

DETERMINATION OF DOPAMINE WITH TWO-DIMENSIONAL MoS₂ MODIFIED GLASSY CARBON ELECTRODE

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ABSTRACT

Dopamine (DA) is a hormone secreted in the brain and it provides communication between nerve cells and ensures that body movements are carried out in a balanced way. Since dopamine is an electroactive compound, its determination by electrochemical techniques can be done with high sensitivity, easy processing and low cost. Molybdenum Sulfide (MoS₂) is one of the important two-dimensional (2D) transition metal dicalcogenes from graphene analogues. Transition metal dicalcogenes (TMD) are denoted by the general formula MX₂ (M = Mo, W, V, Nb, Ta, Ti, Zr, Hf and X = S, Se, Te). With their unique electronic, optical, thermal, mechanical and electrical properties and structures similar to graphene, they have found a wide range of applications by forming an interesting group of materials. In recent years, the most studied of the transition metal dicalcogenes are MoS₂ and WS₂. In the study, glassy carbon electrode (GCE) was modified with MoS₂. In experimental studies, two different methods were used for the preparation of modified electrodes. The first of these methods is the drip coating (DC) method. The electrode prepared by modifying the GCE with this method is called MoS₂(1)/GCE electrode. The second method applied is the electrochemical coating method. The electrode prepared by modifying the GCE with this method is called MoS₂(2)/GCE electrode. In this study, electrode modification was made for dopamine and dopamine was detected electrochemically with these two electrodes. In order to see the efficiency in DA determination, DA determination in a commercial drug called Dopamin was successfully performed with MoS₂(1)/GCE and MoS₂(2)/GCE with a relative error of 4.9 % and -1.0 %, respectively.

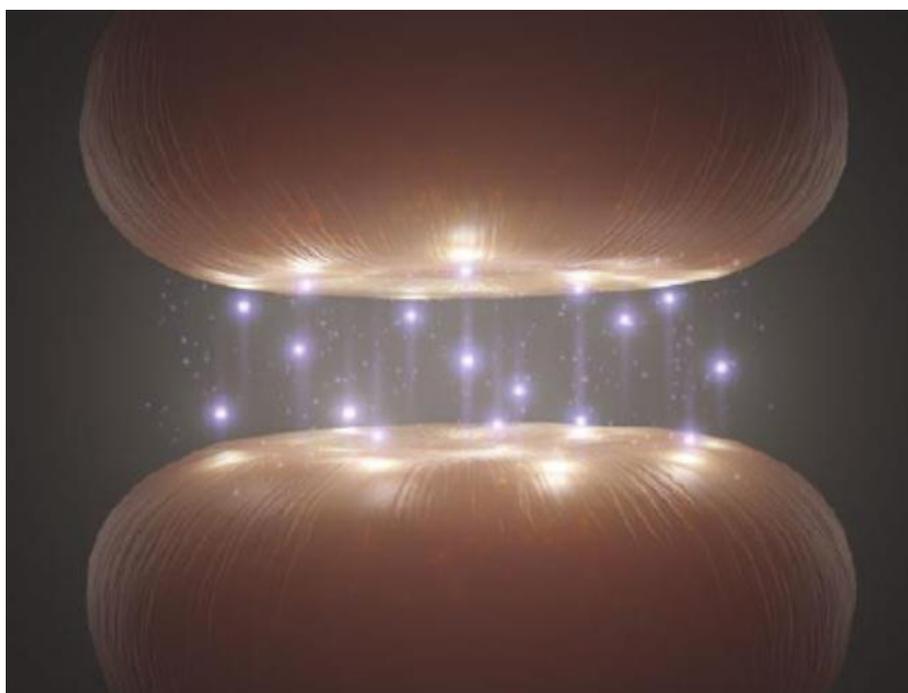
KEYWORDS: Two-Dimensional, Molibdendisulfür, Dopamine, Electrochemical Determination

1. INTRODUCTION

Today, with the increase in the income level, an increase has been observed in people's orientation to a quality and healthy life. While different theories are being put forward every day for a healthy life, the importance of protecting health and early diagnosis has emerged rather than treating the disease. Therefore, new methods are being developed for the determination of molecules of biological importance. Chromatographic (De Benedetto et al., 2014; Kand'ár, Drábková, & Hampl, 2011; Li & Franke, 2009) and spectrophotometric (Güçlü, Sözgen, Tütem, Özyürek, & Apak, 2005; Nevado, Gallego, & Laguna, 1996; M. Zhu, Huang, Li, & Shen, 1997) methods are mostly used in the determination of these molecules. These methods have advantages such as being reproducible and being able to detect more than one

substance at the same time. However, they have disadvantages such as requiring pre-treatment (separation, purification, concentration, etc.), chemical costs spent for these processes, loss of time, and the necessity of using expensive devices. However, it is possible to determine molecules of biological importance with simple and inexpensive devices with high sensitivity, short times, without the need for preprocessing by electrochemical methods (Balamurugan, Senthil Kumar, Thangamuthu, & Pandurangan, 2013; Cao, Luo, Ding, Zou, & Bian, 2008; Wu et al., 2014).

Dopamine (DA) is a hormone naturally produced in the body and secreted in the brain. By acting as a neurotransmitter, DA provides communication between nerve cells and ensures proper body movements and balance function. Dopamine is often referred to as the "happiness hormone," like serotonin. In addition to giving a feeling of happiness, dopamine is involved in many different functions in humans and animals such as movement, memory, comprehension, concentration, mood, pleasure, sleep and learning. An excess or deficiency of dopamine causes some serious health problems. In the absence of dopamine, disruptions in the nervous system may occur and lead to Parkinson's disease. The increase in the amount of dopamine as a result of drug or cigarette use leads to addiction. This chemical works in the brain by transmitting millions of signals within seconds. Thanks to these signals, body systems work, movement is provided and cognitive activities occur. These electrical signals, which are transmitted very quickly through the nerve cells, are carried by certain chemicals at the junction points of the nerve cells. These chemicals are called neurotransmitters, and without them there can be no transmission. The bright spots between nerve cells in Picture 1 represent neurotransmitters. "Dopamine" is one of the most important of the more than 100 neurotransmitters discovered by science. Picture 1 shows the function of neurotransmitters between nerve cells.



