



# Fabrication of novel palladium-platinum based graphene/ITO electrodes and third metal addition effect through the glucose electrooxidation



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## ABSTRACT

Graphene was coated on Cu foil by chemical vapor deposition (CVD) method. The graphene on the Cu foil was modified by doping N. Then, N-doped graphene (G) was coated on several layers of indium tin oxide (ITO) electrodes. In addition, Pd, Pt, and M (Ag, V, Ni, Zn) metals were electroprecipitated on the graphene/indium tin oxide electrode by electrochemical technique. In this way, the glucose (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>) electrooxidation activities of these electrodes obtained from PdMPT-N doped graphene/indium tin oxide were investigated by cyclic voltammetry (CV), chronoamperometry (CA), and electrochemical impedance spectroscopy (EIS) measurements. The obtained materials were characterized by SEM-EDX. Results revealed that the network of Pd, Pt, Ag, V, Ni, Zn and graphene was clearly visible from the SEM results. As a consequence, PdZnPt-N doped G/ITO showed the most effective C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> electrooxidation activity with a specific activity of 14.5 mA cm<sup>-2</sup>, considerably above the literature's published values. In all electrochemical measurements, PdZnPt-N doped G/ITO exhibited the best electrocatalytic activity, stability, and resistance. PdZnPt-N doped G/ITO electrode is promising electrode for glucose electrooxidation.

## 1. Introduction

The demand for energy is increasing rapidly all over the world day by day. Researchers have turned to alternative energy sources due to the limited use of fossil energy sources and the damage to the environment as a result of the use of these energy sources. In this context, fuel cells attract the attention of the scientific community as one of the most crucial future energy sources. Fuel cells are components that directly convert chemical energy to electrical energy. For mobility, portable electronic gadgets, and stationary power generation, fuel cells are employed as electrical power sources [1]. The rise of the low-temperature direct glucose fuel cell (DGFC) demonstrates the DGFC's growing importance in fuel cell technology and applications [2,3]. Glucose is the most abundant simple sugar in nature with its toxic, flammable and non-volatile superior properties [4,5]. Alloying is one of the best known methods to increase the catalytic activity of monometallic catalysts. Various types of electrocatalysts have been

reported for glucose electrooxidation. Some of the Pd-based alloy electrocatalysts in the literature are; Pt-Pd [6], Pd-Ni [7–9], Pd-Fe [10], Pd-Pt-Au [6], or Pd-Rh [11]. Also, commonly studied metals for glucose electrooxidation are Pt and Au [12–15]. Platinum is used as an electrocatalyst in fuel cell applications due to its high activity and chemical stability [16]. Graphene offers various advantages, including a single atomic layer structure and a large surface area. With its vast area, high quality synthesis, and layer controllability capacity, the chemical vapour deposition (CVD) production method of graphene is superior in many respects compared to other methods documented in the literature [17]. Several-layer graphene nano-hybrids are generated by CVD technique, and these nanoparticles are used in glucose electrooxidation, according to the literature [17–20].

The bimetallic nanoparticle feature fast electrochemical oxidation and a unique synergistic contribution, which enhance conductivity and accelerate electron transfer. In this study in the literature, a catalytically active area was increased through the formation of Au-Pt

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bimetallic nanoparticle morphology [21,22]. Therefore, in this study, it is thought that adding the third metal will increase the synergistic effect between the metals.

The CVD technique was used to coat *N*-doped-Graphene on Cu foil in this investigation. After that, an indium tin oxide (ITO) electrode was coated with *N*-doped-Graphene on Cu foil. Then, Pd metal and second metals (Ag, Ni, Zn, V) were electrochemically precipitated on the *N*-doped-G/ITO electrode by electrochemical technique. In this way, bimetallic electrocatalysts and trimetallic electrocatalysts were obtained by adding Pt metal. SEM-EDX analysis was used to characterize the prepared electrodes. Cyclic voltammetry (CV), chronoamperometry (CA) and electrochemical impedance spectroscopy (EIS) measurements were used to investigate the glucose ( $C_6H_{12}O_6$ ) electrooxidation activities of PdM-N doped G/ITO and PdMPt-N doped G/ITO electrodes.

