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Journal of Composites and Compounds

Electrochemical sensing of Nalbuphine in pharmaceutical samples using amplified MgO/CNTs nanocomposite electrode

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A B S T R A C T

MgO/CNTs nanocomposite was synthesized by a simple strategy and used for modification of paste electrode. The paraffin oil (PI) and 1-Butyl-3-methylimidazolium methyl sulfate (BMMS) were used as binders for fabrication of MgO/CNTs/PI/BMMS/PE. The results confirm the 9% w/w of MgO/CNTs showed high catalytic activity on nalbuphine signal and this value was used as optimum condition for fabrication of sensor. The MgO/CNTs/PI/BMMS/PE introduce as new analytical sensor for determination of nalbuphine in the concentration range 1.0 nM – 400 μ M with detection limit 0.5 nM. In final step, the MgO/CNTs/PI/BMMS/PE was used for monitoring of nalbuphine in injection and serum samples with acceptable recovery data.

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Peer review under responsibility of JCC Research Group

A R T I C L E I N F O R M A T I O N

Article history:

Received 18 September 2021

Received in revised form 28 October 2021

Accepted 2 January 2022

Keywords:

Nalbuphine

MgO/CNTs nanocomposite

Ionic liquid

Drug monitoring

1. Introduction

Measurement of drugs has been used as one of the common methods in evaluating the quality of pharmaceuticals compounds as well as their effectiveness in biological samples [1-3]. The concentration of drugs in the body should be controlled due to the many side effects [4]. Various analytical methods for measuring drug compounds have been introduced, among which electrochemical techniques have been used more than other methods in recent years [5-8]. High voltage overvoltage and weak signal of drug compounds are the most important problems of electrochemical sensors for this purpose [9, 10]. To solve this problem, electrodes modified with conductive catalysts have been widely used [11-13]. Nanomaterials are one of the most important catalysts used to design new electrochemical sensors[14].

Nanomaterials have grown as a new phenomenon in most sciences and have given high potential to various branches of science [15-18]. Electrochemical sensors are one of the tools enhanced by nanomaterials [19-21]. The electrical conductivity of some nanomaterials, such as metal nanoparticles and carbon compounds, has led to a wide variety of high-sensitivity electrochemical sensors by researchers [22-25]. In between, MgO/CNTs nanocomposite was suggested as new and conductive electro-catalyst in fabrication of electrochemical sensors[26].

This work, focused on design and fabrication of MgO/CNTs/PI/BMMS/PE as new analytical sensor for determination of nalbuphine in pharmaceutical samples.

2. Experimental

2.1. Materials and method

Nalbuphine hydrochloride ($\geq 98\%$), magnesium nitrate hexahydrate ($\geq 99\%$), sodium hydroxide ($\geq 98\%$), SWCNTs-COOH, phosphoric acid ($\geq 85\%$) and 1-Butyl-3-methylimidazolium methyl sulfate ($\geq 95\%$) were purchased from Sigma-Aldrich. MgO/CNTs nanocomposite was synthesized by chemical precipitation strategy reported by Tahernejad-Javazmi et al. paper [26]. Linear sweep voltammetry (LSV) and differential pulse voltammetrice methods were used for electrochemical monitoring of nalbuphine.

2.2. Fabrication of MgO/CNTs/PI/BMMS/PE

The MgO/CNTs/PI/BMMS/PE was fabricated by hand mixing 90 mg MgO/CNTs + 910 mg graphite powder in the presence paraffin oil and BMMS (8:2 v/v) as binders. The MgO/CNTs/PI/BMMS/PE add in end of glass tube in the presence of copper wire and used for recording of electrochemical signals.

