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ORIGINAL PAPER

Oxidation and determination of Gabapentin on nanotubes of nickel oxide-modified carbon paste electrode

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Abstract The electrooxidation of *Gabapentin* was studied on nanotubes of nickel oxide-modified carbon paste electrode for the first time. Cyclic voltammetry was employed to investigate the electrooxidation process. A simple, sensitive, and efficient amperometric method was developed for the analysis of the drug, and the corresponding analytical parameters were reported. For Gabapentin, a detection limit of 0.3 μM was obtained in a linear range of 2.4–50 μM. The proposed amperometric method was also applied to the analysis of commercial capsules, and the results were in good agreement with the declared values. Also, the applicability of the method to the direct assay of the drug in human serum and urine was described.

Keywords Gabapentin · Nickel oxide nanotube · Electrocatalysis · Modified electrode

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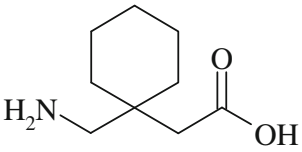
Introduction

Carbon paste electrodes (CPEs) have been widely applied in electroanalysis due to their vast advantages such as low cost, ease of fabrication, miniaturization, surface renewability, compatibility toward different compounds as modifiers, wide potential window, low-charging current, and long life [1–5]. In addition, CPEs can be modified with various compounds such as metals, metal oxides, and biomacromolecules, such as DNA and enzymes, so as to enhance mechanistic studies and the selectivity and sensitivity of electroanalysis studies [3]. The metal oxide particle-modified CPEs include both advantages of carbon pastes and electrocatalytic reactivity of metal oxide surfaces [1–8].

Gabapentin, 1-(aminomethyl) cyclohexane acetic acid, (with the trade name Neurontin, Scheme 1) as a new antiepileptic drug, is used clinically for the treatment of partial onset seizures with or without secondary generalized tonic–clonic convulsions [9]. Gabapentin is also effective in the prevention of frequent migraine headaches, neuropathic pain, and nystagmus and treatment of nerve pain caused by herpes virus or shingles [10].

A variety of techniques have been proposed for the determination of Gabapentin. These include voltammetry [11], gas chromatography [12], gas chromatography–mass spectrometry [13], liquid chromatography–mass spectrometry [14], high performance liquid chromatography (HPLC) [15–17], and capillary electrophoresis [18]. However, sensing of Gabapentin is not simple since this compound lacks natural chromophore or fluorophore for ultraviolet and visible photometric or fluorometric detections. The photometric or fluorometric detections were performed by an extraction following by pre-column derivatization of Gabapentin with a suitable reagent. On the other hand, the

Scheme 1 The chemical structure of Gabapentin



derivatization procedure suffers from the points of time consuming, possibility of incomplete derivatization, addition of detection interferences, and increasing method complexity and costs. Despite wide use of Gabapentin, a simple and reliable analytical technique is required for its assay in bulk drug and pharmaceutical formulations. Analysis of Gabapentin in pharmaceutical formulations is also quite limited and includes spectrofluorometry [19] and colorimetric detection [20] and one HPLC [21] (all of them require derivatization or capillary electrophoresis).

In the study described here, the electrooxidation and determination of Gabapentin at a nickel oxide nanotube-modified carbon paste electrode was investigated. To the best of our knowledge, this procedure has never been carried out successfully.

Experimental section

Chemicals and reagents

All chemicals used in this work were of analytical grade from Merck and were used without further purification. Multi-wall carbon nanotubes (MWCNTs) were received from PlasmaChem GmbH, Germany. Carbon powder with a particle size of less than 50 μm was obtained from Merck. All solutions were prepared by redistilled water. Gabapentin was received from the Center of Quality Control of Drug, Tehran, Iran. The Gabapentin capsules were obtained from a local drugstore. Standard solutions of authentic drug were prepared by dissolving an accurate mass of the bulk drug in an appropriate volume of 100 mM NaOH solution (which was also used as the supporting electrolyte).

Apparatus

Electrochemical measurements were carried out in a conventional three-electrode cell powered by a μ-Autolab potentiostat/galvanostat, type III (Eco Chemie, Utrecht, The Netherlands). The system was run on a PC using GPES 4.9 software. A saturated Ag/AgCl and a platinum disk were used as the reference and counter electrodes, respectively.

