

Full Paper

Preparation of Cephalexin Monohydrate Sensors Based on The Synthesis of CeO₂ NPs for Potentiometric Determination of Cephalexin Monohydrate in Pure and Commercial Products

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Received: 8 July 2024 / Received in revised form: 5 October 2024 /

Accepted: 25 October 2024 / Published online: 31 October 2024

Abstract- Cephalexin monohydrate can be detected using potentiometric sensors made of coated wire electrodes. The Cephalexin monohydrate sensor was made of cerium oxide nanoparticles that were made using green synthesis techniques. An excellent response with -54.76 and -54.78 mV.decade⁻¹ were observed. The detection limits were between 4.2×10^{-10} and 4.0×10^{-10} mol.L⁻¹ for the CEX-PT-CeO₂ NPs with leaves extract of *Myrtus communis* and CEX-PT-CeO₂ NPs with leaves extract of *Mentha* coated wire electrodes. The lifetime was 63 and 77 days for the CEX-PT-CeO₂ NPs with leaves extract of *Myrtus communis* and CEX-PT-CeO₂ NPs with leaves extract of *Mentha* coated wire electrodes. The sensor CEX-PT-CeO₂ NPs was gave a non-Nernstian response equal to -35.96 mV.decade⁻¹ with detection limit 9.8×10^{-10} mol.L⁻¹. A new coated wire electrode was developed in this study. The limit of detection, selectivity, and concentration range of the two sensors were all very high. These sensors were used to find Cephalexin monohydrate in pharmaceutical and pure products.

Keywords- Green synthesis; Potentiometric method; Detection limit; XRD analysis; Coated wire electrode

1. INTRODUCTION

Ion-selective electrodes are an inexpensive, simple, and sensitive analytical method that requires little sample pretreatment. [1]. Coated wire electrodes are one type of ion-selective electrode. [2]. This electrode is made of Pt, Cu, Ag, graphite, and other solids, as long as the membrane's matrix does not react with the internal wire, it can also be used. Coated wire ions are advantageous because they can be used with a small sample volume, are simple to design (there is no internal solution), are mechanically flexible, and can be miniaturized and microfabricated. For the purpose of determining CEX in pharmaceutical and pure products, this work describes the design and investigation of the performance characteristics of a novel coated wire electrode based on CeO₂ NPs. New methods for preparing materials and nanoparticles are necessary for a new development in nanotechnology. For the purpose of determining CEX in pure and pharmaceutical products, this work describes the construction and investigation of a novel coated wire electrode based on CeO₂ NPs. Another development in the innovation of nano relies upon the planning of materials/NPs by means of new methods. NPs can be made-up through substance, organic, and actual strategies as displayed in Figure 1. The nanoparticles can be arranged utilizing actual strategies including lithography, actual fume statement (PVD), ball handling, pyrolysis, squashing, processing, and crushing, as well as substance fume testimony (CVD). Chemosynthesis of nanoparticles incorporates combination by means of sol-gel, electrolysis, photocatalytic decrease in addition to the union through compound decrease microwave-helped strategy [3].

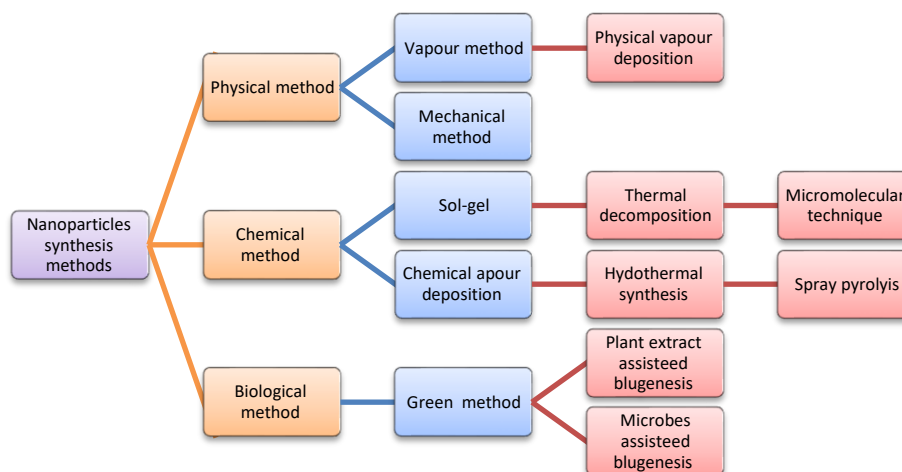


Figure 1. Different methods for the synthesis of Nanoparticles [4]

