

# An Electrochemical Sensor Based on Platinum Nanoparticles and Mesoporous Carbon Composites for Selective Analysis of Dopamine

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An electrochemical sensor was developed by in-situ electrodeposition of platinum nanoparticles (Pt NPs) on the surface of mesoporous carbon (MC). Due to the synergistic effect of Nafion film, Pt NPs, and MC, the as-prepared sensor (Nafion/Pt/MC/GCE) exhibited excellent electrocatalytic activity towards the oxidation of dopamine (DA). Moreover, the oxidation potential of DA on the sensor was perfectly distinguished (no overlap) from those of uric acid (UA) and ascorbic acid (AA) on the sensor. The differences in oxidation peak potential for UA–DA and DA–AA were 123 and 204 mV, respectively, which are sufficiently large for the selective determination of DA. Electrocatalysis of DA on the sensor was investigated by differential pulse voltammetry (DPV) and chronoamperometry, revealing excellent catalytic activity. The catalytic current obtained from the amperometric response was linearly dependent on DA concentration in the range of 0.1 to 193  $\mu\text{M}$  (R 0.994). The sensitivity of DA detection was calculated to be 12.23  $\mu\text{A mM}^{-1}$ , and the limit of detection (LOD) was calculated to be 0.034  $\mu\text{M}$ . Therefore, due to its advantages of high stability, good reproducibility and selectivity, and attractive sensing ability, this sensor shows promise as a new strategy for the detection of DA in biological samples.

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**Keywords:** Electrodeposition; Platinum nanoparticles; Mesoporous carbon; Dopamine

## 1. INTRODUCTION

Dopamine (DA), a natural neurotransmitter, plays a significant role in the control of the central nervous system, and of the cardiovascular, renal, and hormonal functions. As well, it is involved in drug addiction and diseases [1]. A low level of DA may induce neurological disorders such as schizophrenia and Parkinson's disease [2-4]. Therefore, precise detection of DA concentration is very important in the

biomedical chemistry, neurochemistry, and diagnostic research [1]. Numerous methods with high efficiency and small sample size requirements, including spectroscopic, chromatographic, enzymatic, and electroanalytical methods [5-8], have been explored for determining neurotransmitters such as DA, ascorbic acid (AA), norepinephrine, adenosine, and serotonin. In particular, electrochemical techniques have attracted considerable interest due to their rapid response, high sensitivity, low cost, and simple operation. However, the use of electrochemical detection methods encounters a major problem in that conventional electrodes cannot be used to selectively detect DA, because biochemical compounds such as AA and uric acid (UA) usually foul the electrodes and the oxidation potentials of these compounds strongly overlap with that of DA [9]. Therefore, the strict selectivity requirements have motivated a strong effort toward the development of selective interfacial materials for the determination of DA.

To improve the rate of electron transfer between the neurotransmitter and the electrodes and eliminate the interferences of AA and UA, many interfacial materials including noble metal nanoparticles [10,11], conducting polymer layers [12-14], other inorganic or organic compounds, and carbon-based materials or their hybrids have been used for electrochemical sensors fabrications [15-17]. Carbon-based materials including carbon nanotubes (CNTs), graphene oxide (GO), reduced graphene oxide (RGO) fullerene, and carbon nanofibers have widely attracted particular attention in the field of electrochemical sensors due to their remarkable mechanical and electrical properties. These carbon materials possess high electroactive surface area, mechanical flexibility, high electrical conductivity, biocompatibility, and chemical stability [18]. Among these, ordered mesoporous carbon (MC) has also been a very attractive support material due to its periodic mesoporous structure, large pore volume, good thermal and mechanical stability, and excellent electrical conductivity [19]. In particular, MC is used to support noble metals, forming novel hybrids to provide electrochemical sensing platforms with enhanced electronic and catalytic properties [19-21].