Picture 1. Neurotransmitters between nerve cells
(<https://www.istockphoto.com/tr/foto%C4%9Fraflar/n%C3%B6rofilament>)

Dopamine determination is very important. Some of the suggested methods for DA are chemiluminescence (Duan et al., 2015; Q. Zhu et al., 2015), spectrophotometric (Mamiński, Olejniczak, Chudy, Dybko, & Brzózka, 2005; Moghadam, Dadfarnia, Shabani, & Shahbazikhah, 2011) and chromatographic (Baranyi, Milusheva, Vizi, & Sperlágh, 2006; Virag & Whittington, 2002) techniques. Electrochemical determination of DA has a very large place in the literature. The reason for this is that it can be determined electrochemically in a short time, cheaply, easily and reliably. Figure 1 shows the electrochemical oxidation of DA at pH=7 (Oko et al., 2015; Ping, Wu, Wang, & Ying, 2012; Wang et al., 2012; Zhao, Zhang, & Yuan, 2001).

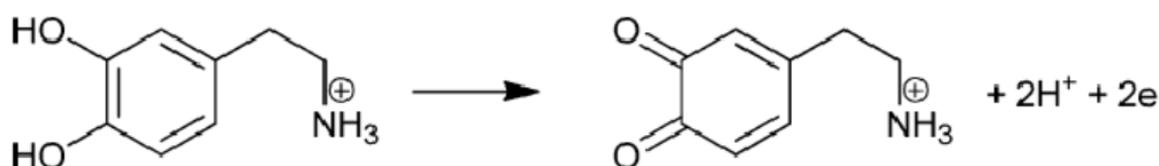


Figure 1. Electrochemical oxidation of Dopamine (at pH=7)

The glassy carbon electrode (GCE) is modified with MoS₂. Molybdenum Sulfide (MoS₂) is one of the important two-dimensional (2D) transition metal dichalcogenides from graphene analogues. MoS₂ is the most interesting material of this group with its graphite-like structure (Zhou, Liu, Wen, Hu, & Gui, 2014). MoS₂ has a hexagonal layer configuration. The atoms in the layer are connected by strong covalent bonds, the layers are connected to each other by weak forces, as in graphite, and each Mo layer is sandwiched between 2 sulfur layers (Huang et al., 2013).

In this study, it was planned to detect dopamine electrochemically by modifying GCE MoS₂ for dopamine and to analyze real samples with the prepared electrode modification.

2. MATERIALS AND METHODS

A three-electrode system was used in electrochemical studies. Measurements were made with a computer-controlled CHI 660E potentiostat. Also C3 cell stand, glassy carbon electrode (GCE) as working electrode and modified glassy carbon electrodes (MoS₂(1)/GCE electrode and MoS₂(2)/GCE electrode), platinum wire as counter electrode and saturated calomel as reference electrode electrode (SCE) was used. MoS₂ in powder form was obtained from Özdögu Madencilik Ltd.Şti. and dopamine was obtained from Sigma Aldrich. Standard solutions of dopamine were prepared in phosphate buffer pH=7.

Again, pH=7 phosphate buffer solution was used as the supporting electrolyte used in the electrochemical measurements. For the preparation of MoS₂(1)/GCE electrode and MoS₂(2)/GCE electrode, drip coating method and electrochemical coating method were applied. The preparation of the MoS₂(1)/GCE electrode prepared by the drip coating method is not described here again, since it was given in detail in our previously published publication (ASAN & Çelikkan, 2017). Electrochemical coating method was used for the preparation of the MoS₂(2)/GCE electrode. Coating Suspension; 100 mg of MoS₂ was kept in 100 mL of

isoamyl alcohol in an ultrasonic bath for 8 hours. It was then centrifuged at 6000 rpm for 10 minutes. After the isoamyl alcohol in the filtrate was separated, MoS₂ was washed twice with deionized water, and a homogeneous suspension was obtained by taking it into 100 mL of water and keeping it in an ultrasonic bath for 1 hour. The cleaned surface of the GCE electrodes was coated in three different times and the optimum time was determined as 3600 seconds. The coating was carried out by keeping the GCE in 0.5 M NaOH containing the coating suspension (10%, v/v) at constant potential (1.0 V) for 60 minutes. For the detailed preparation of this electrode (Asan, 2015) can be looked at.

3. RESULTS AND DISCUSSIONS

3.1. Dopamine Determination with MoS₂(1)/GCE

Electrochemical analysis studies against dopamine were performed with MoS₂(1)/GCE electrode prepared with 5 μ L coating from the coating suspension. An attempt was made to determine DA at ambient temperature with MoS₂(1)/GCE. Since the ambient temperature was around 35°C during the study, dopamine deteriorated rapidly. Therefore, the operation could not be carried out at ambient temperature. In order to prevent the degradation of dopamine, the dopamine solution added during the experiment was taken freshly from the refrigerator each time, and voltammograms were taken by placing the cell in a cold water bath and keeping the temperature constant at 11 °C. During the study, temperature control was carried out using a thermometer. The experimental setup is given in Picture 2.



Picture 2. The image of the cell in a cold water bath in the determination of dopamine with MoS₂(1)/GCE

DP voltammograms against increasing DA concentrations at 11 °C with the MoS₂(1)/GCE numbered electrode (in pH=7 phosphate buffer) are given in Figure 2 and the peak currents read from these voltammograms are plotted against the concentration and given in Figure 3.