Cephalexin monohydrate (CEX), Figure 2, is (6R,7R)-7-(D- α -amino- α -phenylacetamido)-3-methyl-3-cephem-4-carboxylic acid hydrate or (6R,7R)-7-[(2R)-2-amino-2-phenylacetyl]amino]-3-methyl-8-oxo-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylic acid hydrate.

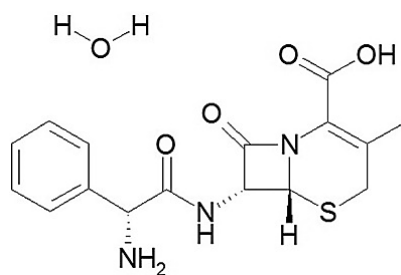


Figure 2. The Chemical Structure of Cephalexin monohydrate [5]

Chemically, it is a crystalline powder that is white or almost white and has a molecular weight of 365.4 g/mol. It dissolves in water but is particularly insoluble in ether and alcohol. [6,8]. Cephalexin (CEX) is a semi-synthetic derivative of cephalosporin of the first generation [9], which is a potent weapon against both gram-positive and gram-negative bacteria. It is capable of weakening the bond with blood protein through a wide range of activities; Consequently, it lacks metabolites. It has a low toxicity and rapid absorption rate. [10]. It is an amphoteric, hydrosoluble compound with an isoelectric point of 4.3 [11]. High-performance liquid chromatography (HPLC) [11-15], the spectrophotometer method [16-19], and the electrochemical method [20] are just a few of the many analytical techniques utilized in the process of determining Cephalexin monohydrate.

2. EXPERIMENTAL SECTION

2.1. Chemicals

Cephalexin monohydrate (CEX) was made by consolidating Cephalexin monohydrate (SDI) with Cephalexin monohydrate (250 mg/container) from Ajanta Pharam Restricted. Fluka gave cerium nitrate $\text{Ce}(\text{NO}_3)_3$, sodium hydroxide (NaOH), $(\text{CH}_3)_2\text{CO}$ 99.9 percent, methanol 99.9 percent, ethanol 99.9 percent, tetrahydrofuran, 99.9 percent Di-n-Butyl Phthalate, and molybdophosphoric corrosive, notwithstanding other logical synthetics and solvents. Logical-grade synthetics were all present.

2.2. Apparatus

UV-visible spectrophotometer was UV-1650 PC Shimadzu model, Japan. The infrared spectrophotometer was SHIMADZU, FTIR-8000 (Japan). XRD Phillips xpert PA analytical technique, Holland was used for XRD analysis. Field Emission Scanning Electron Microscopy (FE-SEM), and transmission electron microscopes were done by French MIRA3. HANNA instruments pH 2110UK, Romania, and Reference electrode, a saturated calomel electrode (RE-2BH) were also used.

2.3. Preparation of CEX-PT Electro-active Material [21]

Mixing (1.0×10^{-2} mol.L⁻¹) resulted in the production of ion-pair (CEX-PT) of both molybdophosphoric acid (PT) and the drug (CEX) at the same concentration. The precipitate was then filtered through Whatman filter paper No. 41 after being washed with deionized water. It was then dried for 24 hours.