Platinum (Pt) NPs have been of particular interest due to their role in many catalytic reactions and the controllable specific the surface of electrode [22]. Many hybrids of Pt NPs supported by carbon material have been used to construct electrochemical sensors. Xu et al. reported a Pt/reduced graphene oxide (Pt/RGO) hybrid modified glassy carbon electrode (GCE) for the detection of DA and UA in the presence of a high concentration of AA [23]. Yogeswaran et al. prepared a multiwalled carbon nanotube (MWCNT)/Nafion/PtAu system for the simultaneous determination of AA, epinephrine, and UA [24]. Therefore, due to their special electronic and catalytic properties, nanocomposites based on Pt NPs and support materials have the synergistic effects of Pt and its support material that significantly improve the catalytic ability of the fabricated sensor.

In this work, to further improve the stability, selectivity, and biocompatibility of the interface material, Nafion, an ion-exchangeable, discriminative and biocompatible polymer, was combined with Pt NPs and MC to prepare an electrochemical sensor. Pt NPs were electrodeposited in situ on the surface of the MC to form Nafion/Pt/MC nanocomposites on a glass carbon electrode (GCE) in Nafion,  $H_2PtCl_6$ , and the MC mixed system. The as-prepared sensor (Nafion/Pt/MC/GCE) exhibited perfect selectivity and excellent electrochemical activity towards the oxidation of DA in the presence of AA and UA. The sensor also showed an attractive sensing ability for detecting DA in blood serum samples.

## 2. EXPERIMENTAL

### 2.1. Materials

Ascorbic acid (AA), dopamine (DA), uric acid (UA), and Nafion were purchased from Sigma-Aldrich in China. Chloroplatinic acid hexahydrate ( $\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ , 99.9%) was purchased from Sinopharm Chemical Reagent Co. Ltd (Shanghai, China). Cubic texture mesoporous carbon (CMk-8) was obtained from XFNANO Materials CO. Ltd (Nanjing, China). The pH of phosphate buffer solution (PBS, 0.10 M, containing 0.10 M KCl) was adjusted by NaOH and HCl solutions with a pH meter (Mettler Toledo). All the other chemicals were of analytical grade and were used as received. Double-distilled water was used to prepare all the aqueous solutions used in this study.

### 2.2. Fabrication of the modified electrodes

The Nafion/Pt/MC/GCE was prepared according to the method reported in the literature [25]. B, 3.5 mg MC was added into 1.5 mL 5 mmol/L  $\text{H}_2\text{PtCl}_6$ , and the mixture was sonicated for 20 min. The suspension was dried at 55 °C for 1 h to obtain  $\text{H}_2\text{PtCl}_6$ /MC sample. The obtained  $\text{H}_2\text{PtCl}_6$ /MC sample was dissolved in 1.6 mL of the mixed solution containing 20% ethanol, 73.75%  $\text{H}_2\text{O}$  and 6.25% Nafion by sonicating for 20 min. Subsequently, 4.0  $\mu\text{L}$  of the mixed solution was placed dropwise on the surface of a glassy carbon electrode ( $\Phi = 3$  mm) and dried in the dark place at room temperature. The modified electrode was reduced in situ by successive scans in 0.5 M  $\text{H}_2\text{SO}_4$  for 25 cycles in the potential range from 0.55 to  $-0.2$  V to obtain Nafion/Pt/MC/GCE.