To obtain information about the structure and size of nickel oxide nanotubes, scanning electron microscopy was performed using a X-30 Philips instrument and transmission electron microscopy (TEM) was performed using a CEM 902A ZEISS instrument with an accelerating voltage of 80 kV. For TEM, samples were prepared by placing a drop of the particles, dispersed in acetone, on a carbon-covered nickel grid (400 mesh) and evaporating the solvent. X-ray diffraction (XRD) patterns were measured by using a Philips X'Pert, The Netherlands, through Cu Ka radiation at 40 kV and 30 mA.

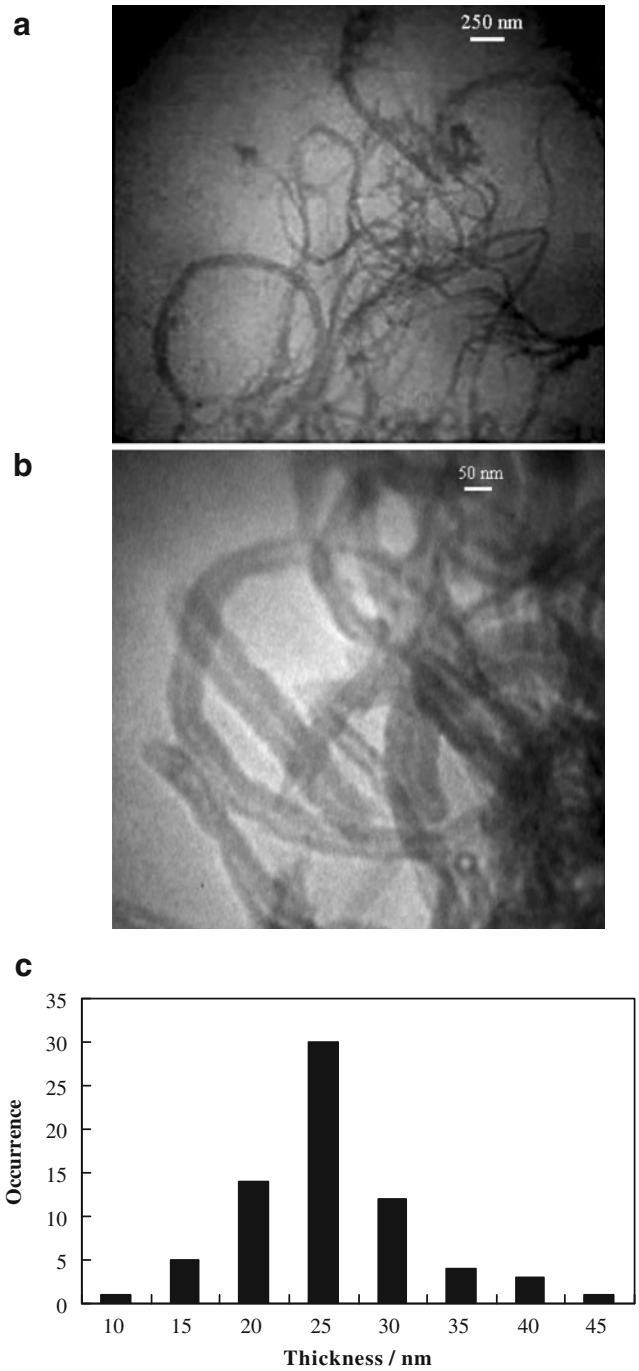


Fig. 1 **a** TEM image of MWCNTs. **b** TEM image of the nickel oxide nanotubes. **c** The corresponding size distribution histogram of the thickness of the oxide layer (the data was obtained from ten images, which contained an average of seven number of tubes)

Preparation of nickel oxide nanotubes

Nickel oxide nanotubes were synthesized using the scaffold of MWCNTs. MWCNTs were firstly pretreated as described previously [22]. For this purpose, MWCNTs were refluxed in a 2.0 M nitric acid solution for 12 h, washed with redistilled water several times, then dried and stored until use. Then MWCNTs were sonicated in a 3:1 sulfuric/nitric acid solution for 6 h in an ultrasonic bath at room temperature and then washed with distilled water until neutralization by vacuum filtration. The obtained sample was taken and dried overnight at 50 °C. This procedure causes scission and carboxylation of MWCNTs and therefore, they acquire negative charges on the outside of the tubes. An aqueous solution of nickel nitrate (5 mL, 0.05 M) was prepared separately and mixed with 200 mg MWCNT. The mixture was sonicated and then heated in an oven at 80 °C for 15 min and continued to heat to dry the sample. Then, the dried mixture was put into a microwave worked at 2.45 GHz and a fixed power level of 850 Watts. The mixture was 20 s irradiated following a 60-s rest, and the microwave irradiation procedure was repeated ten times to produce nickel oxide nanotubes.

