalyst referred to the participation of both Pd and Pt in this reaction [40].

- The disappearance of the Au characteristic peaks at the Pd/Au/GC catalyst (Fig. 1d) might refer to the formation of a “core (Au)-shell (Pd)” structure [40,41].

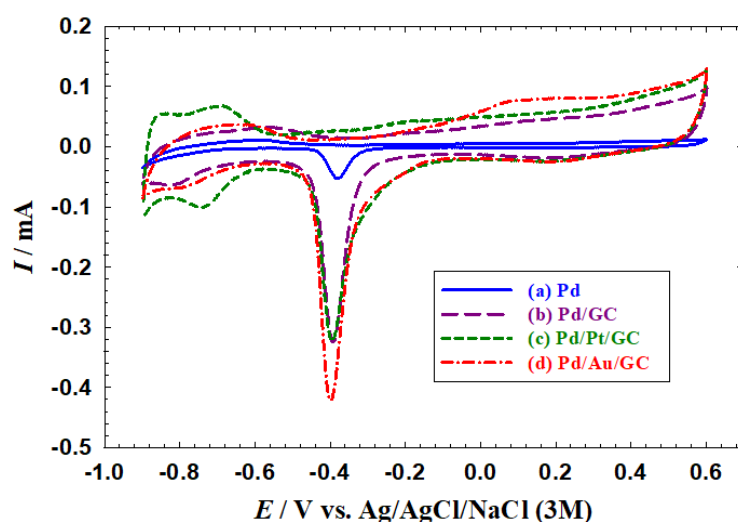


Figure 1. CVs obtained at the (a) Pd, (b) Pd/GC, (c) Pd/Pt/GC, and (e) Pd/Au/GC catalysts in 0.1 M NaOH. Potential scan rate: 100 mV s⁻¹.

Additionally, by using the state-of-art technologies, the surface morphology (Fig. 2) of the proposed catalysts; Pd/C (Fig. 2A), Pd/Pt/GC (Fig. 2B), and Pd/Au/GC (Fig. 2C) was obtained using the FE-SEM. Figure 1A confirmed the successful deposition of Pd in the Pd/GC catalyst as well-distributed spherical nanoparticles having an average diameter of ca. 30 nm. Too larger sizes (100 and 120 nm) of spherical Pd particles were obtained, respectively, at the Pd/Pt/GC (Fig. 2B) and the Pd/Au/GC (Fig. 2C) catalysts. It seems that Pt and Au were simultaneously deposited with Pd in single bigger particles [37,42].

