International Journal of Sciences

Research Article Volume 13 – February 2024 (02)

A Study on the Nicotine Testing Method Using Nano-Structured Fe Doped MgNi2O3

Lei PENG✏, Xi DING, Yifei XU✏, Ellie Vaisman

Abstract: In this study, we have synthesized MgNi2O3 and 2.5wt% Fe-MgNi2O3 nanoparticles by sol-gel combustion method and characterized by powder XRD, FTIR, FESEM and cyclic voltammetry, square wave voltammetry, and amperometric method for confirmation of structural, shape, morphological and electrochemistry properties. These studies reveal that the particles have spherical in shape and unique electrochemical properties. An electrochemical sensor based on 2.5wt% Fe- MgNi2O3 nanoparticles (NP) modified glassy carbon electrode (GCE) has been fabricated and used for electrochemical determination of Nicotine (NIC) as an analyte. Iron-doped MgNi2O3 modified GCE shows one strong well-defined separate oxidation peak of NIC in the experiment of cyclic voltammetry and square wave voltammetry. The modified electrode displayed a Nicotine Testing Platform with high selectivity, sensitivity, good stability, and reproducibility.

Background

Nicotine is one of the highly toxic alkaloids of the Vishnu group of alkaloids, mainly found in tobacco and cigarette smoke. When nicotine enters the human body, it will be transmitted through the bloodstream, stimulating the central nervous system, cholinergic nerves, sympathetic nerves, and then stimulate the release of dopamine, which can lead to euphoric, so nicotine is also one of the most common addictive substances, which will cause psychological and physiological dependence on withdrawal. Prolonged nicotine use can cause heart rate to increase, which induces high blood pressure and reduces appetite. Numerous studies have shown that high doses of nicotine can cause vomiting and nausea, and is especially associated with lung, laryngeal, lip, tongue, oesophageal, bladder, and kidney cancers, among many other diseases, and in severe cases, death. On the other hand, nicotine has its medicinal value, as it is effective in the treatment of ulcerative colitis, Alzheimer's disease and Parkinson's disease.

The main methods for the determination of nicotine are gravimetric analysis, titrimetric analysis, infrared spectroscopy, atomic absorption spectrophotometry, chromatography, enzyme-linked immunosorbent assay (ELISA), etc. Some of these methods have many disadvantages. Some of the above methods have many disadvantages, for example, some methods require preliminary extraction and purification of nicotine from the sample, and require skilled personnel, high extraction process and expensive equipment. For example, some methods require preliminary extraction and purification of nicotine from the sample, and require skilled personnel, high extraction techniques and expensive equipment, which puts high demands on the test; some methods use a large number of samples, and sample loss and cost control are problems that need to be solved.Due to the high hazardousness of nicotine, how to detect the presence of trace nicotine in food, drugs, fertilisers and other chemicals conveniently, accurately and at low cost has become a research hotspot, and the study of nicotine-sensitive materials is one of the most important. In this paper, iron(Fe)-doped magnesium sickle trioxide (MgNizOs) nanoparticles were prepared by sol-gel method and modified with glassy carbon electrodes to fabricate electrode sensors for accurate detection of the presence of nicotine. Experiments showed that the sensor significantly improved the electrocatalysis of oxidised nicotine and enhanced the voltammetric response. The sensor also showed good selectivity, stability, reproducibility, and ease of preparation.

Part 1 Experimental preparation of pure MgWOs and Fe-MgNi2O3

In this study, pure MgNi2O3 and 2.5 wt% Fe-MgNi2O3 nanoparticles were prepared by sol-gel method. The experimental-grade raw materials used for the preparation of the above two samples were Mg(NO3)2, Ni(NO3)2, and Fe(NO3)3, which were taken in certain proportions and stoichiometrically measured, and melted into 50 ml of demineralised water, and stirred for 30 min (300 rpm, the same as the following) using a standard magnetic stirrer. After stirring, then mixed the above three solutions and stirred for another 30 min; after that, use a buret to slowly add citric acid (C6H8O7) as a reducing agent to the above mixed solution, and stirred again for 30 min, and measured the pH value of 3. In order to make the chemical reaction of generating the target samples take place, added ammonia to the above solution slowly, and kept the pH value of the mixed solutionin the range of 8-8.5, in order to create the target samples. 8.5, in order to create an alkaline environment; then 10 ml of PEG6000 solution as surfactant was added