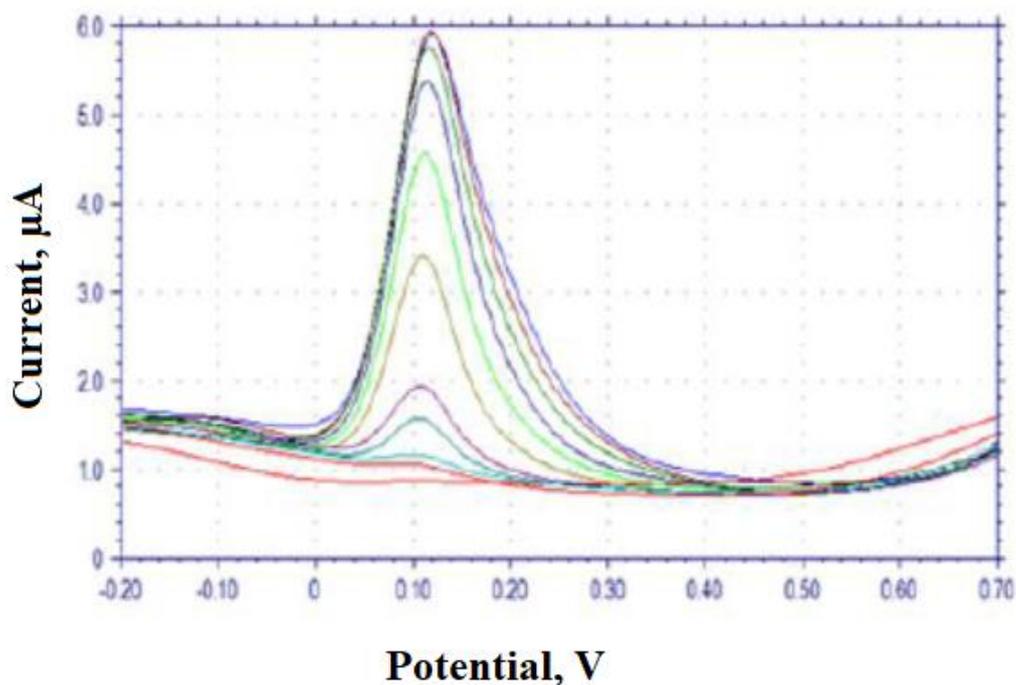


Figure 2. DPVs taken at different concentrations for DA determination with MoS₂(1)/GCE (in pH=7 buffer, 11°C)

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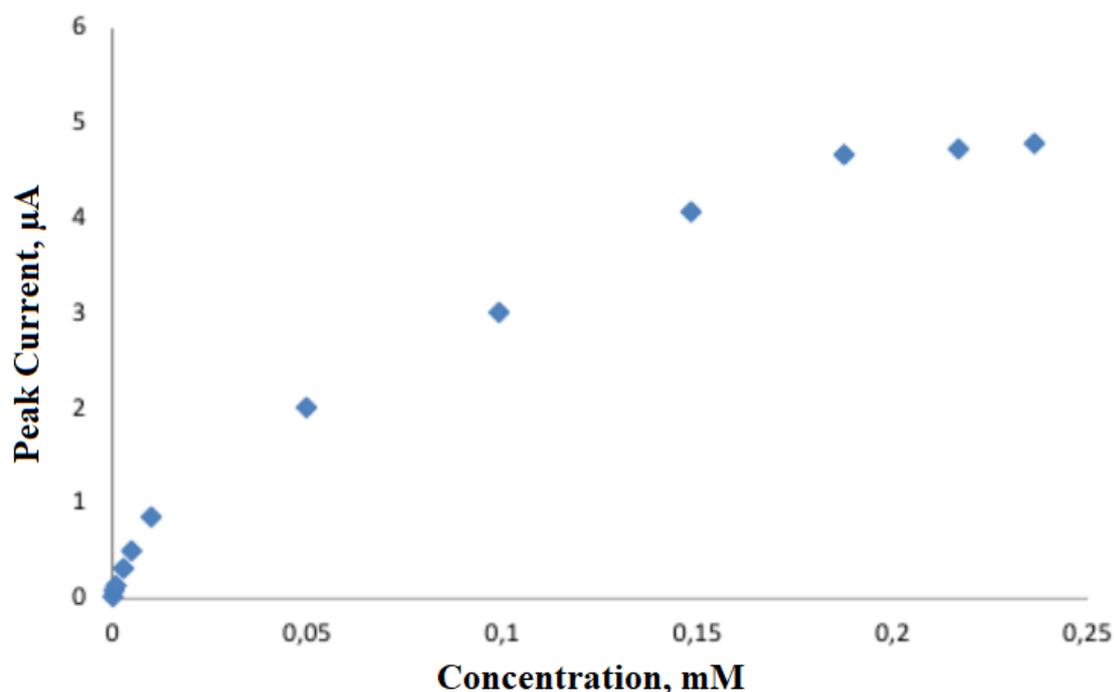


Figure 3. Graph of DA concentration versus peak currents with MoS₂(1)/GCE (at 11°C)

If Figure 3 is examined, first, a higher sensitivity was observed with the addition of DA, and then a decrease in the slope began. For this reason, the graph has been evaluated as having two working ranges. It is given as the 1st linear operating range in Figure 4 and the 2nd linear operating range in Figure 5.

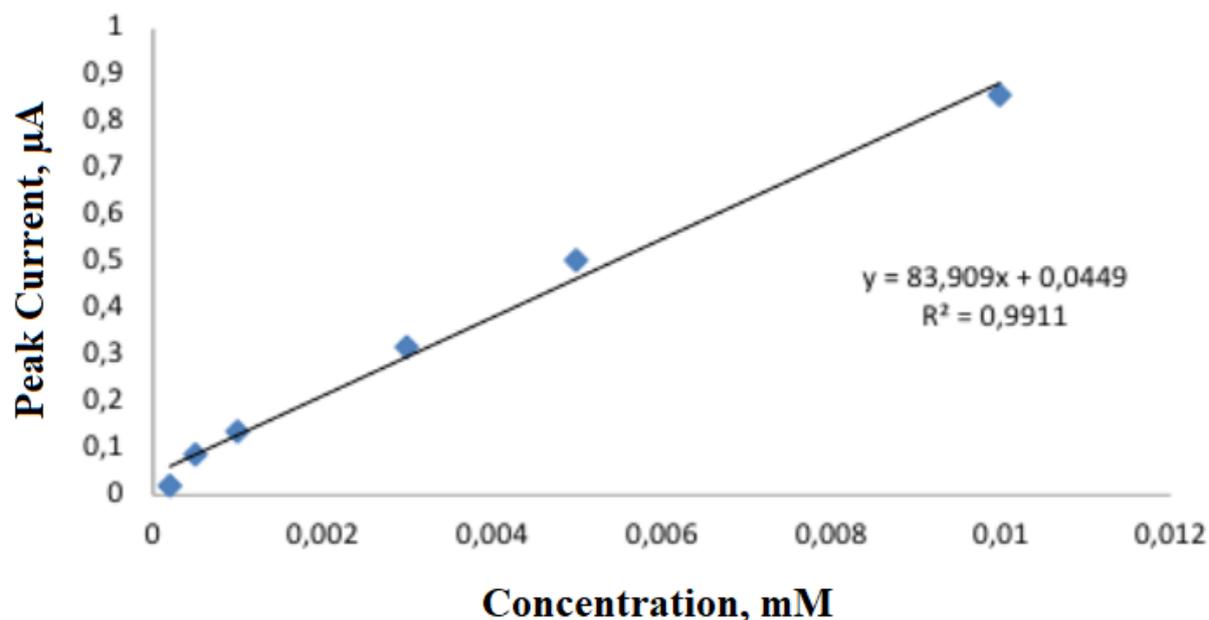


Figure 4. Graph of DA concentration versus peak currents with MoS₂(1)/GCE 1st linear operating range (at 11°C)