Preparation of nickel oxide/carbon microparticle

For comparison, nickel oxide/carbon microparticles were similarly synthesized in the same way that nickel oxide nanotubes were synthesized, rather than carbon powder was employed instead of MWCNTs.

Preparation of the working electrodes

Unmodified carbon paste electrode (UCPE) was prepared by hand-mixing carbon powder and mineral oil with a 80%/20% (*w*/*w*) ratio. The paste was carefully mixed and homogenized in an agate mortar for 20 min. The resulting paste was kept at room temperature in a desiccator before use. The paste was packed firmly into a cavity (1.0 mm diameter and 0.5 mm depth, surface area of 0.0079 cm^2) at the end of a Teflon tube. Electrical contact was established via a copper wire connected to the paste in the inner hole of the tube. The electrode surface was gently smoothed by rubbing on a piece of weighing paper just prior to use. This procedure was also used to regenerate the surface of UCPE.

Nickel oxide/carbon microparticle-modified carbon paste electrode (m-MCPE) was prepared by hand-mixing carbon powder, mineral oil, and nickel oxide/carbon microparticles with a 67.5/20/12.5% (*w*/*w*) ratio. The electrode then transferred to a 100-mM NaOH solution and 25 consecutive cyclic potentials were done in a regime of cyclic voltammetry. This procedure causes some structural changes and transformation of the nickel oxides to stable forms [7, 23].

Nickel oxide nanotube-modified carbon paste electrode (n-MCPE) was prepared by hand-mixing carbon powder, mineral oil, and nickel nanotube with a 67.5/20/12.5 (*w*/*w*) ratio. Similar to m-MCPE, the electrode then transferred to a 100-mM NaOH solution and 25 consecutive cyclic potentials were done in a regime of cyclic voltammetry.

General procedures

Standard solutions of the drug were prepared by dissolving an accurate mass of the bulk drug in an appropriate volume of 100 mM NaOH solution. All solutions were kept in the dark at 4 °C and were used within 24 h to avoid decomposition. Additional dilute solutions were prepared daily by accurate dilution just before use. However, amperograms of the drug solutions recorded a week after preparation did not show any significant change in the assay values.

The calibration curves for the drug in 100 mM NaOH solution were measured with the amperometry technique. Working potential of 670 mV was used in amperometric measurements, in which the transient currents were allowed to decay to steady-state values. All studies/measurements were carried out at room temperature.

Gabapentin capsule assay procedure

In order to analyze the drug capsules, ten capsules were weighed and the contents emptied into a mortar. The empty capsule shells were weighed to determine the average fill weight in each capsule. The fill material was gently ground using a pestle for some minutes to break any aggregated or cemented material. Appropriate accurately weighed amounts of the homogenized powder were transferred into 100 mL calibrated flasks containing 50 mL of 100 mM

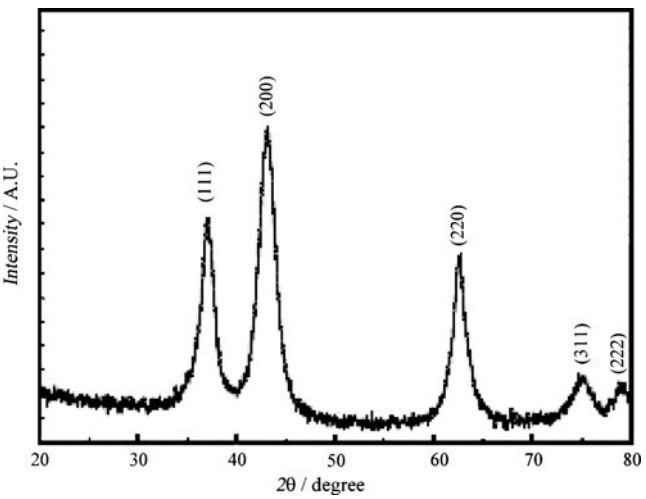


Fig. 2 An XRD pattern of as synthesized nickel oxide nanotubes

NaOH solution. The contents of the flasks were sonicated for 30 min, and then the undissolved excipients were removed by filtration and diluted to volume with the supporting electrolyte. Appropriate solutions were prepared by taking suitable aliquots of the clear filtrate and diluting them with 100 mM NaOH solution.