2.4. Chemical Synthesis of CeO₂NPs [22]

CeO₂NPs were orchestrated by the arrangement of 50 mL of 0.5 mol.L⁻¹ of cerium nitrate hexahydrate in Milli Q water as a forerunner arrangement. With consistent mixing and at surrounding temperature, 2.0 mol. L⁻¹ of NaOH was dribbled gradually. The expansion was performed inside 30 min. The blend was centrifuged at 2500 rpm for 10 min. The acceleration was gathered utilizing Whatman channel paper No. 1, then washed completely with Milli Q water. The framed CeO₂ NPs were dried for 6 h at 100 °C. To vanish the water, the shaped nanoparticles were calcinated in a heater broiler at 600 °C for 4 h (Figure 3).

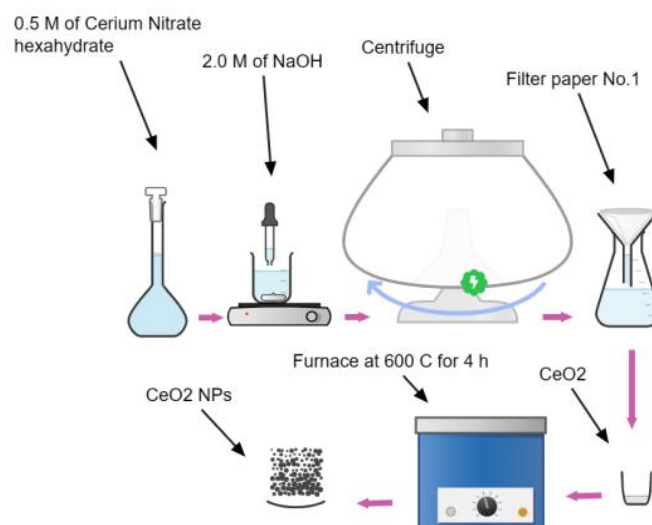


Figure 3. Chemical synthesis of CeO₂ NPs

2.5. Green synthesis of Cerium oxide nanoparticles [23]

To make CeO₂ NPs, 5.0 g of cerium nitrate was added to 70 milliliters of an aqueous solution that also contained 60 milliliters of distilled water and 10 milliliters of prepared leaf extract from *Myrtus communis* or *Mentha*. The magnetic stirrer was used to continuously stir this solution for 6 hours at 80 °C, and the water was evaporated in an oven at 60 °C to produce the gel. The obtained gel was then calcined for two hours in a furnace at 400 °C to eliminate natural mixtures and produce yellow CeO₂ NPs powders (Figure 4).

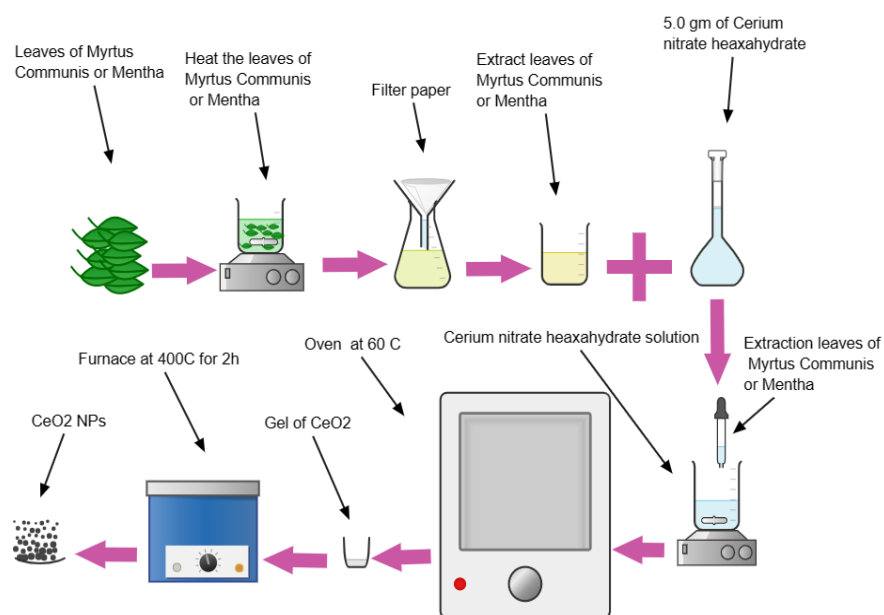


Figure 4. Green synthesis of CeO₂NPs

2.6. Preparation of Standard CFX Solution

By weighting 0.3654 g of the drug, CEX, a standard solution at a concentration of (0.1 mol L⁻¹) was made. After that, deionized water was used to dilute the solution, resulting in a range of CEX concentrations ranging from 1.0×10^{-10} to 1.0×10^{-2} mol.L⁻¹.