### 2.3. Apparatus and measurements

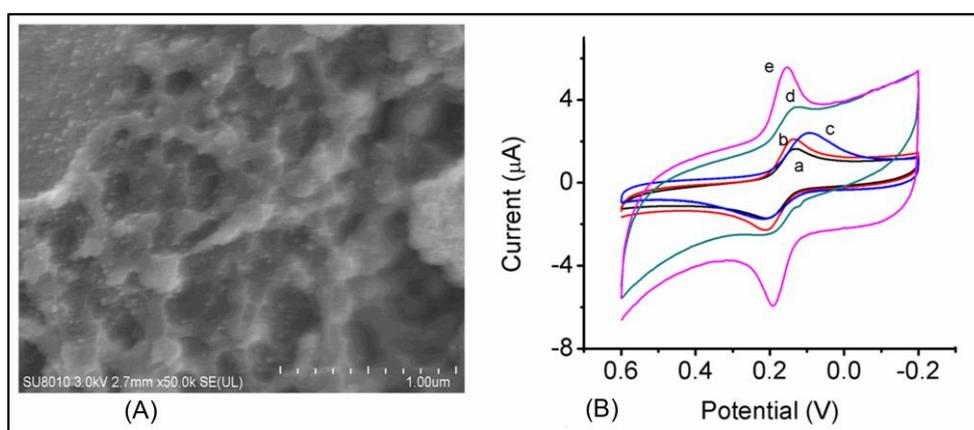
All the electrochemical experiments were performed using a CHI660C electrochemical workstation (Chenhua, China). The fabricated Nafion/Pt/MC/GCE was used as the working electrode. Pt wire and Ag/AgCl electrode (3.0 M KCl) were used as the auxiliary and reference electrodes, respectively. All potentials were reported relative to the Ag/AgCl reference electrode. Electrochemical measurements were performed in 0.10 M PBS (pH 7.0) after deoxygenation by pure nitrogen for 10 min. Scanning electron microscope (SEM) measurements were carried out using a SU8010 field emission scanning electron microanalyzer (HITACHI, Japan) to characterize the Pt/MC surface.

## 3. RESULTS AND DISCUSSION

### 3.1. Characterization of Nafion/Pt/MC/GCE

Figure 1A shows the SEM image of the Nafion/Pt/MC sample. The MC consisted of small grains with submicrometer-scale size of 0.2–0.5  $\mu\text{m}$ . It was observed that the hazy Nafion film wrapped the MC particles. Many Pt nanoparticles with size of  $\sim 10$  nm are uniformly distributed on the MC surface, indicating that in situ electrochemical reduction of  $\text{H}_2\text{PtCl}_6$  forms uniform Pt NPs on the MC surface.

Figure 1B presents the cyclic voltammograms (CVs) of the different modified electrodes in 0.1 M PBS containing 0.050 mM DA. A pair of redox peaks can be observed for all of the electrode because DA is an electroactive neurochemical (curves a–e). However, compared to the electrochemical behavior on bare GCE, Nafion/GCE, Pt/GCE, and Pt/MC/GCE, the charge transfer kinetics of DA on the Nafion/Pt/MC/GCE were clearly improved. A pair of reversible DA redox peak (curve e) with a peak potential separation ( $\Delta E_p$ ) of 38.7 mV were observed for Nafion/Pt/MC/GCE. The current response of the redox couple increased greatly compared to those on the other modified electrodes. Moreover, the value of  $\Delta E_p$  is much smaller than those of the graphene/size-selected Pt nanocomposites (80 mV, [26]) and the Pt/RGO-modified (73 mV, [23]) electrodes, indicating efficient sensing of DA with excellent reversibility. These results demonstrate that porous MC provides the specific surface area for immobilizing Pt NPs, resulting in enhanced catalytic activity due to the synergistic effects of Pt NPs, MC, and Nafion.