Published at: http://www.ijsciences.com/pub/issue/2024-02/
DOI: 10.18483/ijSci.2746; Online ISSN: 2305-3925; Print ISSN: 2410-4477

✏ Lei PENG, Yifei XU (Correspondence)
☎ +

dropwise to the above solution and stirred for 30 min, so that the solution was fully mixed; the above solution was placedin a magnetic stirrer and stirred for 30 min, and the temperature of the solution was maintained at 80t; after that, the solution was left to stand, and the precipitate was found, and the gel was formed. The samples obtained by the author were annealed at a temperature of 6000 storage for 2h, the heating rate was kept at 40 storage/min, and then slowly cooled; Finally, the annealed samples were ground to obtain the final pure MgNi2O3 and 2.5 wt% Fe-MgNi2O3 nanoparticles.

Part 2 Characterisation Study

2.1 X-ray diffraction analysis (XRD)

XRD was carried out using Cu-Kα as x-ray source (λ=1.5406A), θ range was 2.5-40° ,rate was 0.01° and the dimensions of the nanoparticles were calculated by using the Debye-Scherrer formula (d=0.9 X / p cos 0). Figure 1 shows the XRD image with diffraction peaks2 θ values of 37.29, 43.33, 62.91, 75.44, 79.43 corresponding to (111), (200), (220), (311) and (222) respectively. A sharp peak at 2 θ, about 43° (200), was proved to be a single-phase MgNi2O3 by consulting JCPDS-00-034-0410, according to the Debye-Scherrer formula D = kX/pcos θ (where "D" is the grain size and "X"is the x-ray wavelength). " is the x-ray wavelength (1.54A0), " θ" is the diffraction angle, " β" is the half-maximum full width (HMFW) and "k" is the constant 0.89), yielding an average particle size of 41 ~70 nm for the nanoparticles.

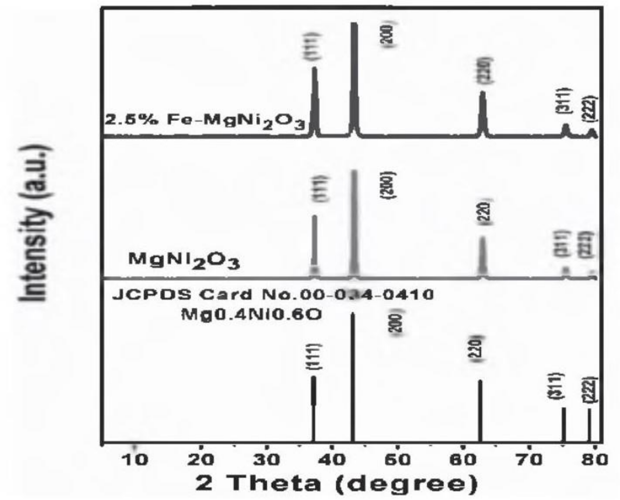


Fig. 1 Nanostructured pure MgNi2O3 and 2.5 wt% Fe-MgNi2O3 XRD images

2.2 Infrared spectral analysis (FTIR)

Figure 2 shows the infrared spectral image of the sample, from which the peak absorption wavelengths of the sample compounds are observed to be 3750 (cm -1) CO-H stretching), 2910 cm7 and 2841 cm_1 (H-C=0:C-H stretching), 2348 cm_1 (O-H Hydrogen stretching), 1558 cm_1 (O-H bending), 659 cm-1 (C-0 vibration) , 452 cm_\ 425 cm_1 (M-0 stretching), these above mentioned chemical bond absorption peaks fully confirm the presence of metal oxides in the samples.