## 2. Materials and methods

### 2.1. Materials

As received, glucose (Merck) and KOH (Merck, 84 percent purity) were employed. All other compounds were analytical grade and were used just as they were. Throughout the studies, distilled water was used.

### 2.2. Preparation of electrodes

First, a few-layer graphene was prepared by the CVD method. *N*-doped graphene was then prepared. The *N*-doped graphene was transferred to the ITO surface. All these production steps were performed as reported in the literature [17,18]. In this way, bimetallic and trimetallic PdM-N G/ITO and PdMPt-N G/ITO electrode materials were prepared and stored for use in experiments.

### 2.3. Surface characterization

The surface of the PdM-N doped G/ITO and PdMPt-N doped G/ITO electrode materials was characterized by SEM-EDX analysis. With the EDX results, the metal ratios in the electrode materials were determined. A FEI QUANTA 250 FEG scanning electron microscope was used to perform SEM-EDX experiments.

### 2.4. Electrochemical measurements

The activity of PdM-N doped G/ITO and PdMPt-N doped G/ITO catalysts coated on the glassy carbon electrode was investigated in 1 M KOH + 0.5 M  $C_6H_{12}O_6$  solution in a three-electrode system. The glucose electrooxidation performance of analysis were made using cyclic voltammetry (CV) method, chronoamperometry (CA) method, and electrochemical impedance spectroscopy (EIS). CHI 660E device was used for electrochemical measurements. The working electrode was glassy carbon, the reference electrode was Ag/AgCl, and the counter electrode was platinum wire. Cyclic voltammetry measurements were performed between  $-0.6$  V and  $0.5$  V.

## 3. Results and discussion

### 3.1. Surface characterization

SEM images of bimetallic PdAg-N doped G/ITO, PdZn-N doped G/ITO, PdNi-N doped G/ITO and PdV-N doped G/ITO catalysts are given in Fig. 1s(a–d). As illustrated in Fig. 1(a–l), the Pd, M (Ag, Zn, Ni, V) and carbon network were clearly visible. As shown in Fig. 1(e, f, g, h, i), the formation of the PdM-N doped-G/ITO electrode has been proven by mapping and as seen in Fig. 1(a, b, c, d), it is seen that the Pd metal is embedded in the N doped-G/ITO. In Fig. 1s(e–k), the yellow, violet, turquoise, orange, pink, yellowish orange and green particles were demonstrated by mapping analysis to be C, N, Pd, Ag, V, Ni and Zn, respectively. EDS analysis result of bimetallic PdM-N doped G/ITO catalysts is given in Fig. 1s–l and elemental analysis is given on mapping analysis.

SEM analysis was performed to obtain information about the composition of the synthesized catalysts. EDX analysis was used to define the elemental composition of these catalysts. SEM images of PdMPt-N doped G/ITO catalysts are given in Fig. 2s(a, d). In Fig. 2s(e, l), mapping images of the elements in these catalysts are given. EDX graph of elemental compositions is given in Fig. 2s and Table 1. In Fig. 2s(e, l), yellow, purple, blue, orange, pink, light orange, green, light green are shown by mapping analysis of C, N, Pd, Ag, V, Ni, Zn and Pt particles, respectively. When the SEM images of Fig. 1s(a–d) and Fig. 2s(a–d) are compared, the surface has a smoother structure as a result of the addition of Pt to bimetallic catalysts. Therefore, we can say that the trimetallic electrode gives better results in glucose electrooxidation.