2.7. Preparation of CEX coated wire electrodes

It was prepared by stirring 7 mL of THF with 0.5 g of CeO₂ nanoparticles, 0.1 g of CEX-PT complex, 0.19 g of matrix (PVC), and 0.35 mL of n-di butyl phthalate to make a polymeric solution. The surface of a Cu-coated wire electrode based on CEX-PT-CeO₂ NPs was then constructed using it. In order to use the resulting membrane as a mixture, a thin layer was formed on the electrode's surface.

3. RESULTS AND DISCUSSION

3.1. The analysis of UV-Vis for the Cerium Nanoparticles

There were three significant absorption peaks at 320.0, 344.0, and 301.2 nm for CeO₂ NPs alone, CeO₂NPs containing an extract of the leaves of *Myrtus communis*, and CeO₂ NPs containing an extract of the leaves of *Mentha*, respectively as shown in Figure 5. It has better optical properties, and the peaks at 320, 301.2, and 344 nm indicates that the charge transition takes place among the O₂ p and Ce 4f states in O²⁻ and Ce⁴⁺. These wavelengths correspond to the quantum size effect of the blue shift. This is similar to the optical properties of cerium

nanoparticles that have been previously described. The absorbance maximums that have been observed before were 321 nm [24] and 335 nm to 352 nm [25].

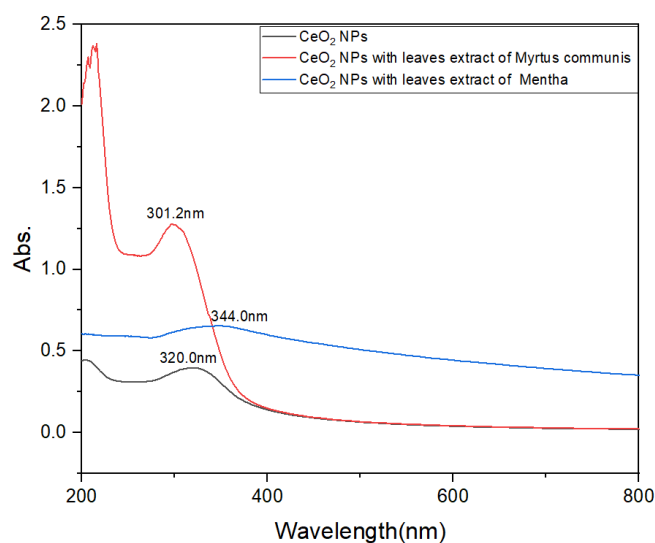


Figure 5. UV-visible spectrum of cerium oxide nanoparticles

3.2. The analysis of Fourier transform infrared (FTIR) for cerium oxide nanoparticles

FTIR for nanoparticles estimated the fluid stage, displayed in Figure 6. Cerium oxide nanoparticles were ready by three techniques, every one of them showed the extending band in round (3285.54 - 3276.69) cm^{-1} and a bowing band at (1338.50-1637.11) cm^{-1} showed up as hydroxy gathering groups. The top at (488.86,452.47 and 459.55) cm^{-1} showed the arrangement of the Chief nanoparticles by synthetic strategy and with leaves concentrates of *Myrtus communis* and *Mentha*.