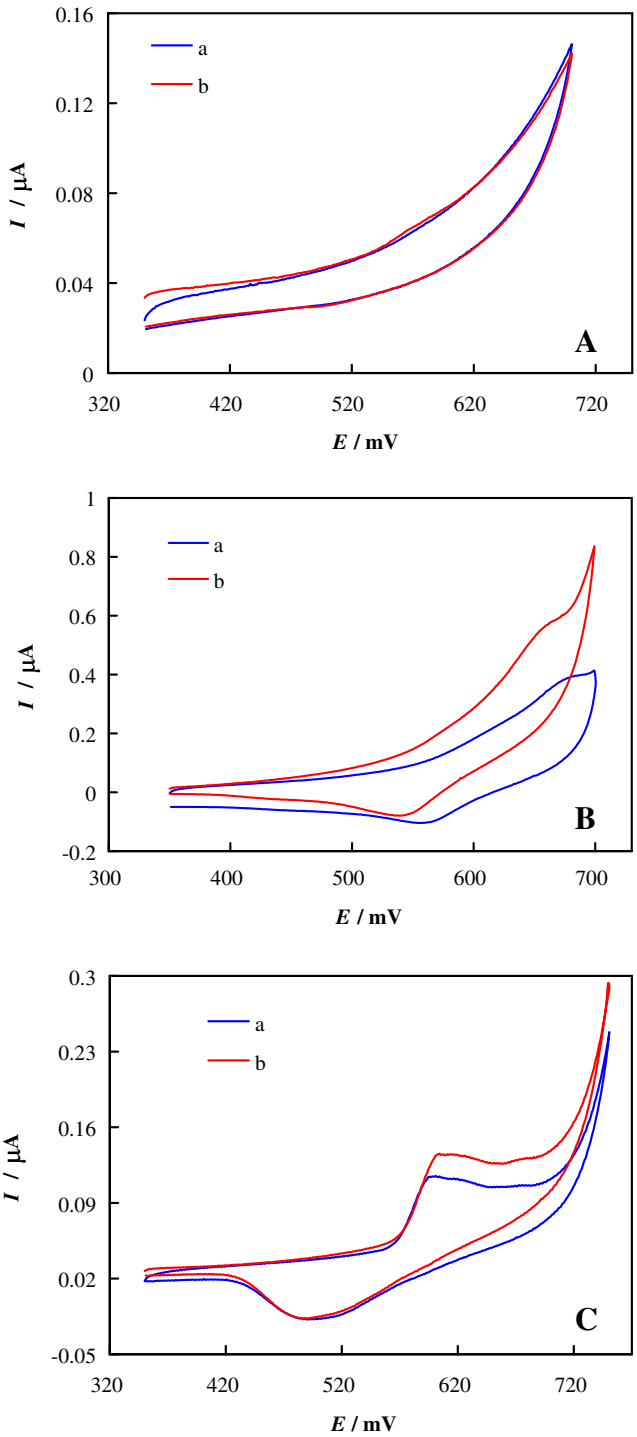
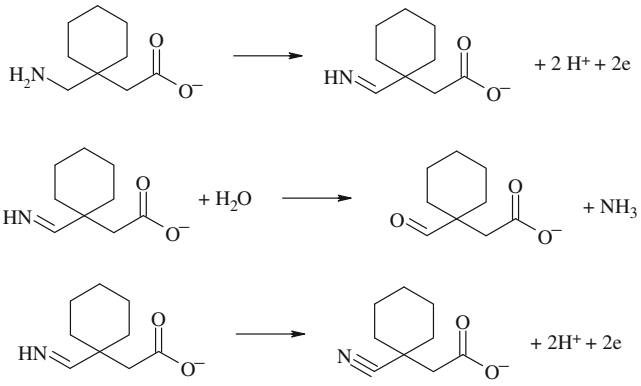


Fig. 3 Cyclic voltammograms of 100 mM NaOH solution obtained using **a** UCPE, **b** n-MCPE, and **c** m-MCPE in the absence (*curves a*) and presence (*curves b*) of 50 µM Gabapentin. The potential sweep rate was 10 mV s^{-1}