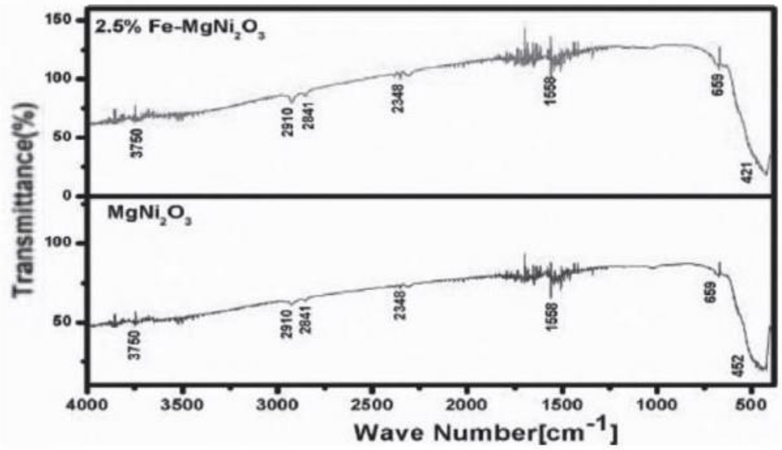
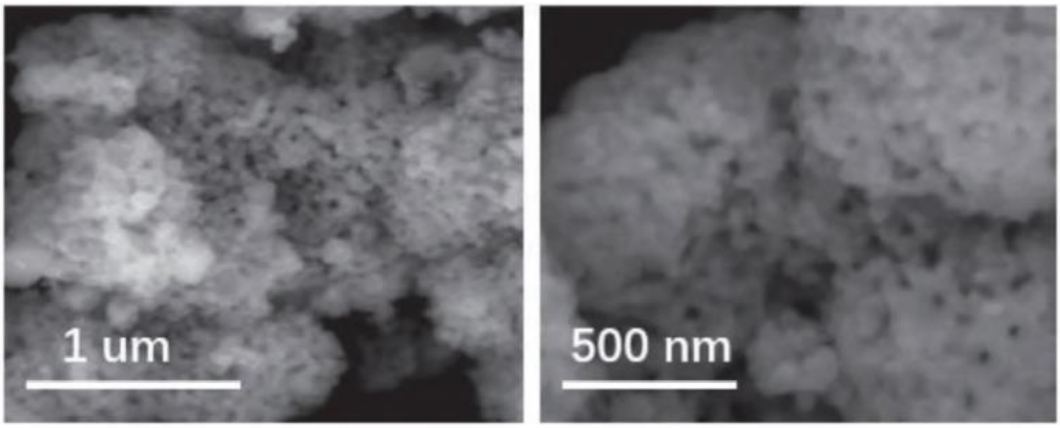


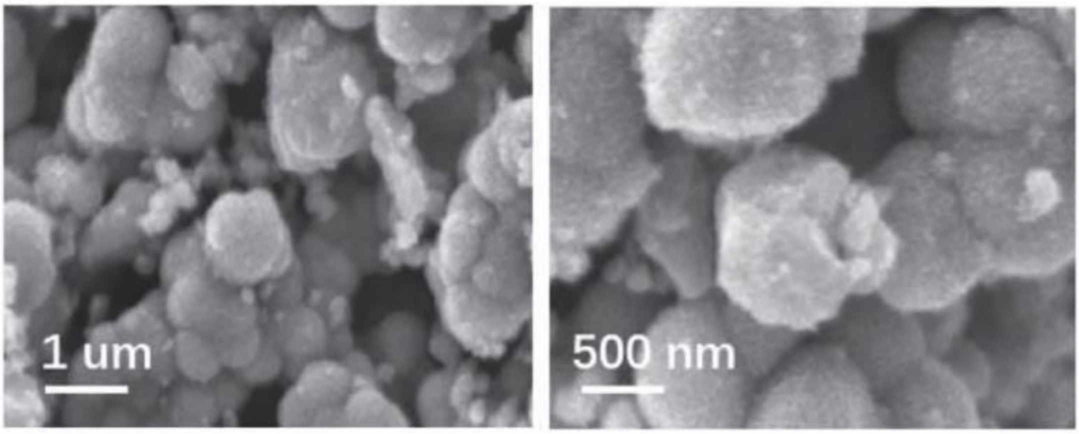
Fig. 2 Nanostructured pure MgNi2O3 and 2.5 wt% Fe-MgNi2O3 infrared spectral images

2.3 Field Emission Scanning Electron Microscopy Analysis (FE-SEM)

Figure 3 shows the surface morphology of pure MgNi2O3 and 2.5 wt% Fe-MgNi2O3 nanoparticles analysed by field emission scanning electron microscopy. Compared with the SEM images of pure MgNi2O3, Fe-MgNi2O3 has a very good structural confinement effect, which makes its morphology show different spherical particles, and the grains are well defined between the particles. In Fig. 3, it can be clearly seen that the Fe-MgNi2O3 nanoparticles are spherical in shape, and compared with the MgNi2O3 nanoparticles, the Fe-doped nanoparticles have a larger particle size is larger, with higher porosity and obviously spherical structure.