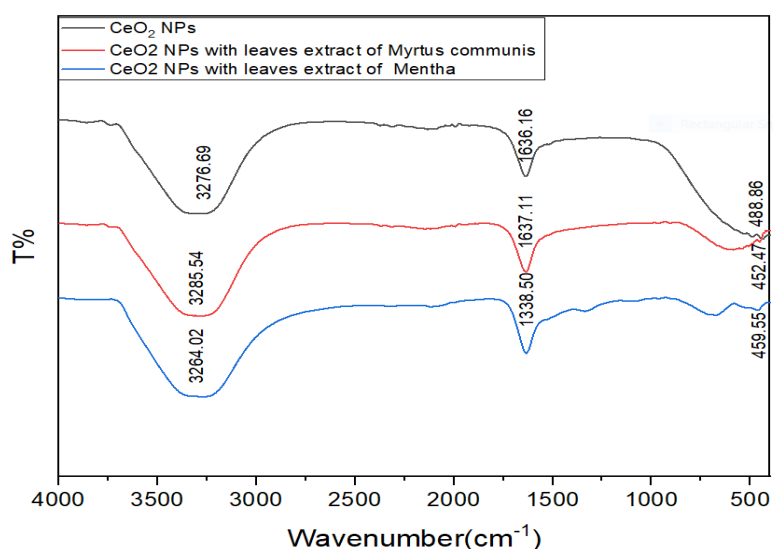


Figure 6. FTIR spectrum of CeO₂ nanoparticles

3.3. The Analysis of X-ray for cerium oxide nanoparticles

As depicted in Figure 7, the standard values for the face-centered cubic structure of cerium oxide were well matched by all of the diffraction peaks. The results were consistent with JCPDS Nos. 03-065-2975, 00-001-0800, and 01-081-0792 for CeO₂ NPs, CeO₂ NPs containing *Myrtus communis* leaves extract, and CeO₂ NPs containing *Mentha* leaves extract, respectively. Cerium oxide nanoparticles with *Myrtus communis* leaves extract, *Mentha* leaves extract, and CeO₂ NPs with *Mentha* leaves extract, respectively, had average grain sizes of approximately 15.25, 3.37, and 5.03 nm.

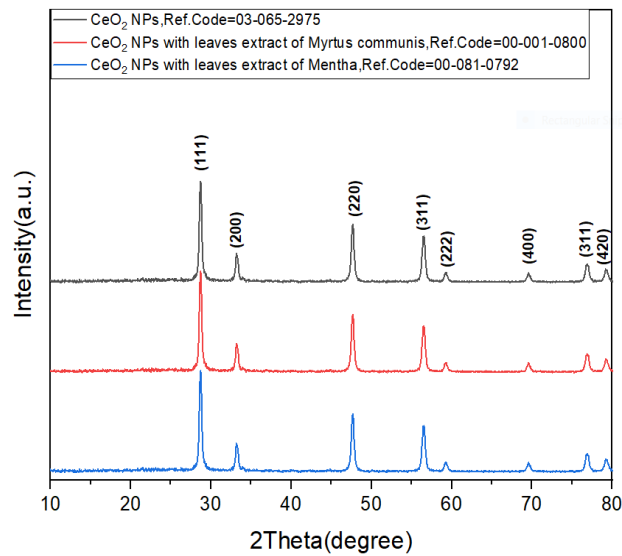


Figure 7. XRD pattern of CeO₂ nanoparticles

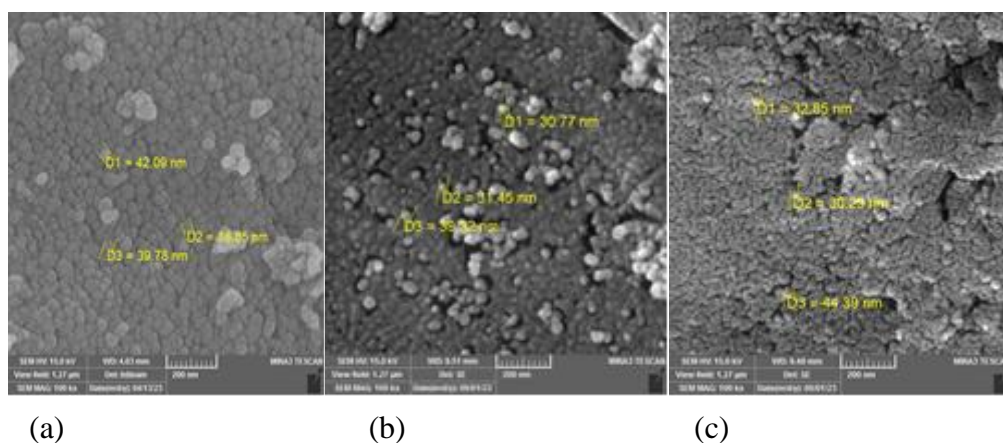


Figure 8. SEM pictures of Cerium oxide NPs: (a) for CeO₂ NPs, (b) for CeO₂ NPs with leaves concentrate of *Myrtus communis*, and (c) for CeO₂ NPs with leaves concentrate of *Mentha*