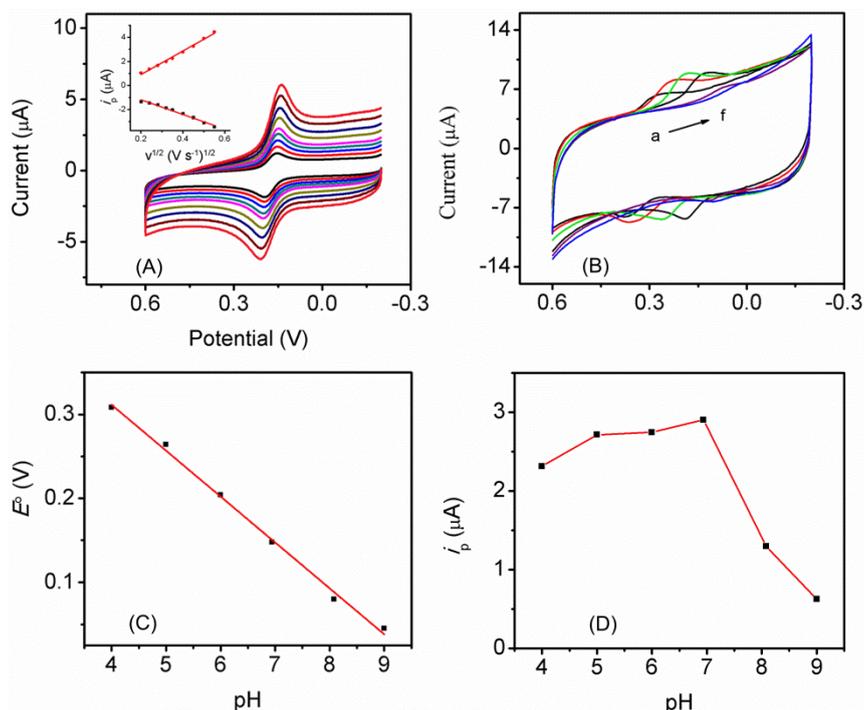


**Figure 1.** (A) SEM image of Nafion/Pt/MC on GEC. (B) CVs of 50  $\mu\text{M}$  DA for bare GCE (a), Nafion/GCE (b), Pt/GCE (c), Pt/MC/GCE (d), and Nafion/Pt/MC/GCE in 0.1 M PBS (pH 7.0).

### 3.2. Electrochemical behavior of Nafion/Pt/MC/GCE

To investigate the electrochemical reaction of DA, the CVs of 0.05 mM DA on the Nafion/Pt/MC/GCE were obtained at different scan rates. As shown in Figure 2A, the redox peak currents were proportional to the square root of the scan rate, which varied from 0.04 to 0.40  $\text{V s}^{-1}$ , indicating that the electrochemical reaction of DA on the modified electrode is a diffusion-controlled process. The effect of pH on the electrochemical behavior of DA was also investigated by CV. Figure 2B shows the dependence of the present sensor on the pH. The DA redox peak potential of DA shifted negatively with increasing PBS pH (4.0–9.0). A linear relationship was obtained from the plot of formal potential ( $(E_{pa} + E_{pc})/2$ ) as a function of pH and was described by  $E^\circ (\text{V}) = -0.055 \text{ pH} + 0.53$  ( $R = 0.9969$ , Figure 2C). The slope of  $-0.055 \text{ V/pH}$  was approximately equal to the theoretical value ( $-0.059 \text{ V/pH}$ ) for a single-proton coupled and reversible one-electron-transfer system. Moreover, the cathodic peak current of DA increased with increasing pH in the solution until pH 7.0, and then the current decreased for pH exceeding 8.0 (Figure 2D). Therefore, 0.1 M PBS with pH 7.0 was chosen as the optimal pH,

which is similar to that in the human body. Furthermore, the optimized quantities of Pt, MC, and the electrolyte solutions including 0.1 M PBS, tris(hydroxymethyl)aminomethane hydrochloride (Tris-HCl buffer, pH 8.0), sodium acetate-acetic acid buffer (pH 5.0), and KCl solution were obtained. The best electrochemical behavior of DA on the Nafion/Pt/MC/GCE was obtained in 0.1 M PBS for the amounts of Pt and MC specified above.



**Figure 2.** (A) CVs of Nafion/Pt/MC/GCE in 50  $\mu\text{M}$  DA solution at different scan rates of 0.04, 0.06, 0.08, 0.10, 0.12, 0.16, 0.20, 0.25, and 0.30  $\text{V s}^{-1}$ . (Inset: relationship between the redox peak current and the square root of the scan rate.) (B) CVs of the Nafion/Pt/MC/GCE in 50  $\mu\text{M}$  DA solution with various pH (from a to f: 4.0, 5.0, 6.0, 7.0, 8.0, and 9.0). (C) Relationship between the formal peak potential and relative pH. (D) The current of the reduction peak ( $i_p$ ) from CVs in (D) at relative pH.