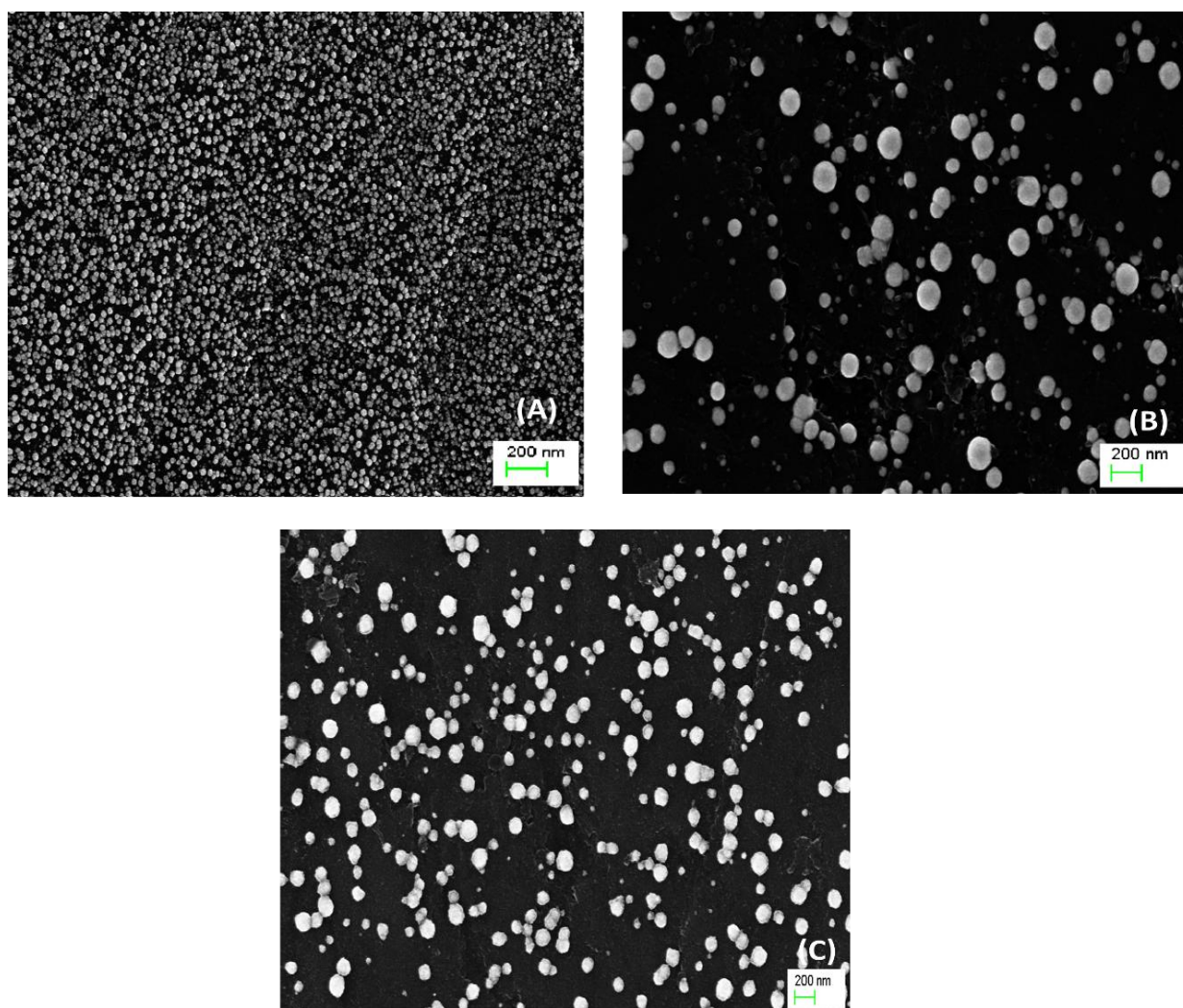


Figure 2. FE-SEM images of the (A) Pd/GC, (B) Pd/Pt/GC, and (C) Pd/Au/GC catalysts.

To further confirm the successful deposition of all ingredients of the proposed catalysts, EDX analysis was obtained. Figure 3 shows the EDX analyses of the Pd/GC (Fig. 3a), Pd/Pt/GC (Fig. 3b), and the Pd/Au/GC (Fig. 3c) catalysts. As clearly shown, all catalysts' components (C, O, Pd) were existed in the three proposed catalysts additionally with Pt and Au that were observed respectively at the Pd/Pt/GC and the Pd/Au/GC catalysts.

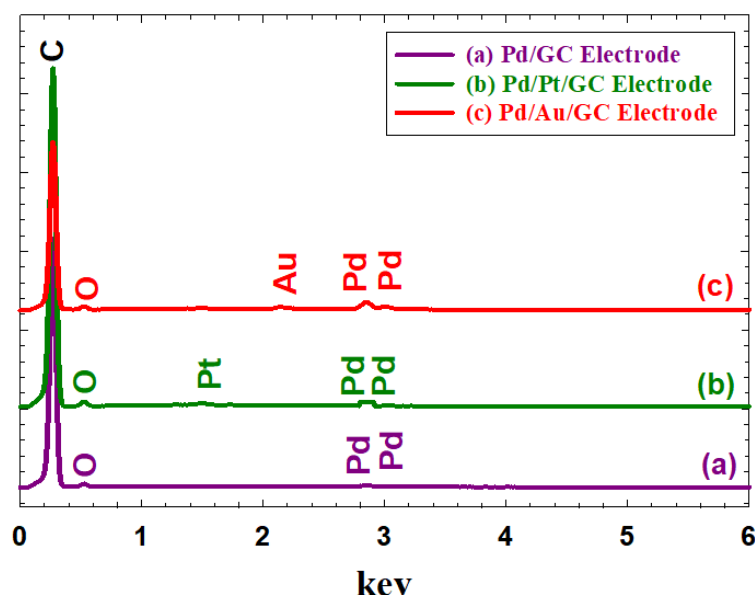


Figure 3. EDX analyses of the (a) Pd/GC, (b) Pd/Pt/GC, and (c) Pd/Au/GC catalysts.