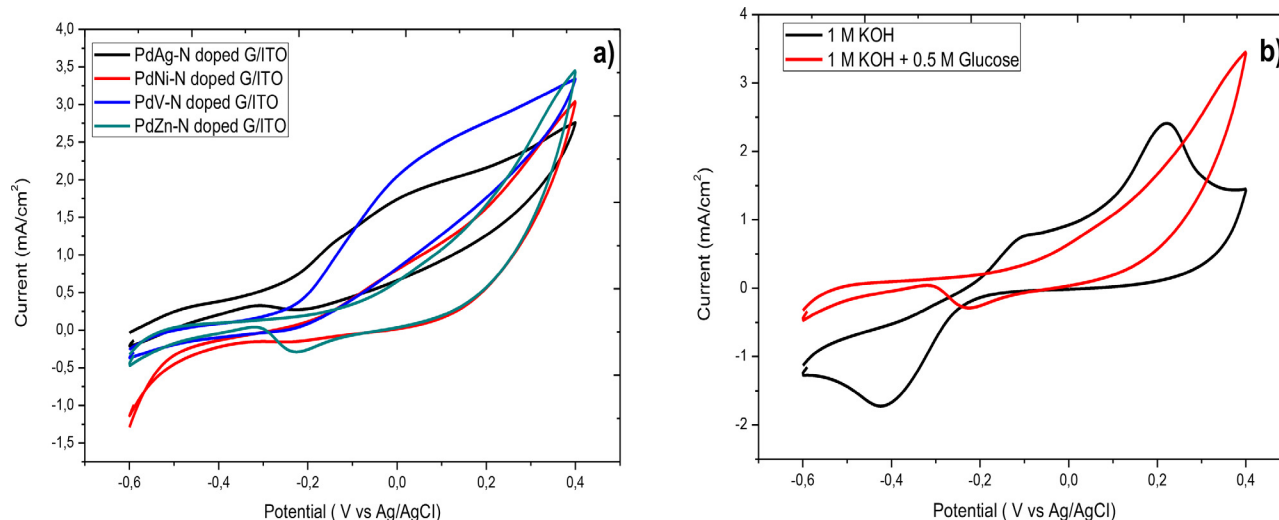


Fig. 1. The CV results obtained in (a) PdM(Ag, Ni, V, Zn)-N doped Graphane/Indium Tin Oxide electrodes in 1 M KOH + 0.5 M glucose solution (b) PdZn-N doped Graphane/Indium Tin Oxide electrode in 1 M KOH and 1 M KOH + 0.5 M glucose solution at 50 mV/s.