### 3.3. Response of Nafion/Pt/MC/GCE on DA

From conventional electrodes, the oxidation potentials of AA, DA, and UA overlap strongly, hampering the selective determination of DA. To evaluate the distinguishing ability towards the oxidation of these three biomolecules, the ternary mixture containing AA, DA, and UA was investigated by differential pulse voltammetry (DPV). As shown in Figure 3A, the DPV peaks of UA, DA and AA are indistinguishable on bare GCE. However, on the Nafion/Pt/MC/GCE, separated oxidation peaks for UA, DA, and AA were clearly observed at 0.293 V, 0.170 V, and  $-0.034$  V, respectively. The peak separations between UA and DA and between DA and AA were 123 and 204 mV, which are comparable to or larger than with those obtained with nanoporous carbon (132 mV and 198 mV, [27]), poly(bromocresol green) (140 mV and 160 mV, [28]), and Au@Pd-RGO (164 mV and 176 mV, [8]) modified electrodes. These results suggest that the presented sensor based on Nafion/Pt/MC nanomaterials possesses good selectivity for the catalytic oxidation of DA. DPV was carried out to

investigate the electrocatalysis of DA in 0.1 M PBS. For the Nafion/Pt/MC/GCE, the oxidation peak current of DA dramatically increased with increasing concentration (Figure 3B). The linear responses for the detection of DA was obtained in the concentration range of 0.2–100.0  $\mu\text{M}$  ( $R = 0.9952$ , inset in Figure 3B).

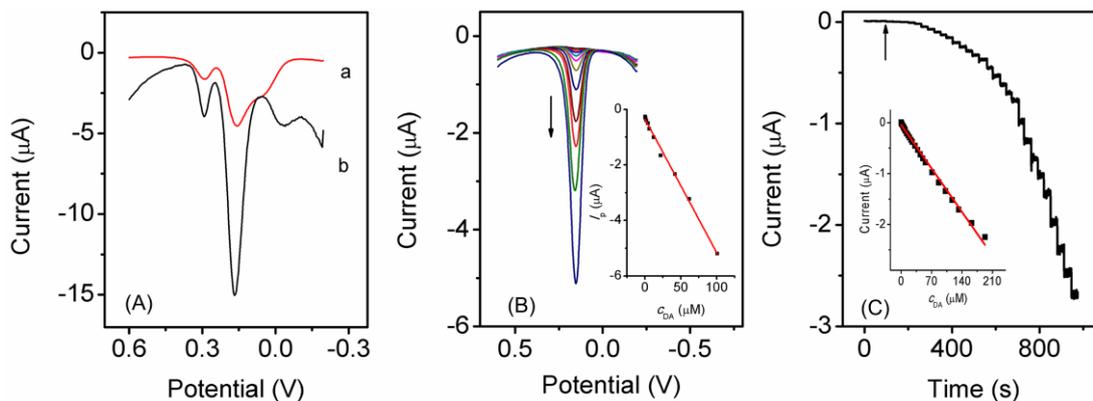
Chronoamperometry was used to further investigate the electrocatalytic response of the sensor to DA oxidation. Figure 3C shows a typical steady state amperometric response of the Nafion/Pt/MC/GCE with the successive addition of DA into the continuously stirred solution of PBS at the applied potential of 0.18 V. Clearly, the sensor responded quickly to DA oxidation and the catalytic current achieved the steady state within 1.3 s of the DA addition. The calibration plot shows a good linear relationship between the oxidation current and DA concentration ranging from 0.1 to 193  $\mu\text{M}$  ( $R = 0.994$ ). The sensitivity for DA detection was obtained as 12.23  $\mu\text{A mM}^{-1}$  and the limit of detection (LOD) was calculated as 0.034  $\mu\text{M}$  ( $S/N = 3$ ). These results indicate that the proposed sensor can be used to detect DA quantitatively. Table 1 compares analytical performances of our proposed sensor to other reported methods for DA sensing in terms of linear range and LODs. The wide linear ranges, high response sensitivity, and low LOD of our sensor are better than or comparable to those reported in the literature, which may be ascribed to the synergistic effects of Pt NPs, MC, and Nafion film. Therefore, the Nafion/Pt/MC material can be potentially used for electrochemical sensor fabrication.