3.2. Electrocatalytic activities of the catalysts toward EOM

Figure 4 shows the alkaline 2POR at the proposed catalysts. All catalysts showed the typical behaviour of the 2POR where two anodic peaks were observed at ca. -0.15 and -0.28 V, respectively, in the positive and the negative going scans, which agreed with previous literature [27,31,34]. The low oxidation current (I_p , ca. 0.029 mA) obtained at the Pd catalyst (Fig. 4a) was attributed to the surface poisoning with the strongly adsorbed oxidation product, acetone, at the Pd surface [36]. The poisoning with acetone was assumed to block most of the Pd active sites and therefore diminish the overall activity of the 2POR. A better scenario was observed at the Pd/GC (Fig. 4b) catalyst at which the I_p reached ca. 0.279 mA (ca. 10 times higher than that obtained at the Pd catalyst).

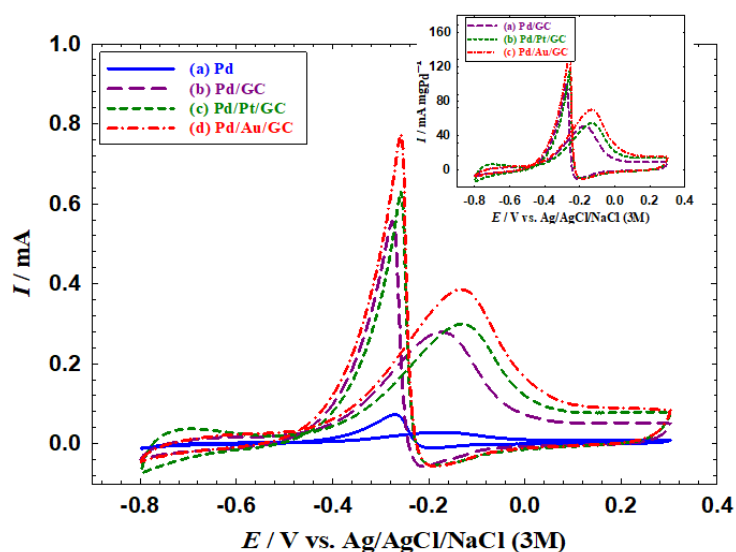


Figure 4. CVs obtained at the (a) Pd, (b) Pd/GC, (c) Pd/Pt/GC, and (d) Pd/Au/GC catalysts in 0.1 M NaOH solution containing 0.3 M 2-propanol. Potential scan rate: 100 mV s^{-1} . Inset shows the current values normalized to the Pd mass (specific currents) of the (a) Pd/GC, (b) Pd/Pt/GC, and (c) Pd/Au/GC catalysts.

The consecutive modification of the Pd/GC catalyst with Pt and Au could further diminish such a poisoning impact where the I_p value reached 0.299 and 0.386 mA respectively at the Pd/Pt/GC (Fig. 4c) and the Pd/Au/GC (Fig. 4d) catalysts. The inset of Fig. 4 shows the current values normalized to the mass of deposited Pd (specific currents) for the Pd/GC, Pd/Pt/GC, and the Pd/Au/GC catalysts. The obtained values were 51, 55, and 71 mA $\text{mg}_{\text{Pd}}^{-1}$, respectively.

The enhancement behavior in the catalytic activity was also reflected in measuring the catalytic stability. Figure 5 shows the current transients ($i-t$) of the proposed catalysts. The enhancement in the catalytic stability in terms of achieving higher currents after 1800s of continuous electrolysis. As observed, the current trend was in the order of Pd (Fig. 5a) < Pd/GC (Fig. 5b) < Pd/Pt/GC (Fig. 5c) < Pd/Au/GC (Fig. 5d).