3.4. The analysis of Scanning and transmission electron microscopy for cerium oxide nanoparticles

Figure 8 depicts the SEM image of CeO₂ nanoparticles for CeO₂ NPs, CeO₂ NPs containing *Myrtus communis* leaves extract, and CeO₂ NPs containing *Mentha* leaves extract, respectively. The CeO₂ nanoparticles that were produced had a spherical shape and were produced almost uniformly. It was determined that the synthesized CeO₂ nanoparticles had an average diameter of 38.57, 33.84, and 35.83 nm, respectively. The sample was completely made up of spherical, smooth, and much less agglomerated particles, as the TEM images in Figure 9 demonstrated.

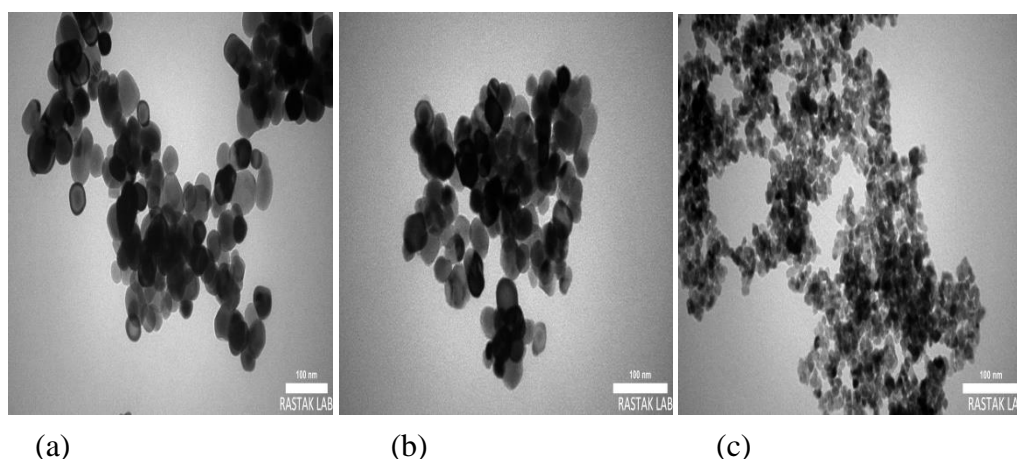


Figure 9. TEM pictures of CeO₂ NPs: (a) for CeO₂ NPs, (b) for CeO₂ NPs with leaves concentrate of *Myrtus communis* and (c) for CeO₂ NPs with leaves concentrate of *Mentha*

3.5. The Nature and response characteristics of the electrodes

The original CEX-PT-CeO₂ NPs covered wire terminals had Nernstian reactions of -54.76 and -54.78 mV decade⁻¹ at 25 °C, with lower cutoff points of discovery of 4.2×10^{-10} and 4.0×10^{-10} mol L⁻¹, individually, for CXE-PT-CeO₂ NPs covered wire electrodes containing leaves concentrate of *Mentha* and CXE-PT-CeO₂ NPs covered wire electrodes containing leaves CXE-PT-CeO₂ NPs with *Myrtus communis* leaves extract and CXE-PT-CeO₂ NPs with *Mentha* coated wire electrodes had life spans of 63 and 77 days, respectively, for the manufactured sensors. A non-Nernstian response of -35.96 mV decade⁻¹ was observed for the CXE-PT-CeO₂ NPs wire-coated electrode at concentrations ranging from 8.6×10^{-10} to 1.0×10^{-10} mol. L⁻¹ had a life span of close to 23 days and a limit of detection of 9.8×10^{-10} . Cerium oxide nanoparticles have attracted a lot of attention for the development of high-sensitive biosensors due to their catalytic activity, electrochemical and fluorescent quencher properties, large surface area, and oxygen transfer capability. CNPs can be used as a whole sensor, a part of a recognition or transducer, or both. Including these nanoparticles in the design of sensors could significantly increase the sensitivity of detection [26]. The attributes of CXE-PT-CeO₂