**Table 1.** Comparison of the response characteristics of different sensors for the DA determination.

Electrode	Methods	Linear range ( $\mu\text{M}$ )	LODs ( $\mu\text{M}$ )	Reference
$\text{Fe}_3\text{O}_4/\text{RGO}/\text{GCE}$ <sup>a</sup>	DPV	0.5–10.0	0.12	[29]
CTAB/GNSP	DPV	4–52	0.6	[30]
Graphene-AT /GCE	DPV	5–25	5	[31]
GO-PAN/GCE	DPV	1–14	0.5	[32]
RGO/AuNPs	DPV	1.0–100	14	[33]
PBG/GCE	DPV	0.05–10	0.017	[28]
GEF/CFE	DPV	0.7–45.21	0.5	[34]
PG/GCE	$i-t$ <sup>b</sup>	5.00–710	2.00	[35]
Pt/GCE	DPV	0.03–8.1	0.03	[10]
Nafion/Pt/MC/GCE	DPV	0.1–193	0.034	This work

<sup>a</sup> Abbreviations: GO: reduced graphene oxide; GCE: Glassy carbon electrode; CTAB: Cetyltrimethylammonium bromide; GNSP: Graphene nanosheets; AT: 2-Amino-thiazol; PAN: polyaniline; PBG: poly(bromocresol green); GEF: Graphene flowers; CFE: carbon fiber electrode; PG: pristine graphene.

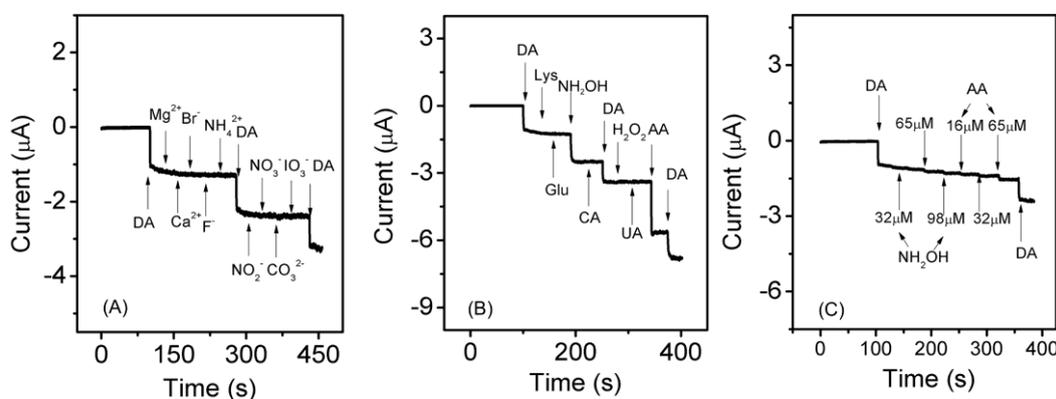
<sup>b</sup>  $i-t$ : amperometric  $i-t$  curve



**Figure 3.** (A) DPVs of (a) bare GCE and (b) Nafion/Pt/MC/GCE in 0.1 M PBS containing 0.2 mM UA, 0.2 mM DA and 2 mM AA. (B) DPVs of Nafion/Pt/MC/GCE in 0.1 M PBS with various concentrations of DA (inset: the calibration curves of the oxidation peak current versus DA concentration). (C) The amperometric current-time curves of Nafion/Pt/MC/GCE in 0.1 M PBS with successive injections of DA at the applied potential of 0.18 V (inset: the calibration curves between catalytic currents and DA concentrations).