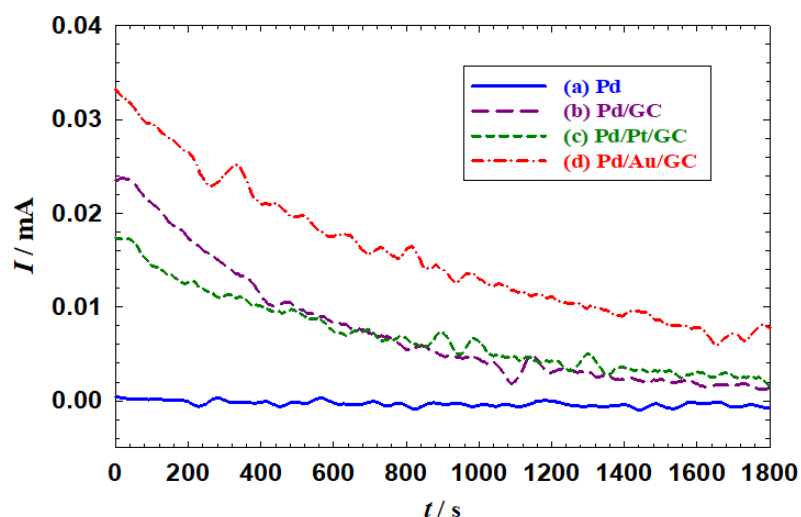


Figure 5. Current transients measured at -0.4 V at the (a) Pd, (b) Pd/GC, (c) Pd/Pt/GC, and (d) Pd/Au/GC catalysts in 0.1 M NaOH solution containing 0.3 M 2-propanol.

To understand the mechanism of enhancement toward the 2POR, in terms of the charge transfer, Nyquist plots were obtained and analysed (see Figure 6). As well-known, Nyquist plots assess the charge transfer resistance (R_{ct}) of the catalyst, which is obtained from the semicircle diameter; evaluating the reaction kinetics [37,40]. In agreement with the data of Figs. 4a and 5a depicting, respectively, the poor activity and stability of the Pd catalyst, Fig. 6a assigned the highest R_{ct} value (45.39 k Ω) for the same "pristine" Pd catalyst toward the 2POR. This indicates a sluggish kinetics toward the 2POR at the Pd catalyst.

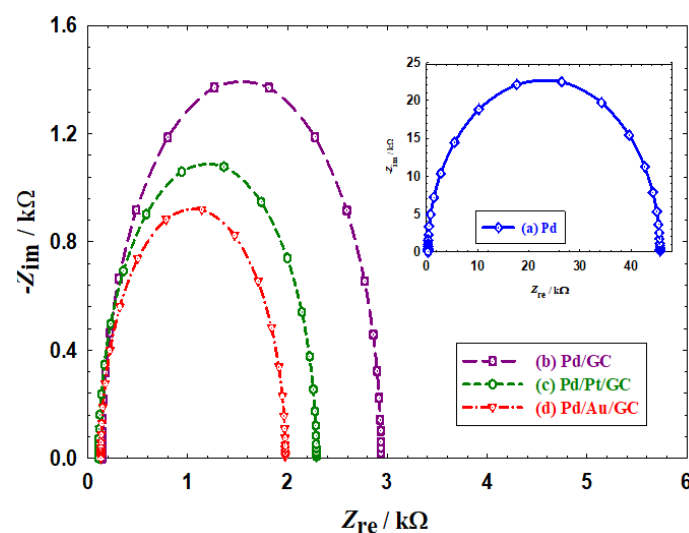


Figure 6. Nyquist plots obtained recorded at AC potential amplitude of -0.4 V obtained at the (a) Pd, (b) Pd/GC, (c) Pd/Pt/GC, and (d) Pd/Au/GC catalysts in 0.1 M NaOH solution containing 0.3 M 2-propanol. Frequency ranged from 10 mHz to 100 kHz.