2.3. Real sample preparation

Nalbuphine ampoule (10 mg/mL) and serum samples were purchased from local pharmacy and diluted by PBS (pH= 7.0). The sample

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<https://doi.org/10.52547/jcc.4.1.1>

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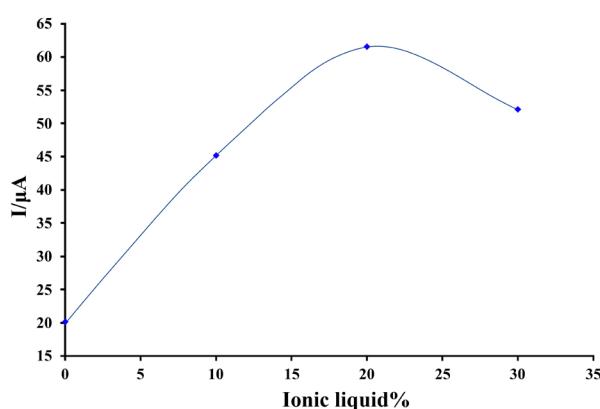


Fig. 1. Oxidation current of nalbuphine vs. BMMS percentage in fabrication of sensor.

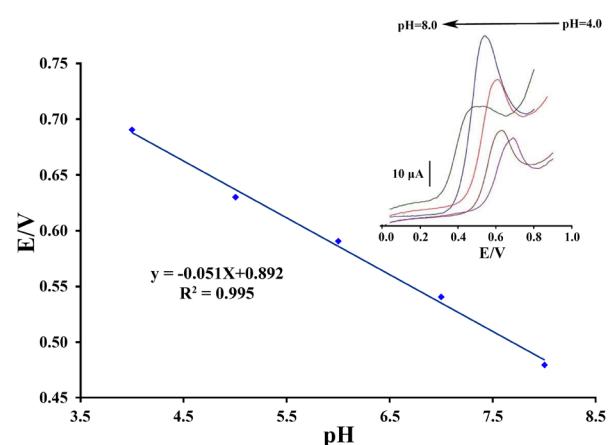


Fig. 2. Oxidation current of nalbuphine vs. MgO/CNTs percentage in fabrication of sensor.

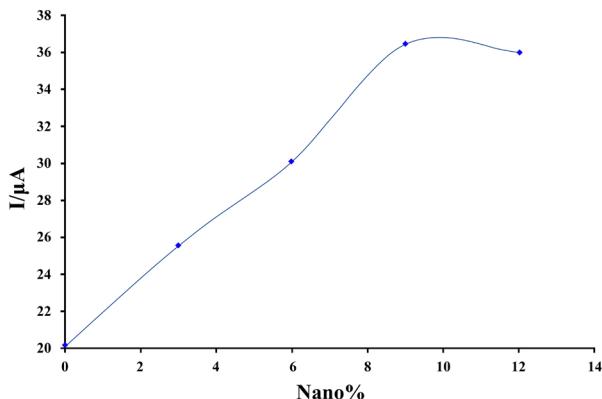


Fig. 3. E-pH curve for electro-oxidation of 500 μ M nalbuphine was recorded Mgo/CNTs/PI/BMMS/PE. Inset) Relative LS voltammograms.

was transfer into electrochemical cell and directly used for analysis of nalbuphine in real samples.

3. Results and discussion

3.1. Optimization of mediators

The values of mediators were optimized by recording of LSV 500 μ M in the optimum condition. For this goal, PE modified with different percentage of BMMS compare to PI was fabricated and oxidation signal of nalbuphine was recorded. The results showed in Figure 1 and confirm the 20% v:v of BMMS showed high catalytic activity on nalbuphine signal and this value was used as optimum condition for fabrication of sensor.

In addition, PE modified with different percentage of MgO/CNTs compare to graphite powder was fabricated and oxidation signal of nalbuphine was recorded. The results showed in Figure 2 and confirm the 9% w:w of MgO/CNTs showed high catalytic activity on nalbuphine signal and this value was used as optimum condition for fabrication of sensor.