### 3.4. Properties and application of the present sensor

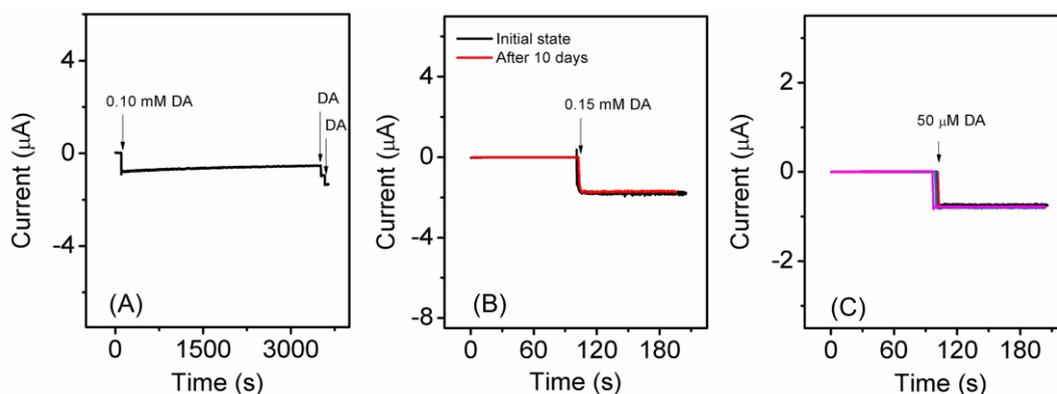
The selectivity, stability, and reproducibility of the proposed sensor were investigated to evaluate the sensing properties. To evaluate the selectivity, the effect of potential interferences on the determination of DA was demonstrated by a typical amperometric *i-t* curve recorded at the optimized potential of 0.17 V (Figure 4A-C). The presence of  $Mg^{2+}$ ,  $Ca^{2+}$ ,  $Br^-$ ,  $F^-$ ,  $NH_4^+$ ,  $NO_2^-$ ,  $NO_3^-$ ,  $CO_3^{2-}$ ,  $IO_3^-$ , L-lysine (Lys), glucose (Glu), citric acid (CA),  $H_2O_2$ , and UA did not obviously interfere with detection of 50  $\mu M$ . However, AA and hydroxylamine hydrochloride ( $NH_2OH$ ) with high concentration (0.165 mM) resulted in obvious interference.



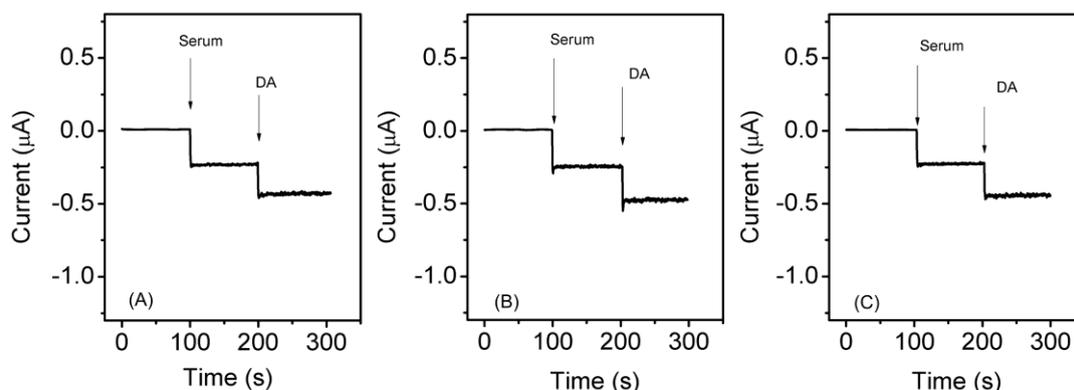
**Figure 4.** Amperometric responses of the Nafion/Pt/MC/GCE in 0.1 M PBS with successive injections of (A) 50  $\mu M$  DA, 0.165 mM  $Mg^{2+}$ ,  $Ca^{2+}$ ,  $Br^-$ ,  $F^-$ ,  $NH_4^+$ ,  $NO_2^-$ ,  $NO_3^-$ ,  $CO_3^{2-}$ , and  $IO_3^-$ ; (B) 50  $\mu M$  DA, 0.165 mM Lys, Glu,  $NH_2OH$ , CA,  $H_2O_2$ , UA, and AA. (C) 50  $\mu M$  DA, 32, 65, 98  $\mu M$  HONH<sub>2</sub>, and 16, 32, 65  $\mu M$  AA.