On the other hand, the Pd/GC (Fig. 6b), Pd/Pt/GC (Fig. 6c), and the Pd/Au/GC catalysts offered much lower R_{ct} values (2.79 , 2.18 , and 1.85 $k\Omega$, respectively); indicating faster charge transfer kinetics toward the 2POR. It is thought that such an enhancement came from the weak adsorption of acetone and/or its faster removal at the modified catalysts [26].

3.3. Parameters affecting 2POR

To achieve a better electrocatalytic 2POR, the effect of changing the NaOH concentration and scan rate were examined. Figure 7A-D shows the effect of changing the NaOH concentration on the I_p value of the 2POR at all proposed catalysts. It was obvious that increasing the NaOH concentration increases the I_p value in a linear way with a correlation coefficient of ca. 0.98 . It seems that increasing the OH^- concentration facilitated the removal of the adsorbed intermediates that increased the availability of more active sites for the 2POR [43].

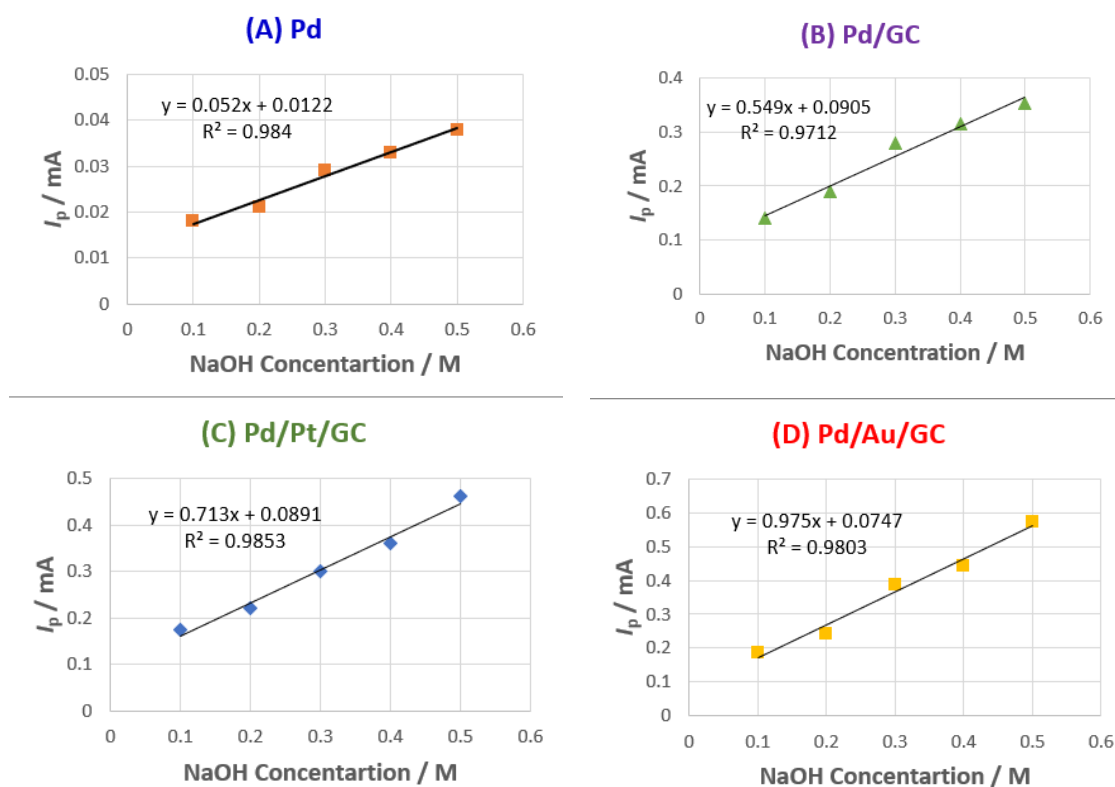


Figure 7. The linear correlation between I_p and NaOH concentration at the (A) Pd, (B) Pd/GC, (C) Pd/Pt/GC, and (D) Pd/Au/GC catalysts. The CVs measured for 2POR at 100 mV s⁻¹ in NaOH with different concentrations containing 0.3 M methanol (pH = 3.5).