(a) Electric field emission scanning electron microscope image of nanoparticles of pure MgNi2O3 sample



(b) Electric field emission scanning electron microscopy of nanoparticles of 2.5wt% Fe-MgNi2O3 sample microscope images

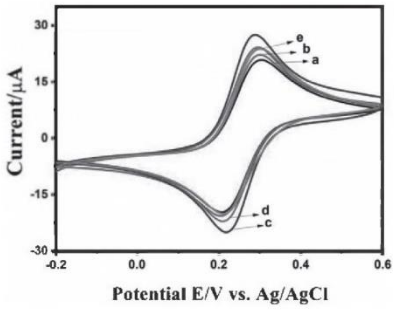
Fig. 3 Scanning electron microscope image of sample nanoparticles with electric field emission

2.4 Electrochemical characterization

The author used cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) to study the electrochemical characterisation of the samples.

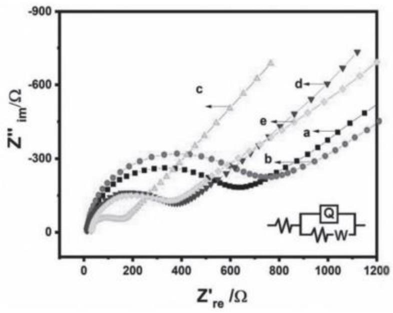
(EIS) for electrochemical characterisation of the samples; firstly, five polished glassy carbon electrodes (GCE) were prepared as working electrodes, and the working surfaces of the glassy carbon electrodes were grinded with 0.05 (μm) alumina powder abrasive solution to remove the impurities, and then washed with demineralised water for a number of times, and set aside; 1 mg of each of the four samples, namely, pure MgO, NiO, MgNi2O3, and 2.5 wt% of Fe- MgNi2O3 nanoparticles were dispersed in 1 ml of demineralised water (MgO and NiO were experimental- grade samples purchased according to the present experiment, and MgNi2O3 and 2.5 wt% Fe- MgNi2O3 were prepared by the sol-gel method in the previous section), and then the above four sample solutions were ultrasonicated for 20 min, so as to make the sample nanoparticles completely dispersed in the mineral water. in mineral water. Five polished glassy carbon electrodes prepared previously were used to carry the analysed samples (a) Bare/GCE, (b) MgO/GCE, (c NiO/GCE, (d MgNi2O3/GCE, and (e) 2.5 wt% Fe-MgNi2O3/GCEo The other two electrodes used for the cyclic voltammetry method were the Ag/AgCI reference electrode and the clamp-wire auxiliary electrode, respectively, and the scanning rate was 50 mVs^.

Figure 4 shows the electrode oxidation-reduction curves of the above five samples determined by cyclic voltammetry, and it can be clearly seen that the peak current (Ipa) values of Bare/GCE,MgO/GCE, NiO/GCE. MgNi2O3/GCE and 2.5 wt% of Peak current (Ipa) values for Fe-MgNi2O3/GCE, respectively, are 20.80, 22.11, 24.06, 23.76 and 27.51 |jl A. Compared to other current peaks, the 2.5 wt% Fe-MgNi2O3/GCE current peaks are between MgO/GCE and NiO/GCE.



a–Bare/GCE ; b–MgO/GCE ; c–NiO/GCE ; d–$MgNi_2O_3$/GCE ; e–2.5wt% Fe–$MgNi_2O_3$

Fig. 4 Cyclic voltammetry electrode oxidation-reduction curve



a–Bare/GCE,b–MgO/GCE,c–NiO/GCE,d–$MgNi_2O_3$/GCE,e–2.5wt% Fe–$MgNi_2O_3$

Figure 5 Electrochemical impedance spectrum

2.4.1 Oxidative selectivity and sensitivity

In order to study the sensitivity of the samples to nicotine, the authors placed the (1 ml of aqueous solution with Bare/GCE. MgNi2O3/GCE and 2.5 wt% FeMgNi2O3 nanoparticles),dropped on the surface of a polished glassy carbon electrode; after drying, the glassy carbon electrode was placed on the surface of a polished glassy carbon electrode.

The glassy carbon electrode surface; after drying, the glassy carbon electrode was placed on the 300 uM nicotine and 0.1 M PBS solution. The other 2 mixed solutions were used as references for square wave voltammetric analysis (SWV), as shown in Figure 6. The experimental results show that 2.5 wt% of the Fe-MgNi2O3is significantly higher than the response current of pure MgNi2O3o The same phenomenon was also observed in the other two mixed solutions, indicating that the FeMgNi2O3/GCE electrode has better selectivity and sensitivity to nicotine, and then there is a wide linear dynamic range and low measurement limits.