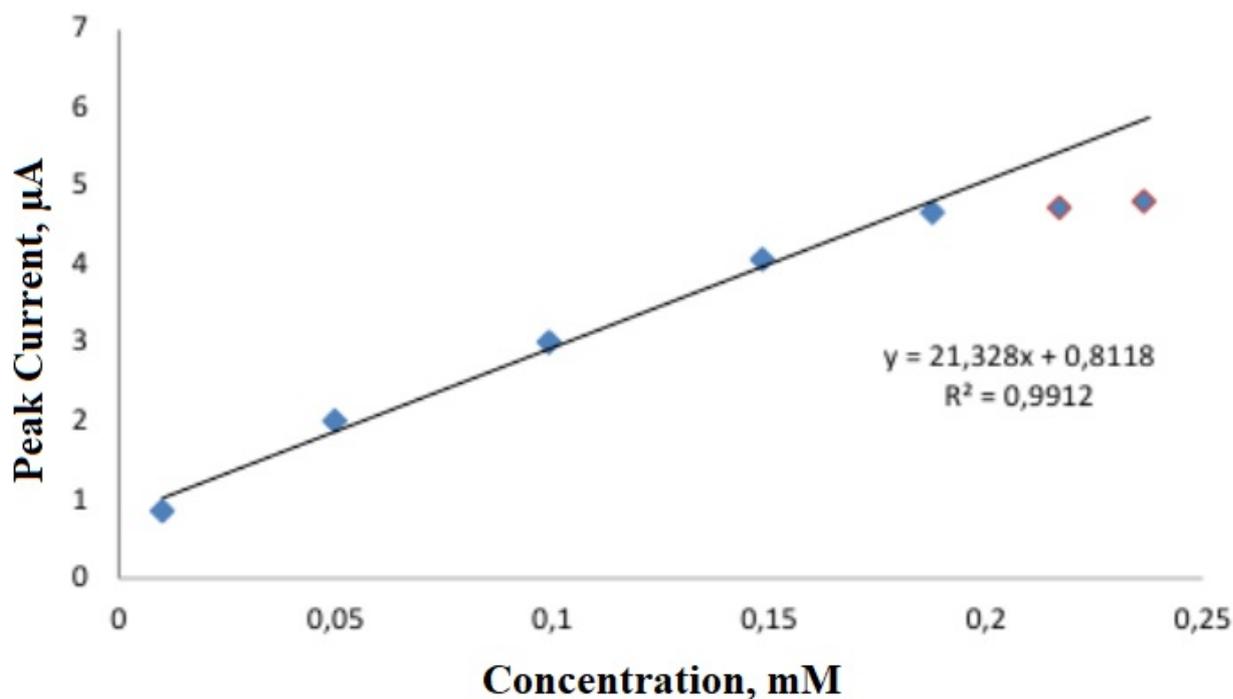


Figure 5. Graph of DA concentration versus peak currents with MoS₂(1)/GCE 2nd linear operating range (at 11°C)

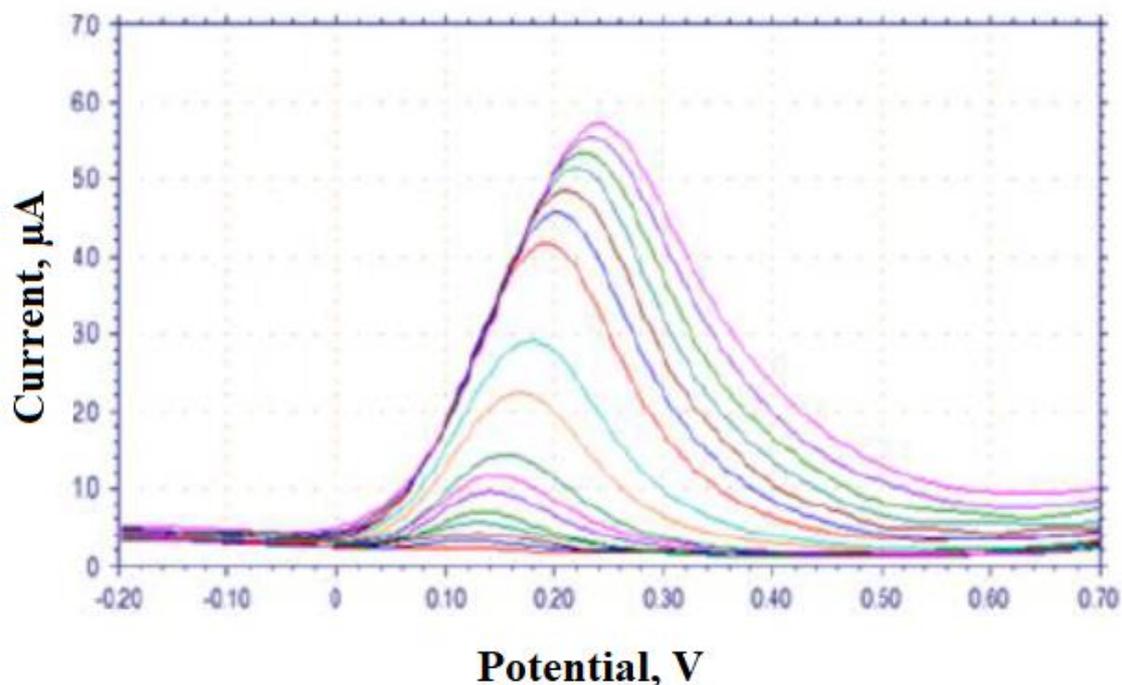


Figure 6. DPVs taken at different concentrations for Dopamine determination by GCE
(in pH=7 buffer, 11°C)

DPVs against DA concentrations at 11°C in pH=7 phosphate buffer with GCE are overlapped in Figure 6 and the peak currents read against concentration are given in Figure 7 by plotting.