**Table 1**  
Elemental analysis of PdMPt-N doped G/ITO catalysts.

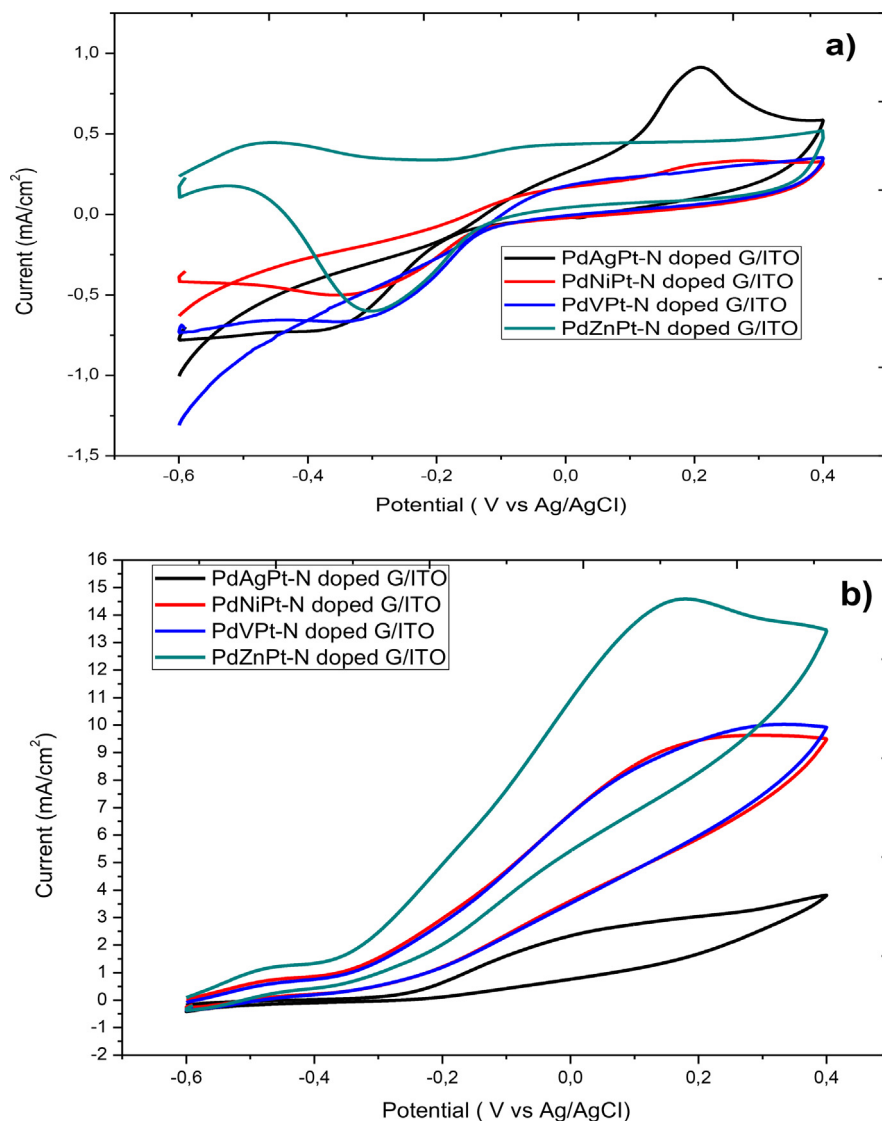
	Elements (%)		Samples	
	PdAgPt-N doped G/ITO	PdVPt-N doped G/ITO	PdNiPt-N doped G/ITO	PdZnPt-N doped G/ITO
C	34.71	33.70	14.98	12.87
N	55.10	61.30	75.19	76.99
Pd	2.50	2.68	2.73	2.83
Pt	1.96	1.66	4.09	–
Ag	5.73	–	–	–
V	–	0.66	–	–
Ni	–	–	3.01	–
Zn	–	–	–	7.32

The network of Pd, Pt, Ag, V, Ni, Zn and graphene is clearly visible from the SEM results.

### 3.2. Electrochemical measurements

The catalytic activity, stability, and resistance of PdM(Ag, Ni, V, Zn)-N doped G/ITO and PdMPt-N doped G/ITO electrodes were investigated to CV, CA, and EIS electrochemical analyses in 1 M KOH + 0.5 M glucose solution for glucose electrooxidation,

respectively. Fig. 3a, b demonstrates CV measurements of PdM-N doped G/ITO electrodes. Glucose electro-oxidation peaks are associated with increased current density [16]. It could be seen from Fig. 3a, PdZn-N doped G/ITO had better catalytic activity when compared to other PdM-N doped G/ITO electrodes at the total potential. Additionally, the PdZn-N doped G/ITO ( $3.45 \text{ mA/cm}^2$ ) electrode had higher specific activity than Pd-N doped-G/ITO ( $2 \text{ mA/cm}^2$ ) electrode obtained in our previous study [18]. Fig. 1b shows the results of the PdZn-N doped G/ITO electrode in 1 M KOH and 1 M KOH + 0.5 M



**Fig. 2.** The CV results obtained in a) 1 M KOH and b) 1 M KOH + 0.5 M glucose of PdMPt-N doped Graphane/Indium Tin Oxide electrodes at 50 mV/s.