Scheme 2 The proposed reaction mechanism for the electrooxidation of Gabapentin

Analysis of spiked urine and human serum samples

It is known that Gabapentin is not metabolized and mainly excreted by kidney. Also, it does not bind plasma proteins and its pharmacokinetics is not affected by foods and other drugs [24, 25]. Therefore, it analyzes directly in biological fluids. Drug-free human and urine serum samples, obtained from healthy volunteers, were filtrated through a filter paper and stored frozen until the assay. For the analysis of Gabapentin in these biological fluids, known amounts of Gabapentin were added into a solution of Gabapentin-free urine and serum samples, which was further diluted with the supporting electrolyte. The calibration curves for Gabapentin in human serum blood and urine samples were measured with the amper-

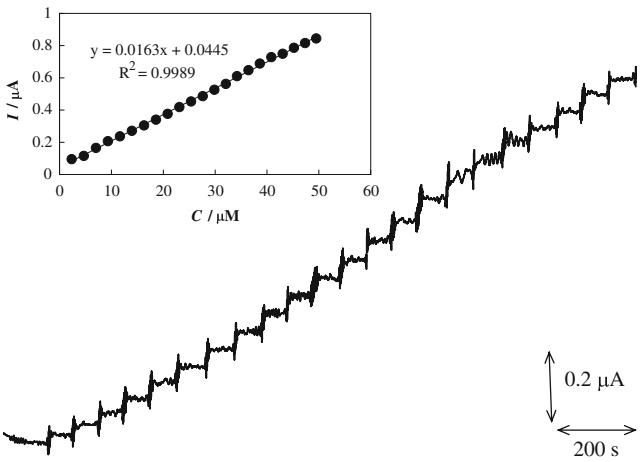


Fig. 4 Typical amperometric signals obtained during successive increments of Gabapentin to a 100-mM NaOH solution using n-MCPE. The applied potential was 670 mV. *Inset* the corresponding calibration curve

Table 1 The determined parameters for calibration curve and accuracy and precision (n=3) for oxidation of Gabapentin on n-MCPE

Linear range (μM)	2.4–50
Slope (A M^{-1})	(0.016±0.004)
Intercept (A)	(0.045±0.007)×10^{-6}
LOD (μM)	0.3
LOQ (μM)	1.0
RSD (%)	3.9%
Bias[a] (%)	2.4%

RSD relative standard deviation

[a] The value was reported for 20 μM

ometric technique under the same conditions as for pure Gabapentin.

Recovery experiments

In order to study the accuracy and reproducibility and to check the interference from the excipients used in the formulations of these techniques or present in the biological fluids, recovery experiments were carried out using the standard addition method. For this purpose, known amounts of Gabapentin were added to the pre-analyzed capsule dosage form, serum and urine. The mixtures were analyzed by the proposed amperometric technique. After five repeated experiments, the recovery results were obtained.

Results and discussion

Electron microscopy

Figure 1 shows a TEM image of MWCNTs (a), a TEM image of the nickel oxide nanotubes (b), and a size distribution histogram of the thickness of the nickel oxide layer (c). It can be seen that the long fila of MWCNTs are relatively twisted together and have an average diameter of 10–50 nm and of 0.5–1 μm length. Moreover, nickel oxide precipitated on the outside of the tubes of carbons and covered the "hard template" of MWCNTs as a shell and formed tubes of nickel oxide. The average thickness of the tubes of nickel oxide is about 25 nm.

XRD measurements

Figure 2 shows an XRD pattern of as synthesized nickel oxide nanotubes. In the pattern, slightly broadened peaks are appeared. It is due to the nanometer-size effect of the samples [26]. Main diffraction peaks at 37.3°, 43.3°, 62.9°, 75.4°, and 79.4° are indexed to (1 1 1), (2 0 0), (2 2 0), (3 1 1), and (2 2 2) reflections, respectively. The peak positions are in good agreement with the standard diffraction data of powder cubic nickel oxide, indicating that the nanotubes are polycrystalline with face center cubic structure [27].