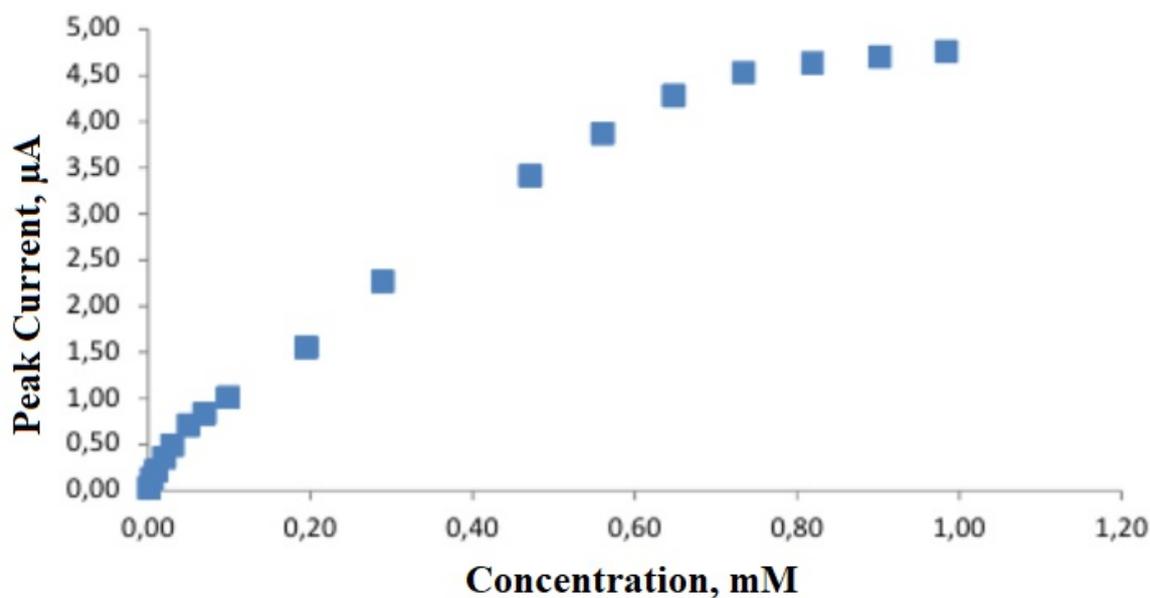


Figure 7. Plot of DA concentration versus peak currents for GCE (at 11°C)

The experimental data obtained in the dopamine environment for the comparison of MoS₂(1)/GCE with GCE are given in Table 1 collectively.

Table 1. Comparison of MoS₂(1)/GCE and the values obtained in DA environment with GCE

	Electrode Name		
	GCE	MoS ₂ (1)/GCE	
Linear Operating Range (μM)	1 – 650	0.2 -10	10 – 190
Line Equation ($\mu\text{A}/ \text{mM}$)	$y=6.4808x+0.254$	$y=83.909x+0.0449$	$y=21.328x+0.8118$
R^2	0.9926	0.9911	0.9912
Sensitivity ($\mu\text{A}/ \text{mM}$)	6.48	83.91	21.33

If Table 1 is examined, MoS₂(1)/GCE was found to be 12.9 times more sensitive in the 1st linear operating range and 3.3 times more sensitive in the 2nd linear operating range for DA compared to GCE. As a result, the electroanalytical sensitivity of the MoS₂ modified electrode for dopamine was better than that of the GCE. Analytical performance values for MoS₂(1)/GCE and DA are given in Table 2.

Table 2. Analytical performance values for DA with MoS₂(1)/GCE

1.Linear Working Range(LWR) (μM)	0.2 - 10
(1. LWR) Sensitivity ($\mu\text{A}.\text{mM}^{-1}$)	83.9
2.Linear Working Range(LWR) (mM)	10 – 190
(2. LWR) Sensitivity ($\mu\text{A}.\text{mM}^{-1}$)	21.3
LOD (nM)	52.2
LOQ (nM)	174

3.2. Determination of Dopamine with MoS₂(2)/GCE

Differential pulse voltammograms at varying DA amounts with MoS₂(2)/GCE were taken and superimposed, and the resulting graph is given in Figure 8, and the peak currents against concentration are plotted in Figure 9.

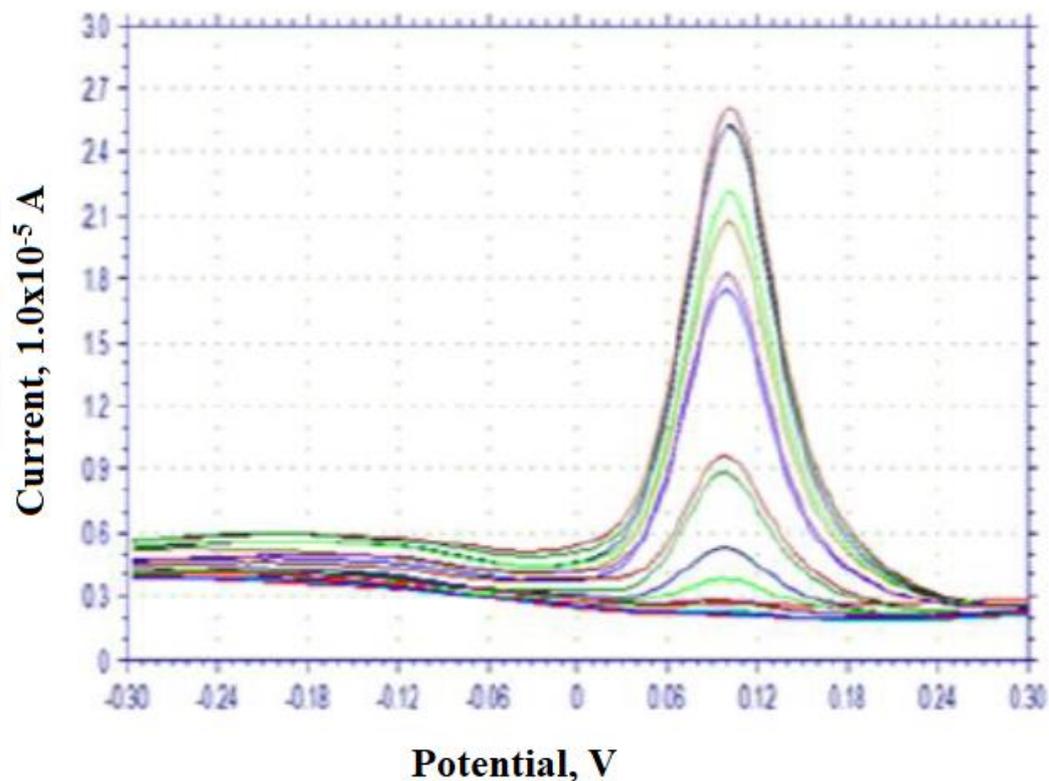


Figure 8. DPVs taken at different concentrations for DA determination with MoS₂(2)/GCE (in pH=7 buffer)