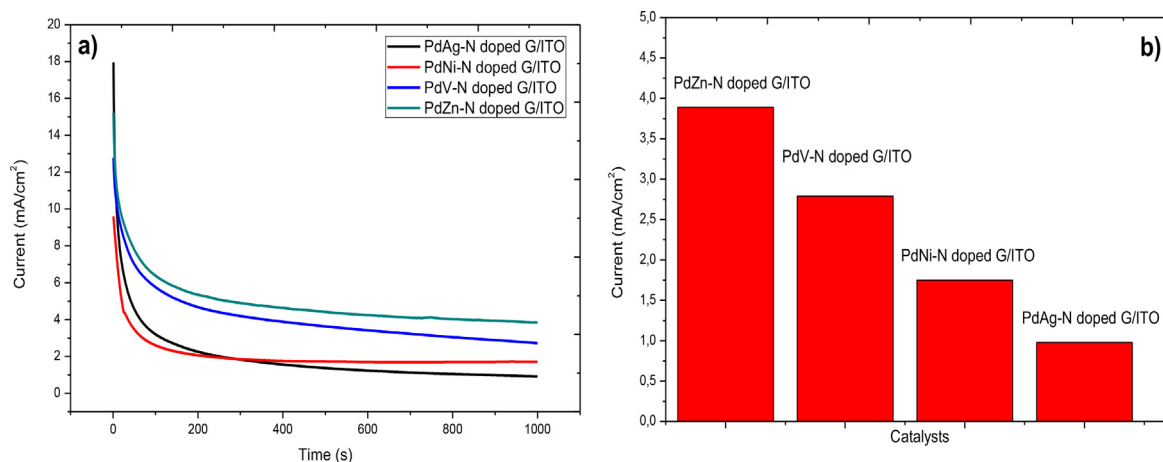


Fig. 3. CA graphics of (a) PdM-N doped Graphane/Indium Tin Oxide electrodes at  $-0.5$  V and (b) specific activities after 1000 s in 1 M KOH + 0.5 M glucose.

**Table 2**  
Electrochemical properties of PdMPt-N doped G/ITO electrodes for glucose electrooxidation.

Catalyst	Total Current (KOH, mA/cm <sup>2</sup> )	Specific Activity (Glucose, mA/cm <sup>2</sup> )	Forward Peak Potential, V	Onset Potential (V)
PdZn-N doped G/ITO	1.47	3.45	0.40	-0.19
PdAgPt-N doped G/ITO	0.58	3.82	0.40	-0.30
PdNiPt-N doped G/ITO	0.32	9.56	0.25	-0.37
PdVPt-N doped G/ITO	0.36	10.00	0.31	-0.38
PdZnPt-N doped G/ITO	0.52	14.58	0.18	-0.39

glucose solutions. The peak difference obtained in the total potential (Fig. 1b) or the peak obtained in the forward scan (Fig. 2b) shows electrooxidation resulting from glucose adsorption [23,24]. The CV analyses of PdMPt-N doped G/ITO electrodes were shown in Fig. 2a, b. As seen from Fig. 1b and 2a, the peak occurring in the about  $-0.5 \sim -0.1$  V potential range indicates the reduction peak of PdO [25]. Table 2 illustrates the total current (KOH), specific activity (glucose), forward peak, and onset potential. As seen in Table 1, the PdZnPt-N doped G/ITO electrode ( $14.58 \text{ mA/cm}^2$ ) exhibited the best electrocatalytic activity than the PdMPt-N doped G/ITO electrodes. In addition, it was obtained better activity about 4.2 and 7.3 times than PdZn-N doped G/ITO and previous study Pd-N doped-G/ITO,

**Table 3**  
Comparison of the specific activities of glucose electrooxidation electrodes in the literature.

Electrode	Specific Activity (mA/cm <sup>2</sup> )	Reference
Pd-N-dopedG/ITO	2	[18]
Graphene/ITO electrode	6.58	[26]
N-doped few-layers G/indium tin oxide	9.52	[17]
Ni <sub>2</sub> P/NF	6.37	[27]
Pt-graphene/ITO	9.21	[28]
PdZnPt-N doped G/ITO	14.5	This study