Microwave irradiation as one of thermophysical methods has been employed for the synthesis of nanostructured materials. This method has a high penetration depth of heat,

Table 2 Determination and recovery of Gabapentin in capsules

Sample type	Amount labeled/mg	Amount added/mg	Amount found/mg	Recovery (%)	RSD (%)	Bias (%)
(1)	100	–	103.9	103.9	2.0	3.9
(1)	–	100	95.7	95.7	3.1	−4.3
(1)	–	100	96.5	96.5	3.5	−3.5
(2)	100	–	97.4	97.4	3.2	−2.6
(2)	–	100	97.7	97.7	3.6	−2.3
(2)	–	100	98.6	98.6	3.0	−1.4
(3)	300	–	291	97.0	1.1	−3.0
(3)	–	300	314.5	104.8	1.8	4.8
(3)	–	300	290.4	96.8	1.3	−3.2
(4)	300	–	290.8	96.9	5.1	−3.1
(4)	–	300	294	98.0	2.8	−2.0
(4)	–	300	291.3	97.1	4.7	−2.9
(5)	300	–	297.1	99.0	2.2	−1.0
(5)	–	300	306.1	102.0	3.9	2.0
(5)	–	300	302.2	100.7	3.3	0.7
(6)	400	–	409.1	102.3	1.3	2.3
(6)	–	400	407.6	101.9	1.9	1.9
(6)	–	400	410.6	102.6	2..1	2.6

Table 3 The determined parameters for the calibration curve of Gabapentin spiked to human serum and urine samples

	Serum	Urine
Linear range (μM)	2.4–50	2.4–50
Slope (A M^{-1})	0.010±0.005	0.011±0.004
Intercept (A)	$(0.050\pm0.007)\times10^{-6}$	$(0.071\pm0.003)\times10^{-6}$
R^2	0.99	0.99
LOD (μM)	0.6	0.5
LOQ (μM)	2.0	1.7
RSD (%)	3.7	3.1

is very fast and uniform, thermal gradients are minimized, the time for the particle diffusion is reduced, and hence, the products can be obtained in a relatively short time [28–30]. In addition, it is well-known that thermal decomposition of nickel nitrate results in pure nickel oxide and microwave decomposition of nickel nitrate to nickel (II) oxide has been reported elsewhere [29]. Moreover, the carbonaceous materials enhance the microwave absorption and act as susceptors [29]. In the present work, MWCNTs act as both the susceptor (which absorb the microwave) and a scaffold (a hard template) and nickel nitrate was decomposed to the nickel oxide under microwave irradiation (Fig. 2).

Cyclic voltammetry

Figure 3a shows cyclic voltammograms of 100 mM NaOH solution obtained using UCPE in the absence (curve a) and presence (curves b) of 50 μM Gabapentin. The potential sweep rate was 10 mV s^{-1}. Gabapentin represented no peak in the voltammogram indicating electroinactivity of Gabapentin on carbon-based surfaces in the swept potential range. Figure 3b represents typical cyclic voltammograms of 100 mM NaOH solution obtained using n-MCPE in the absence (curve a) and presence (curves b) of 50 μM Gabapentin. The potential sweep rate was 10 mV s^{-1}. In the absence of the drug, a pair of redox peak with a mid-peak potential of 617 mV appeared in the voltammogram. The voltammograms shown are similar to those previously reported [7, 31, 32], and the redox transition involved is attributed to the transformation of Ni(II)/Ni(III) species immobilized at the electrode surface. Nickel oxide nanotubes were transformed to $Ni(OH)_2$ counterparts by hydration upon exposure of the electrode surface to the solution. Then, the modifier is oxidized from $Ni(OH)_2$ to NiOOH back and forth by potential cycling. In the presence of Gabapentin, the anodic current and the associated anodic charge increased dramatically, whereas the cathodic current and the corresponding charge decreased. Regarding the reaction product(s) of the electrooxidation process on n-MCPE, Gabapentin as a primary straight chain amine can be oxidized on nickel-based electrodes to the corresponding imine, nitril, and/or aldehyde analogs [32–34], as shown in Scheme 2.