To understand the effect of AA and  $\text{NH}_2\text{OH}$  on the selectivity of the sensor, the experiments were repeated for the AA and  $\text{NH}_2\text{OH}$  concentrations of 16, 32, 65, and 98  $\mu\text{M}$ . The results show that no obvious increase can be found in the current response for concentrations as high as 98  $\mu\text{M}$  for  $\text{NH}_2\text{OH}$  and 65  $\mu\text{M}$  for AA. Therefore, the presented Nafion/Pt/MC material showed good selectivity for DA detection except in cases where high concentrations of AA and  $\text{NH}_2\text{OH}$  present.

Figures 5A and 5B show the operational and storage stabilities of the sensor that were evaluated by detecting DA. There is no obvious decay in the catalytic current of DA for 1 h successive response and the sensor still shows sensitive response to DA. Moreover, no loss of catalytic activity for DA was found after storage for 10 days. The results indicate that the present sensor has excellent operational and storage stability, enabling reliable analysis for DA. The reproducibility of the sensor was examined by using five modified electrodes obtained with the same treatment on five different GCEs (Figure 5C). The relative standard deviation (RSD) of 3.2% for the same DA concentration was obtained, indicating an acceptable reproducibility.



**Figure 5.** Successive current response of the Nafion/Pt/MC/GCE in 0.1 M PBS towards 0.10 mM DA for 1 h and 50  $\mu\text{M}$  DA after 1 h. (B) Current response of the Nafion/Pt/MC/GCE towards 0.15 mM DA before and after 10 days of storage. (C) Reproducibility of five Nafion/Pt/MC/GCE with five different GCE in 0.1 M PBS for injections of 50  $\mu\text{M}$  DA.



**Figure 6.** (A) Amperometric responses of the Nafion/Pt/MC/GCE in 0.1 M PBS with the additions of serum samples followed by additions of 16.5  $\mu\text{M}$  DA. (B) and (C) The determination for serum samples were performed repeatedly.

**Table 2.** Concentrations of DA in serum samples determined by the present sensor

Serum ( $\mu\text{M}$ )	Added ( $\mu\text{M}$ )	Found ( $\mu\text{M}$ )	Recovery (%)
16.2	16.5	32.6	99.4
16.4	16.5	35.0	112.7
15.7	16.5	33.5	107.9

To illustrate a practical application of routine DA analysis, the presented sensor was used to measure DA in serum samples. The results obtained by the standard addition method are shown in Figure 6 and Table 2. The mean recoveries were between 99.4 and 112.7%, which demonstrated that the proposed sensor has potential application in practical DA determination.

#### 4. CONCLUSIONS

In summary, an electrochemical sensor was developed by using electrochemical reduction of Pt NPs on the surface of MC. With the synergistic effect of Nafion film, Pt NPs, and MC, the as-prepared sensor (Nafion/Pt/MC/GCE) exhibited excellent electrocatalytic activity towards the oxidation of DA. Moreover, the oxidation potential of DA on the sensor was perfectly distinguished from those of UA and AA, and no overlap between the UA and AA peaks was observed. High stability, good reproducibility and selectivity, and attractive sensing ability for detecting DA prove that the proposed method provides a promising strategy for the detection of DA in biological samples. The obtained recoveries were between 99.4 and 112.7%, which demonstrated that the proposed sensor has potential application in practical DA determination.

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