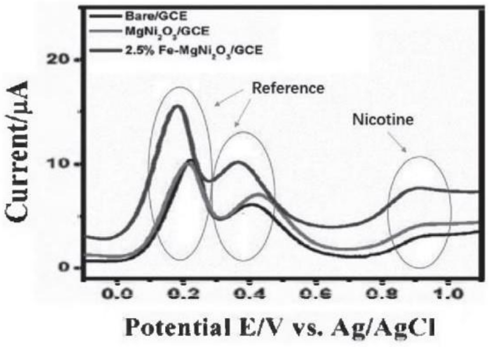


Fig. 6 SWV analysis of GCE, MgNi2O3/GCE and 2.5wt% Fe- MgNi2O3 vs. SWV analysis profiles for 300 uM nicotine

2.4.2 Analysis of anti-interference capability

Since a variety of other compounds such as folic acid, glucose, riboflavin, etc. may be present in compounds such as food, pharmaceuticals, fertilisers, etc. in addition to the possible presence of nicotine, it is necessary that the samples have a certain degree of anti-interference ability. Therefore, the author used the amperometric method to study the anti- interference ability of the samples.

interference ability; according to the above experimental method for detecting the oxidative selectivity of the Ascorbic acid, folic acid, glucose, hypromellose, sodium ion, potassium ion, lactose, epinephrine, levotryptophan, levulin and levulinic acid were added to a mixture of 0.1M PBS solution. Adenosine, L-tryptophan, Riboflavin and Urea as interfering compounds. A polished glassy carbon electrode of 2.5 wt% Fe-MgNi2O3 was used for the immunity test by amperometric method. amperometric method, the anti-interference ability test was carried out and the results are shown in Figure 7. The experimental results show that in the presence of the above interference compounds, the current of 2.5 wt% Fe-MgNi2O3 does not change significantly, indicating its strong anti-interference ability.

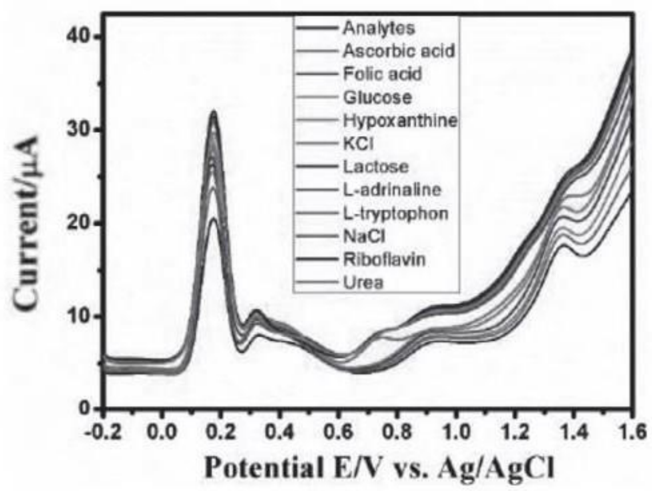


Fig. 7 Amperometric detection of 2.5 wt% Fe-MgNi2O3 in the presence of interfering substances detection pattern by amperometric method

2.4.3 Reproducibility and stability

In order to evaluate the reproducibility of 2.5 wt% Fe-MgNi2O3/GCE, the author performed 25 voltammetric (CV) determinations on 0.4 mM nicotine solution. The results showed that the relativestandard deviation (RSD) of the 25 CV data was 4.17 wt %, which was lower than the acceptable standard of 5% in the industry. In order to evaluate the stability of 2.5 wt% Fe-MgNi2O3/GCE, the author performed 25 cyclic voltammetry (CV) measurements on 0.4 mM nicotine solution, and the deactivation of the electrode was 1.2 wt%. These results indicate that the 2.5 wt% Fe- MgNi2O3/GCE electrode has good reproducibility and stability.

Part 3 Application

1. Background

In order to further evaluate the performance of the nicotine detection methods, the author developed a device under the Tradename "Nicotest", which is sponsored by Sourci Pty Ltd Australia, originally designed by Huayan Zhixin (Guangzhou) Technology Co., Ltd. (hereinafter referred to as Huayan Zhixin). This verification content includes accuracy, precision, detection limit, detection range, and stability of the nicotine detection device.