It is well-known that the overall reactivity of an electrode modifier can be size- and shape-dependent [7, 35]. Figure 3c represents cyclic voltammograms of 100 mM NaOH solution obtained using m-MCPE in the absence (curve a) and presence (curves b) of 50 μM Gabapentin. The potential sweep rate was 10 mV s^{-1}. Although nickel-based electrodes show similar signatures in the cyclic voltammograms (Fig. 3b and c) and the voltammograms represented here are similar to those reported for other nickel-based electrodes [7, 31, 32], the currents in the voltammograms using n-MCPE are much higher. Although both electrodes are fabricated using the same mass of active nickel species, the nanotubes of nickel oxide can represent a higher surface area and/or the nanosize effect. From the cyclic voltammograms represented in Fig. 3b and c, it can be seen that the currents were higher for n-MCPE compared with m-MCPE. This can be initially related to the higher surface area of n-MCPE. Therefore, it will cause higher sensitivity for the amperometric sensor constructed based on n-MCPE (vide infra). On the other hand, the surface effect-free efficiencies for the composites (for the same concentration of Gabapentin) can be calculated as: $(I_{Gabapentin} - I_b)/I_b$, where $I_{Gabapentin}$ is the anodic current in the presence of Gabapentin and I_b is the base anodic current. These efficiencies were obtained as 0.12±0.05 and 0.62±0.06 for m-MCPE and n-MCPE, respectively. This will also cause a higher sensing utility which is due to the nature of the nickel oxide nanotubes. However, the higher efficiency of n-MCPE is related to the nanosize effect of n-MCPE and/or the higher apparent

Table 4 Recovery of Gabapentin in human serum and urine samples

Sample	Amount added (μM)	Amount found (μM)	Absolute recovery (%)	RSD (%)
Serum	10	10.2	102	3.5
	20	20.2	101	3.4
	30	31.0	103.3	3.9
Urine	10	10.3	103	3.7
	20	21.1	105.5	3.1
	30	31.7	105.7	3.4

charge-transfer rate constant. Nanotubes of nickel oxide are stably distributed on the carbon surface which is fully and easily accessible for the substrates, and consequently, can be readily and completely used as an electrochemical sensing unit.

Electroanalysis of Gabapentin

In order to develop a simple and time-saving procedure for the analysis of Gabapentin in pure form as well as pharmaceutical formulations, the amperometry technique was employed. Typical amperometric signals obtained during successive increments of Gabapentin to a 100-mM NaOH solution using n-MCPE are depicted in Fig. 4. Gentle stirring for a few seconds was needed to promote solution homogenization after each injection. The electrode response is quite rapid and proportional to the Gabapentin concentration. The corresponding calibration curve for the amperometric signals is shown in Fig. 4, inset. The limits of detection (LOD) and quantitation (LOQ) of the procedure were calculated according to the 3SD/m and 10SD/m criteria, respectively, where SD is the standard deviation of the intercept and *m* is the slope of the calibration curves [36]. The determined parameters for calibration curves of drug, accuracy and precision, LOD and LOQ, and the slope of calibration curves are reported in Table 1. The precisions of the method are calculated as the relative standard deviation. The procedure was repeated on the same day on the same spiked solutions at concentrations in the range of the standard series.

The applicability of the proposed amperometric method for the sample dosage form was examined by analyzing the capsules. It was found that the amounts of drug determined using this method are in good agreement with the reported values. The values of experimentally determined drugs and declared values in capsules are tabulated in Table 2.

The applicability of the proposed amperometric method for the determination of Gabapentin in biological fluids of human serum blood and urine was attempted. Amperometric signals were recorded for Gabapentin spiked to serum and urine samples using an applied potential of 670 mV. The results are listed in Table 3.

The percentage recovery of Gabapentin was determined by comparing the currents of a known drug concentration in human serum and urine with their equivalents in calibration curves; these results are summarized in Table 4. Good recoveries of Gabapentin were achieved from these matrices, denoting that application of the proposed amperometric method to the analysis of Gabapentin in biological fluids could be easily assessed.

Selectivity of the amperometric procedure for the assay of Gabapentin was examined in the presence of some common excipients in the same ratios usually used in pharmaceutical preparations (for example, gelatin, talc, cornstarch, titanium dioxide, and yellow iron oxide). The results showed no significant interference from excipients of capsules of Gabapentin. Therefore, the procedure was able to assay Gabapentin in the presence of excipients and hence, it can be considered selective.

Conclusion

A carbon paste electrode modified with nickel oxide nanotubes was prepared with carbon microparticles, Nujol, and nickel oxide nanotubes. It was employed, for the first time, for the electrooxidation and determination of Gabapentin. The higher current of electrooxidation of Gabapentin using nickel oxide nanotubes compared to those of microsize counterparts (nickel oxide/carbon microparticle) was related to the acceleration of the electrooxidation process by nanosize effect of the nanotubes. An amperometric procedure was successfully applied for the quantification of the Gabapentin with high sensitivities in pharmaceutical samples and also biological fluids. The limit of detection of the procedure was better than those reported in the literature [13, 16, 17, 37].

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