3.2. pH optimization

The LSV 500 μ M nalbuphine was recorded MgO/CNTs/PI/BMMS/PE in the pH range 4.0 – 8.0 (Figure 3 inset). Linear plot was observed between oxidation potential of nalbuphine and pH with equation $E =$

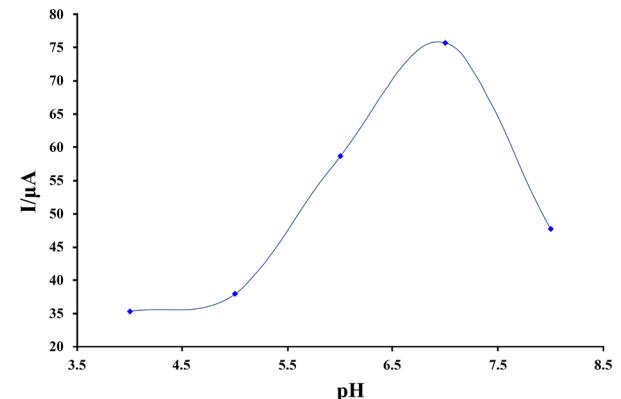


Fig. 4. I-pH curve for electro-oxidation of 500 μ M nalbuphine was recorded Mgo/CNTs/PI/BMMS/PE.

$0.051 \text{ pH} + 0.892$ ($R^2 = 0.995$), that confirm equal electron and proton in redox reaction of nalbuphine (Figure 3). In addition oxidation current of nalbuphine showed maximum oxidation current at pH=7.0 and this condition was selected as optimum condition (Figure 4).

3.3. Catalytic activity

The catalytic activity of MgO/CNTs and MgO/CNTs in paste matrix on oxidation signal of nalbuphine was tested in this step (Figure 5). For this goal, the LS voltammogram of 500 μ M nalbuphine was recorded at surface of PI/PE (curve a), MgO/CNTs/PI/PE (curve b), PI/BMMS/PE (curve c) and MgO/CNTs/PI/BMMS/PE (curve d).

As can be seen, oxidation current of nalbuphine is about 20.065 μ A, 36.45 μ A, 61.50 μ A and 76.3 μ A at a surface of PI/PE, MgO/CNTs/PI/PE, PI/BMMS/PE and MgO/CNTs/PI/BMMS/PE, respectively. As can be seen, after modification of PI/PE with MgO/CNTs and BMMS, the oxidation signal of nalbuphine was improved about 3.8 times that con-

Table 1.

Determination of nalbuphine in real samples

Sample	Added (μ M)	Expected (μ M)	Founded (μ M)	Recovery %
Injection	-	2.00	2.05 ± 0.11	-
	10.00	12.00	11.87 ± 0.45	98.91
Serum	-	-	<LOD	-
	20.00	20.00	20.98 ± 1.11	104.9

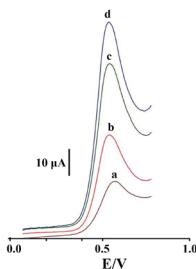


Fig. 5. LSV 500 μ M nalbuphine at surface of PI/PE (a), MgO/CNTs/PI/PE (b), PI/BMMS/PE (c) and MgO/CNTs/PI/BMMS/PE (d).

firm high conductivity and catalytic activity of MgO/CNTs and BMMS as two electro-catalysts.

3.4. Linear dynamic range and real sample analysis

The MgO/CNTs/PI/BMMS/PE was successfully used for monitoring of nalbuphine in concentration range 1.0 nM – 400 μ M with equation $I = 0.1241 C + 1.6565$ ($R^2 = 0.9968$). The MgO/CNTs/PI/BMMS/PE showed a detection limit 0.5 nM.

The ability of MgO/CNTs/PI/BMMS/PE checked for sensing of nalbuphine in real samples and data are presence in Table 1. The recovery data confirmed that MgO/CNTs/PI/BMMS/PE has good quality for sensing of nalbuphine in real samples.

4. Conclusion

The MgO/CNTs/PI/BMMS/PE was fabricated as powerful and sensitive analytical tool for monitoring of nulbuphine in pharmaceutical samples. The LSV 500 μ M nalbuphine was recorded MgO/CNTs/PI/BMMS/PE in the pH range 4.0 – 8.0. Oxidation current of nalbuphine showed maximum oxidation current at pH=7.0 and this condition was selected as optimum condition. The results showed MgO/CNTs/PI/BMMS/PE can be detected nulbuphine in the concentration range 1.0 nM – 400 μ M with detection limit 0.5 nM. The recovery data confirmed that MgO/CNTs/PI/BMMS/PE has good quality for sensing of nalbuphine in real samples.

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