2. Evaluation Method

The nicotine detection device provided by Huayan Zhixin was used to test samples of high, medium, and low concentrations, with 12 standard samples in each group for evaluation. Three experimenters independently extracted each group of samples according to the method provided by the product, and tested them according to the method provided by the product. The standard deviation (SD) and detection mean were calculated. One experimenter tested the blank sample provided for product test 20 times to calculate the standard deviation (SD) and detection mean. Nicotine standard solutions of different concentrations were prepared using a nicotine standard solution, and the mean value was taken from 6 tests for each concentration. One experimenter tested the same standard sample according to the product provided, testing once every hour for 12 hours, and calculated the standard deviation (SD) and detection mean.

(1) Accuracy determination

Accuracy is determined based on the deviation of the detection value from the calibrated value of the standard sample, expressed as a recovery rate.

The calculation method for the recovery rate is as follows:

Mean detection value / sample calibrated value × 100%.

(2) Determination of precision

Precision is based on within-batch precision, which refers to the reproducibility of the same sample produced by the same batch of products on one sample, and is expressed in terms of relative standard deviation (RSD).

The calculation method for relative standard deviation (RSD) is as follows: RSD = standard deviation / mean value of detection × 100%

(3) Determination of the detection limit

The product does not require quantitative determination using the standard curve method.

Perform continuous independent determinations on 20 blank samples and calculate according to the following formula:

$Detection\ limit = \bar{x} + 3s$

$\bar{x} - Mean\ value\ of\ 20\ blank\ sample\ test\ results$;

$s - Standard\ deviation\ of\ 20\ blank\ sample\ test\ values.$

(4) Detection range test:

Use propylene glycol and glycerin to simulate the negative configuration of e-liquid. Use nicotine standard solution to make nicotine e-liquid standard solutions of different concentrations. Take the average of 6 tests for each concentration.

(5) Stability Test: One experimenter tests the same standard sample provided every hour for 12 hours. Calculate the standard deviation (SD) and the mean of the test results.

3. Content of Experiment

(1) Test samples of e-liquid (provided by Huayan Zhixin (Guangzhou) Technology Co., Ltd.,

and accompanied by a nicotine testing report provided by Ingeer Testing Technology Service Co., Ltd.).

The test values are for high, medium, and low concentrations. The low concentration sample is 10.67 mg/mL, medium concentration sample is 17.02 mg/mL, and high concentration sample is 38.95 mg/mL.

(2) Test product

Nicotine Detector (provided by Huayan Zhixin).

(3) Instruments and equipment

Nicotine detector (provided by Huayan Zhixin).
Nicotine rapid test kit
Including: screen-printed electrode, reagent A, reagent B (provided by Huayan Zhixin). Micropipette: single channel 20-200 μL, 100-1000 μL, 1-5 mL. Electronic balance with a sensitivity of 0.001g, centrifuge tubes, centrifuge, oscillator, micropipette tips, timer.

(4) Reagents

Water used for testing: Meets the requirements of GB/T6682 on first-grade lab used water. Nicotine standard product: Stanford Chemicals 100mg, 99.1%.
1,2-Propanediol: Aladdin ACS, ≥99.5%
Glycerol: Aladdin ≥99.5% (GC)

(5) Testing Method:

Follow the instructions of the nicotine rapid detection kit.

(6) Evaluation Results and Conclusions (6.1) Nicotine in e-liquid

(6.1.1) Evaluation result

	Evaluator 1		Evaluator 2		Evaluator 3	
Low concentration/ mg/kg	12.376	11.378	13.426	10.926	13.943	11.540
	12.334	10. 296	11.959	10.707	12.663	11.426
	12.404	11.155	11.876	11.491	11.773	11.427
	11.998	10. 294	11.966	11.146	12. 893	10.852
	12.122	10.611	11.497	9.879	12.273	9.875
	12.082	9.350	13.420	9.889	11.250	10.403
Medium concentration/ mg/kg	14.893	17.031	16.101	16.188	17.109	16.973
	17.221	17.994	16.813	15.853	15.742	16.265
	14.154	16.182	16.315	17.444	14.735	19.605
	15.277	15.361	16.171	15.421	16.786	16.720
	17.492	15.699	16.003	17.425	16.758	18.327
	16.602	16.741	16. 513	13.593	16.931	19.585
High concentration/ mg/kg	35.993	36.465	35.257	38.059	36.996	37.476
	34.867	35.884	35.721	32.997	36.337	34.714
	35.020	36.843	35.097	35.441	37.212	36.988
	35.263	34.836	35.203	34.407	37.021	35.696
	34.417	33.296	35.984	35.622	35.504	36.513
	36.489	34.401	33.267	35.721	32.088	32.344