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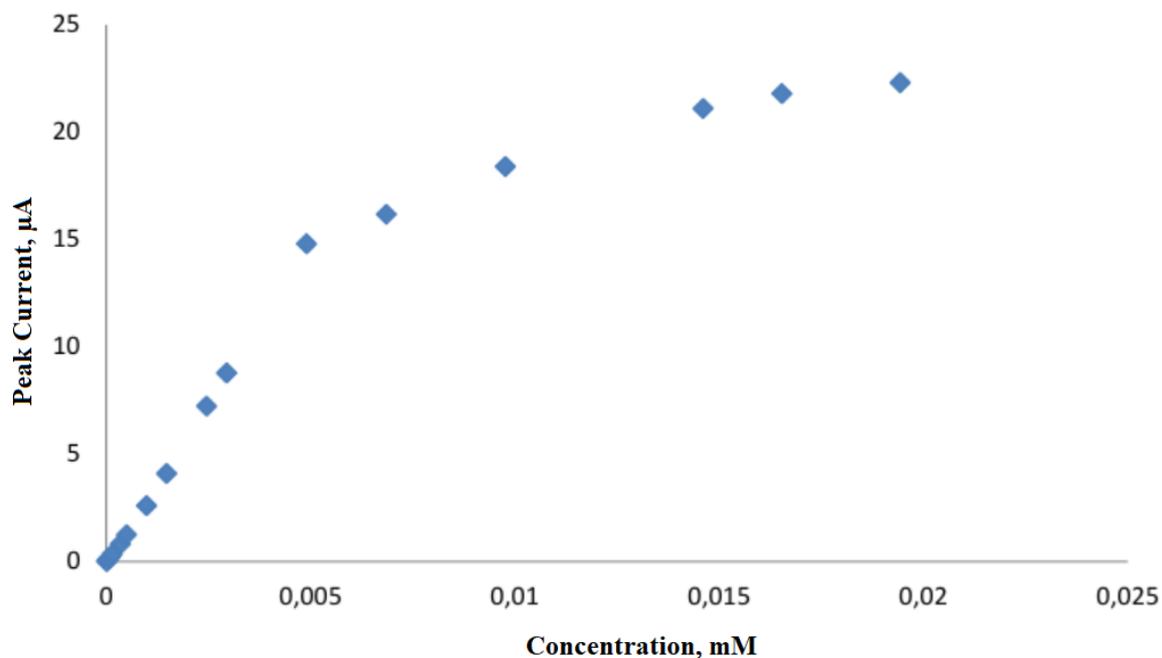


Figure 9. Graph of peak currents versus DA concentration in pH=7 buffer with MoS₂(2)/GCE

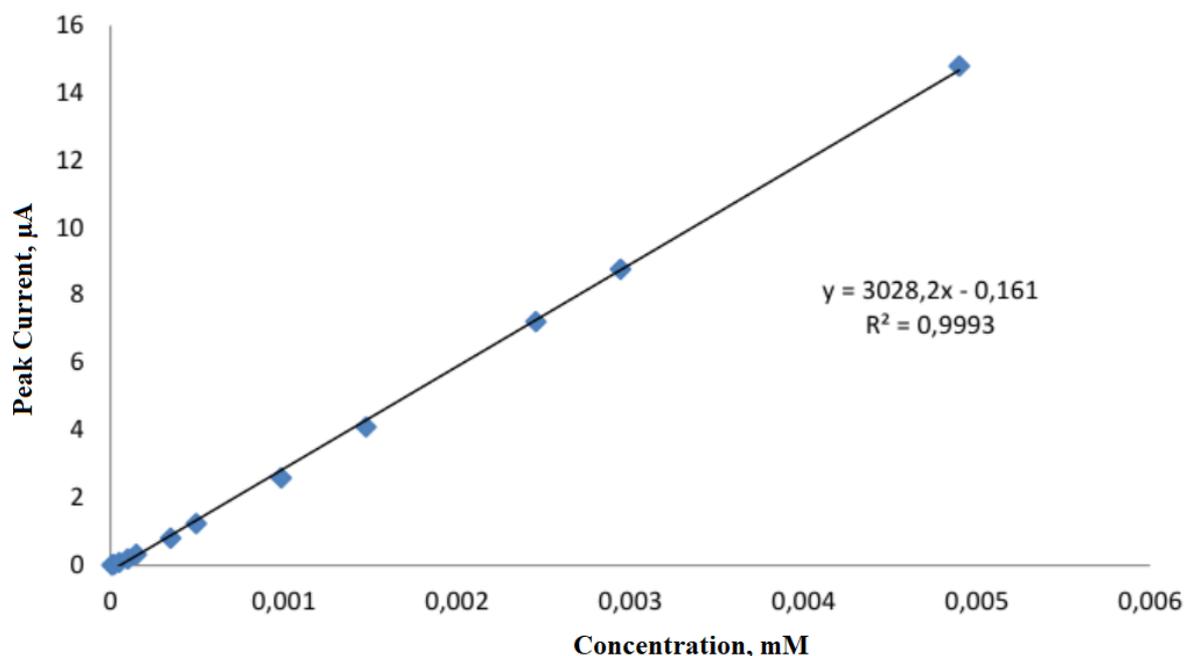


Figure 10. 1st linear operating range for DA determination with MoS₂(2)/GCE

As can be seen from the graph, the sensitivity of the first operating range is quite high. The 1st linear operating range is presented in Figure 10, and the 2nd linear operating range is presented in Figure 11.