Additionally, Fig. 8A-D shows the effect of changing the scan rate on the I_p value of the 2POR at all proposed catalysts. It was clear that a slight shift of the I_p to more positive values was observed with increasing the potential scan rates, suggesting a kinetic limitation [44]. Moreover, as the scan rate increases, the I_p value increases and a linear dependence of the square root of the scan rate and the I_p was observed with a correlation coefficient ranging from 0.95 to 0.99, which confirmed the diffusion-controlled process [45].

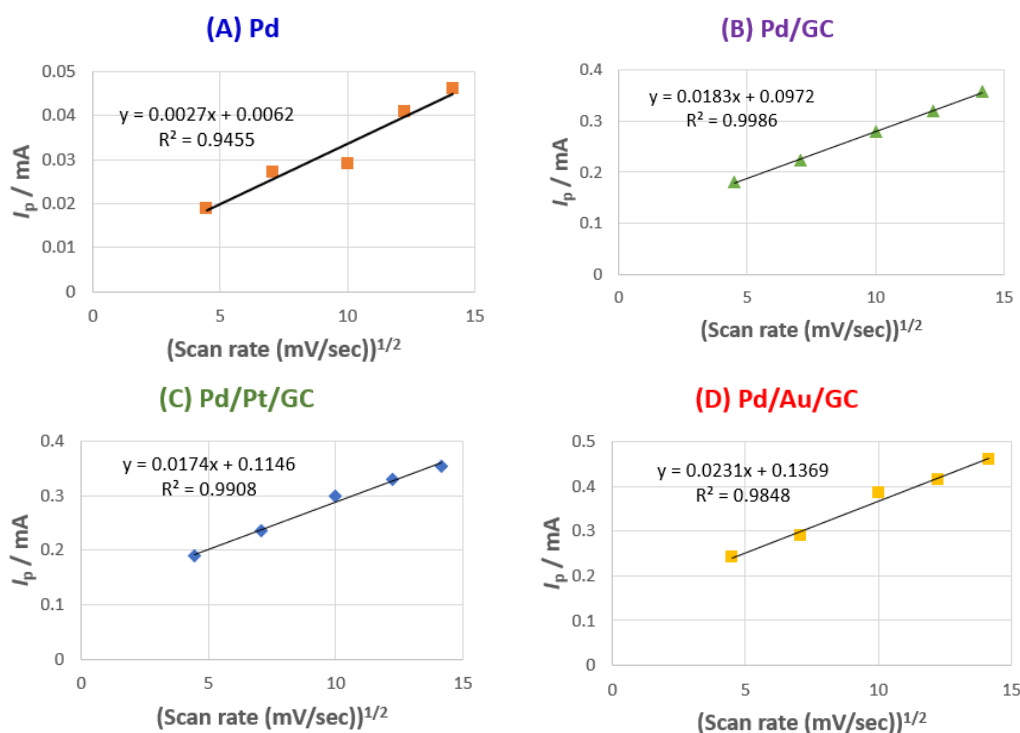


Figure 8. The linear correlation between I_p and the square root of the potential scan rate at the (A) Pd, (B) Pd/GC, (C) Pd/Pt/GC, and (D) Pd/Au/GC catalysts. The CVs measured for 2POR in 0.1 M NaOH containing 0.3 M methanol (pH = 3.5).

4. Conclusion

This research aimed at examining the electro-catalytic performance toward 2POR in an alkaline medium at several proposed (Pd/GC, Pd/Pt/GC, and Pd/Pt/GC) nanocatalysts. The Pd/Au/GC catalyst showed the highest catalytic activity and stability in terms of the highest current values. Nyquist plots assessed the charge transfer enhancement where the Pd/Au/GC catalyst showed the fastest charge transfer kinetics (the lowest R_{ct} value) toward the 2POR. From another perspective, the effects of changing the potential scan rate and the NaOH concentration on the oxidation currents were monitored and several mechanistic correlation were deduced in view of the recommended modifications at all the proposed catalysts.

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Data Availability: The data presented in this study are available on request from the corresponding author.

Conflict of Interest: The authors declare no conflict of interest.

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