(6.1.2) Accuracy and precision

Sample	Calibrated concentration	Mean	Recycling rate	Relative standard deviation
Low concentration	10.67	11.52	108%	9.3%
Medium concentration	17.02	16.50	96%	7.6%
High concentration	38.95	35.42	91%	3.9%

(6.1.2) Detection limit

	Test data				
Blank sample	0.568	0.683	0.676	0.769	0.725
	0.581	0.807	0.598	0.651	0.710
	0.51	0.525	0.896	0.681	0.717
	0.667	0.807	0.598	0.724	0.734
$\bar{x}$-0.681, s-0.098 Detect limit = 0.978					

(6.3) Test range testing:

Sample	Concentration (mg/mL)	Mean	Recovery rate
1	5	5.798	116.0%
2	10	11. 122	111.2%
3	20	20.082	100.4%
4	40	37.378	93.4%
5	80	71.296	89.1%
6	100	84.155	84.2%

(6.4) Stability evaluation

Sample	E-liquid 17.02mg/mL		
1	2	3	4
15.856	16.265	17.452	16. 232
5	6	7	8
17.492	16.714	16.786	14.765
9	10	11	12
17.425	16.602	15.699	16.171
Mean: 16.454, SD: 0.807			

(6.4) Evaluation Conclusion

(1) Accuracy: For the samples within the scope of this verification, the recovery range for the high, medium, and low concentrations is 90-110%;

(2) Precision: For the samples of high, medium, and low concentrations within the scope of thisverification, the relative standard deviation (RSD) is within 10%;

(3) Detection limit: The detection limit for the products within the scope of this verification is0.978mg/mL;

(4) Stability: The products within the scope of this verification have good stability;

(5) The nicotine detector uses screen-printed electrodes, which are easy to operate. The pre- treatment process of the samples does not require stringent conditions and is simple and controllable.

Part 4 Conclusion

The results of this study show that the nicotine detection device designed using this electrode has good sensitivity and can theoretically measure nicotine levels above 0.02 mg/ml with a very low measurement limit.

During the experiment, the anti-interference ability was tested by adding interfering compounds such as ascorbic acid, folic acid, glucose, hypromellose, sodium ion, potassium ion, lactose, adrenaline, levotryptophan, riboflavin, and urea, and the results showed that there was no significant change in the current, which demonstrated that the method has a strong ability to resist interferences.

Reproducibility experiments showed that the nicotine detection device with this electrode design has good reproducibility and stability.

Reference

1. Rakic Vesna, Damjanovic Ljiljana, Rac Vladislav, Stosic Dusan, Dondur Vera, Auroux Aline. The adsorption of nicotine from aqueous solutions on different zeolite structures[J]. Water research, 2010(44):2047-2057.
2. M. Chena, M. Alegrea, A. Durgavanshib, D. Bosec and J. Romero.
3. Rapid and sensitive determination of nicotine in formulations and biological fluid using micellar liquid chromatography with electrochemical detection[J]. Journal ofChromatography B, 2010(878): 2397-2404.
4. Eduardo Costa Figueiredo, Daniela Mayra de Oliveira, Maria Elisa Pereira Bastosde Siqueira, Marco Aur e lio Zezzi Arruda. On-line molecularly imprinted solid-phase extraction for the selective spectrophotometric determination of nicotine in the urine of smokers[J]. Analytica Chimica Acta, 2009(635):102- 107.
5. Fatemeh Kardani, Ali Daneshfar, Reza, Sahrai. Determination of nicotine, anabasine, and cotinine in urine and saliva samples using single-drop microextraction[J]. Chromatography B, 2010 (878):2857-2862.
6. Tomasz Wielkoszy n ski, Krystyna Tyrpie n , Magdalena Szumska. The enzyme-linked immunosorbent assay (ELISA) method for nicotine metabolites determination in biological
7. fluids].Journal of Pharmaceutical and Biomedical analysis, 2009(49):1256-1260.