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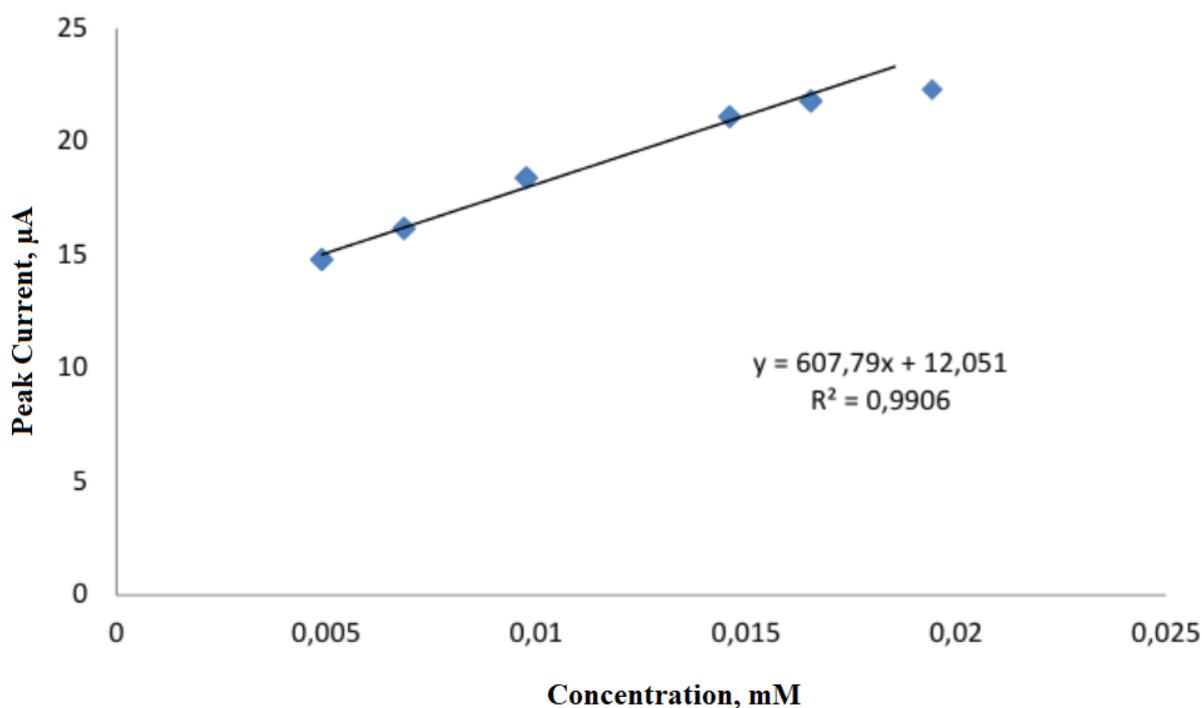


Figure 11. 2nd linear operating range for DA determination with MoS₂(2)/GCE

Table 3 has been prepared to compare the electrode sensitivity of MoS₂(2)/GCE with GCE and MoS₂(1)/GCE.

Table 3. Comparison of MoS₂(2)/GCE values obtained in DA environment with GCE and MoS₂(1)/GCE

	Electrode Name				
	GCE	MoS ₂ (1)/GCE	MoS ₂ (2)/GCE	MoS ₂ (2)/GCE	MoS ₂ (2)/GCE
Linear Operating Range, μM	1 - 650	0.2 - 10	10 - 190	0.01 - 5	5 - 16.5
Line Equation ($\mu\text{A}/\text{mM}$)	$y=6.4808x+0.254$	$y=83.909x+0.0449$	$y=21.328x+0.8118$	$y=3028.2x-0.161$	$y=607.79x+12.051$
R ²	0.9926	0.9911	0.9912	0.9993	0.9906
Sensitivity ($\mu\text{A}/\text{mM}$)	6.5	83.9	21.3	3028	607.8

When Table 3 is examined, it is seen that the sensitivity in the determination of DA with MoS₂(2)/GCE increases 466 times compared to GCE and 36 times compared to the electrode numbered MoS₂(1)/GCE in the 1st linear operating range. In addition, it was found that the sensitivity value in the 2nd linear operating range with MoS₂(2)/GCE increased 94 times compared to GCE and 28.5 times compared to the electrode numbered MoS₂(1)/GCE. Analytical performance values for MoS₂(2)/GCE and DA are presented in Table 4.

Table 4. Analytical performance values for DA with MoS₂(2)/GCE

1.Linear Working Range(LWR) (μM)	0.01 - 5
(1. LWR) Sensitivity ($\mu\text{A}.\text{mM}^{-1}$)	3028
2.Linear Working Range(LWR) (mM)	5 - 16.5
(2. LWR) Sensitivity ($\mu\text{A}.\text{mM}^{-1}$)	607.8
LOD (nM)	12.8
LOQ (nM)	42.6

3.3. Determination of DA in a Commercial Drug named Dopmin with MoS₂(1)/GCE

For the determination of DA in a commercial drug called Dopmin with MoS₂(1)/GCE, a sample solution of 4.22 μM was prepared by adding 2 μL of drug solution to 100 mL pH=7 phosphate buffer. DPV was taken from three sample solutions. Then, DA solutions were prepared at known concentrations in pH=7 buffer solution and the concentrations were calculated for three samples by the calibration line method. The calibration line is given in Figure 12.

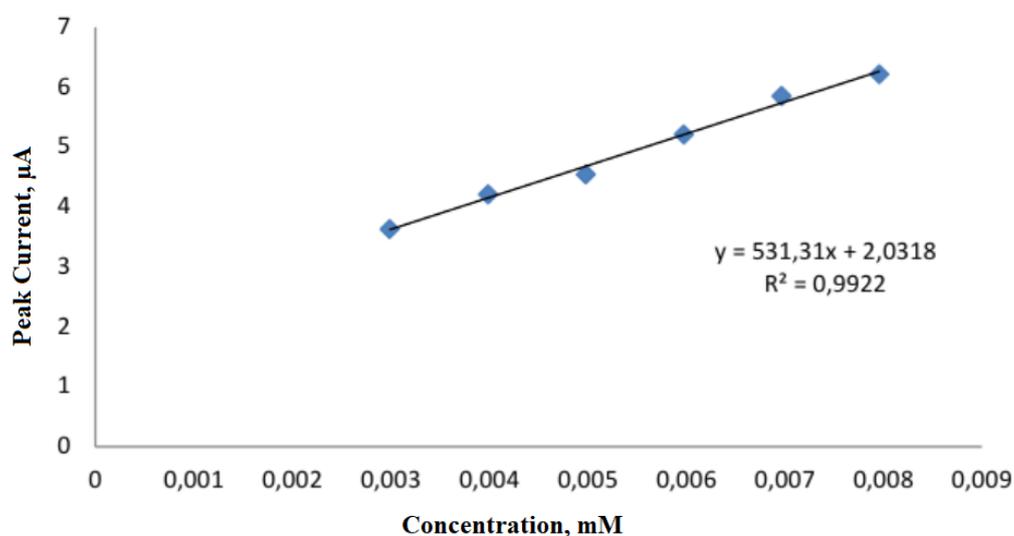


Figure 12. Determination of DA by the calibration line method in a drug called Dopmin with MoS₂(1)/GCE

In Table 5, DA values obtained from real samples are presented with their relative errors.