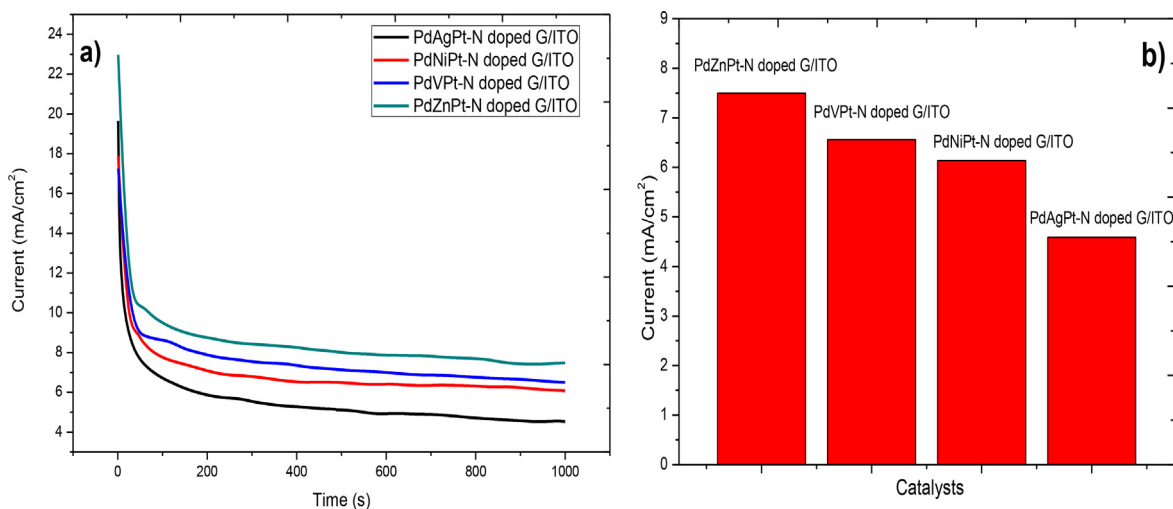
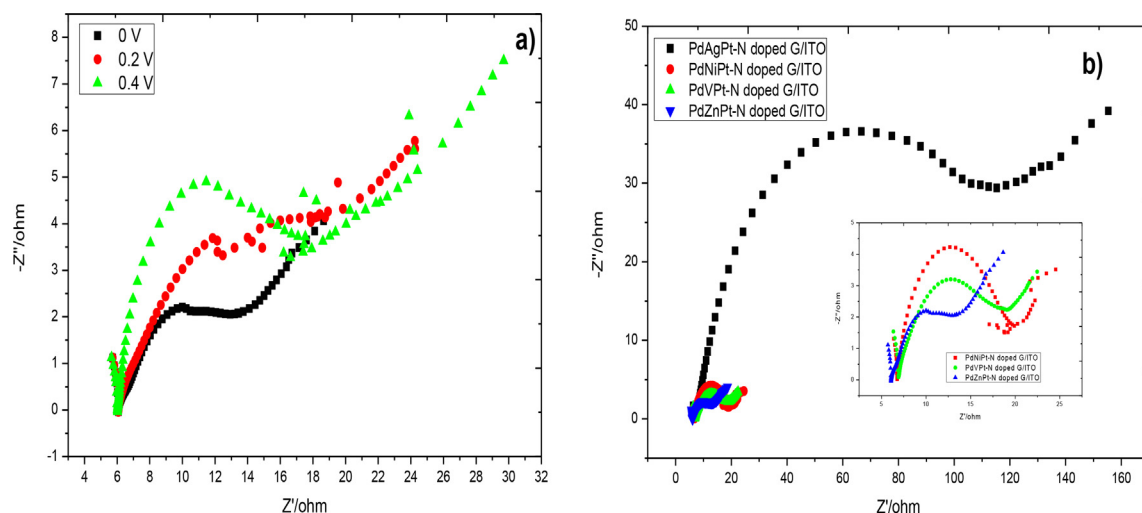


Fig. 4. CA graphics of (a) PdMPt-N doped Graphane/Indium Tin Oxide electrodes at  $-0.5$  V and (b) specific activities after 1000 s in 1 M KOH + 0.5 M glucose.