Table 5. DA concentrations obtained from real samples with MoS₂(1)/GCE, with their relative errors

Sample	Peak Current for X (μA)	Value Found for X (μM)	Actual Value (μM)	% Relative Error
1	4.346	4,36	4.22	3,21
2	4.510	4,67	4.22	10.6
3	4.301	4.26	4.22	1.03

(X)_{average} = 4,43 μM, t = 4,30 (at 95% Confidence Level)

% 95 CL = 4,43 ± 0,53 μM

At 95 % CL (3.90 μM to 4.96 μM). The actual value is 4.22 μM between this range.

The results are within the 95 % confidence interval.

3.4. Determination of DA in a Commercial Drug named Dopmin with MoS₂(2)/GCE

For the determination of dopamine in a commercial drug called Dopmin with MoS₂(2)/GCE, a sample solution of 4.22 μM was prepared by adding 2 μL of drug solution to 100 mL pH=7 phosphate buffer. DPV was taken from three samples taken from the solution. Then, DA solutions were prepared at known concentrations in pH=7 buffer solution and the concentrations were calculated for three samples by the calibration line method. The calibration line is given in Figure 13.

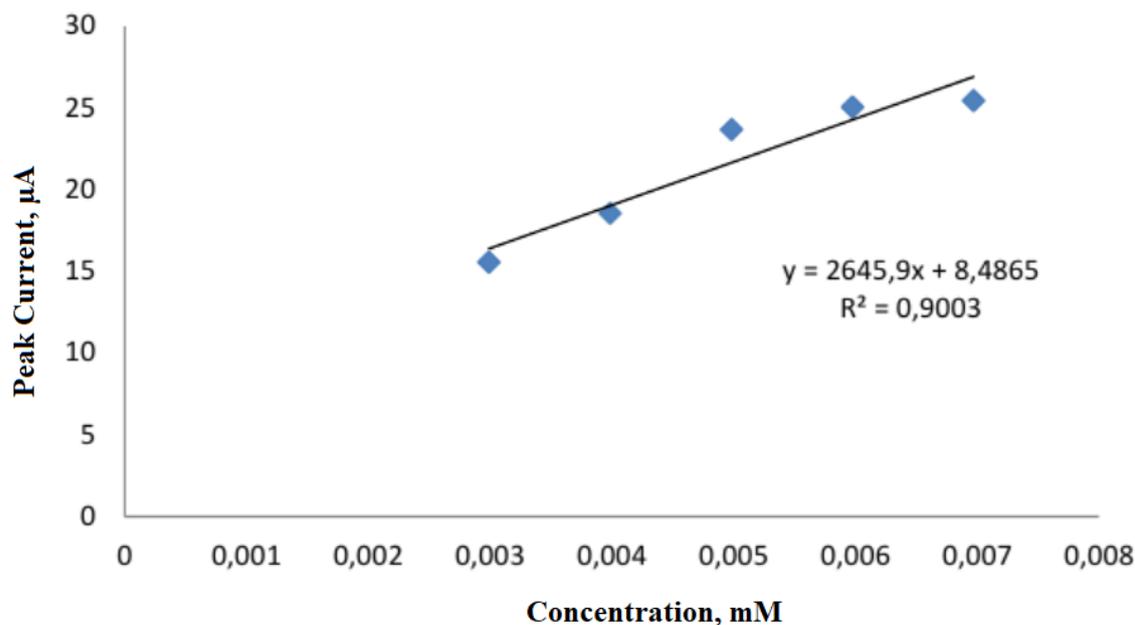


Figure 13. Determination of DA with MoS₂(2)/GCE in a drug called Dopmin by calibration line method

In Table 6, Dopamine values obtained from real samples are presented with their relative errors.

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Table 6. DA concentrations obtained from real samples with MoS₂(2)/GCE, with their relative errors

Sample	Peak Current for X (μA)	Value Found for X (μM)	Actual Value (μM)	% Relative Error
1	1.851	3.79	4.22	-10.2
2	1.980	4.28	4.22	1.50
3	2.030	4.46	4.22	5.80

$(\bar{X})_{\text{average}} = 4,18 \mu\text{M}$, $t = 4,30$ (at 95% Confidence Level)

% 95 CL = $4,18 \pm 0,86 \mu\text{M}$

At 95 % CL (3.32 μM to 5.04 μM). The actual value is 4.22 μM between this range.

The results are within the 95 % confidence interval.

4. CONCLUSIONS

MoS₂(1)/GCE was found to be 12.9 times more sensitive in the 1st linear operating range and 3.3 times more sensitive in the 2nd linear operating range for DA compared to GCE. With MoS₂(1)/GCE, the sensitivity for dopamine was increased up to 12.9 times. The 1st linear

working range for dopamine was found to be 0.2 μM – 10 μM , the sensitivity 83.9 $\mu\text{A mM}^{-1}$ and the limit of detection 5.2×10^{-8} M. 2. linear operating range 10 μM - 190 μM , sensitivity 21.3 $\mu\text{A mM}^{-1}$ was determined. The 1st linear working range for dopamine with MoS₂(2)/GCE was 0.01 μM – 5 μM , the sensitivity was 3028 $\mu\text{A.mM}^{-1}$ and the detection limit was 1.3×10^{-8} M. 2. linear operating range 5 μM – 16.5 μM , sensitivity 608 $\mu\text{A.mM}^{-1}$ was determined. MoS₂(2)/GCE was found to be 466 times more sensitive in the 1st linear operating range and 93.5 times more sensitive in the 2nd linear operating range compared to GCE. Thus, it was achieved to increase the sensitivity for dopamine up to 466 times with MoS₂(2)/GCE. Determination of DA in a commercial drug called Dopmin to see the efficiency in determination of DA with MoS₂(1)/GCE and MoS₂(2)/GCE, relative to 4.9% and -1.0% with MoS₂(1)/GCE and MoS₂(2)/GCE, respectively. executed successfully with no error.

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