**Fig. 5.** Nyquist plots of (a) various potentials of PdZnPt-N doped Graphane/Indium Tin Oxide electrode and (b) comparison of PdMPt-N doped Graphane/Indium Tin Oxide electrodes at 0 V in 1 M KOH + 0.5 M glucose resolution.

respectively. As the activity increased, negative shifts occurred in the onset and forward peak potentials.

CA analysis was performed to measure the stability of PdM-N doped G/ITO and PdMPt-N doped G/ITO electrodes. Fig. 3(a, b) and Fig. 4(a, b) illustrates CA graphics of PdM-N doped G/ITO and PdMPt-N doped G/ITO electrodes, respectively. After 1000 s, PdZnPt-N doped G/ITO were better activity with 7.5 mA/cm<sup>2</sup> specific activity compared to PdZn-N doped G/ITO (3.9 mA/cm<sup>2</sup>), PdVPt-N doped G/ITO (6.5 mA/cm<sup>2</sup>), PdNiPt-N doped G/ITO (6.0 mA/cm<sup>2</sup>), and PdAgPt-N doped G/ITO (4.5 mA/cm<sup>2</sup>). As in the CV data, the PdZnPt-N doped G/ITO electrode exhibited the best activities and consistency compared to the other electrodes. The activity comparison of some electrodes used for glucose electrooxidation is given in Table 3. As indicated in the table, the specific capacity of the PdZnPt-N doped G/ITO electrode synthesized in this study is the highest.

Fig. 5(a, b) illustrates the Nyquist plots obtained from the EIS analysis to examine the electrocatalytic resistance of the electrodes. These plots are usually known as semicircles, where the electrocatalytic resistance increases as the diameter of the circle decrease [29,30]. The resistance to charge transfer ( $R_{ct}$ ) is associated with the diameter of the semicircle because as the diameter decreases,  $R_{ct}$  decreases, and so the catalytic activity increases [31]. Fig. 5a shows the Nyquist plots of the PdZnPt-N doped G/ITO electrode taken at 0.0, 0.2, and 0.4 V potentials. As seen in Fig. 5a, the Nyquist plot taken at the potential of 0.0 V showed the best electrocatalytic activity. It can be clearly seen from Fig. 5b that the  $R_{ct}$  could be listed as follows; PdAgPt-N doped G/ITO > PdNiPt-N doped G/ITO > PdVPt-N doped G/ITO > PdZnPt-N doped G/ITO. As a consequence, PdZnPt-N doped G/ITO showed the highest electrocatalytic activity as in CV and CA results.

#### 4. Conclusions

At present study, Pd, Pt, and M (Ag, V, Ni, Zn) doped on graphene/ITO electrode by electrochemical technique. These electrodes were characterized by SEM EDX. These characterization results revealed that electrodes were prepared successfully. These electrodes were employed to determine their C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> electrooxidation activities by CV, CA, and, EIS measurements. PdZnPt-N doped G/ITO showed the best C<sub>6</sub>H<sub>12</sub>O<sub>6</sub> electrooxidation activity with a specific activity of 14.5 mA cm<sup>-2</sup>. This electrode is very good specific activity which is above the literature values. Furthermore, PdZnPt-N doped G/ITO demonstrated great stability and resistance, indicating that the

PdZnPt-N doped G/ITO electrode is a promising glucose electrooxidation electrode.

#### CRediT authorship contribution statement

**Aykut Caglar:** Data curation, Writing – original draft. **Tulin Avci Hansu:** Writing – review & editing. **Ozlem Sahin:** Visualization, Investigation. **Hilal Kivrak:** Supervision.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supplementary data

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