NPs, CXE-PT-CeO₂ NPs covered with *Mentha* leaves extricate, and CXE-PT-CeO₂ NPs covered with *Myrtus communis* leaves are displayed in Table 1.

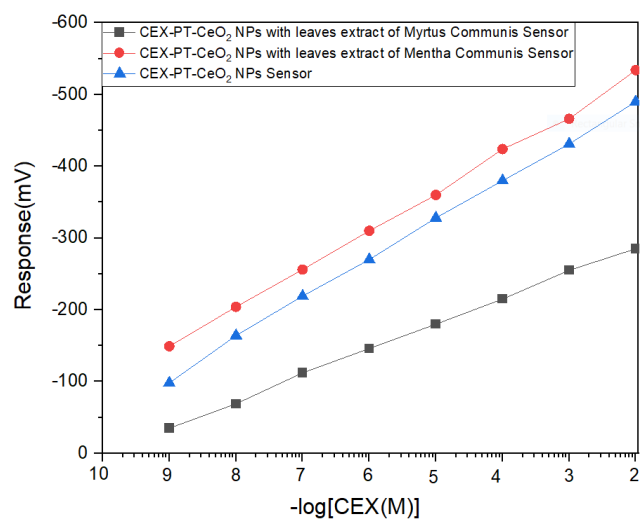


Figure 10. Calibration Curve of CEX-PT-CeO₂ NPs, CEX-PT-CeO₂ NPs with leaves concentrate of *Myrtus communis*, and CEX-PT-CeO₂ NPs with leaves concentrate of *Mentha* coated wire electrodes

Table 1. Response of CEX-PT-CeO₂ NPs electrodes for Cephalexin monohydrate solution

Parameters	CEX-PT-CeO ₂ NPs coated wire electrode	CEX-PT-CeO ₂ NPs with leaves extract of <i>Myrtus communis</i> coated wire electrode	CEX-PT-CeO ₂ NPs with leaves extract of <i>Mentha</i> coated wire electrode
Slope (mV/decade)	-35.96	-54.76	-54.78
Intercept	358.27	638.58	599.2
Regression equations	Y=(-35.96) log[CEX] -358.27	Y=(-54.76) log[CEX] -638.58	Y=(-54.78) log[CEX] -599.2
Correlation coefficient, r	0.9994	0.9992	0.9995
Range of Concentration (mol.L ⁻¹)	8.6×10 ⁻¹⁰ -1.0×10 ⁻²	3.0×10 ⁻⁹ -1.0×10 ⁻²	1.0×10 ⁻⁹ -1.0×10 ⁻²
LOD (mol.L ⁻¹)	9.8×10 ⁻¹⁰	4.2×10 ⁻¹⁰	4.0×10 ⁻¹⁰
Range of pH	3.0-4.0	3.5-6.5	3.0-7.5
Lifetime (day)	23	63	77

3.6. The Influence of pH

The capability of the cephalexin monohydrate (CEX) arrangement was estimated at 1.0×10⁻³ M from the pH worth of 0.5 to 14.0 by involving arrangements of HCl or NaOH as

concentrated to conform to the pH. The outcomes showed that the worth of the likely stayed steady notwithstanding the pH varieties of 3.0-4.0, 3.5-6.5, and 3.0-7.5 for CEX-PT-CeO₂ NPs for sensors: As portrayed in Figure 11, CEX-PT-CeO₂ NPs, CEX-PT-CeO₂ NPs covered with *Mentha* leaves extricate, and CEX-PT-CeO₂ NPs covered with *Myrtus communis* leaves extract.

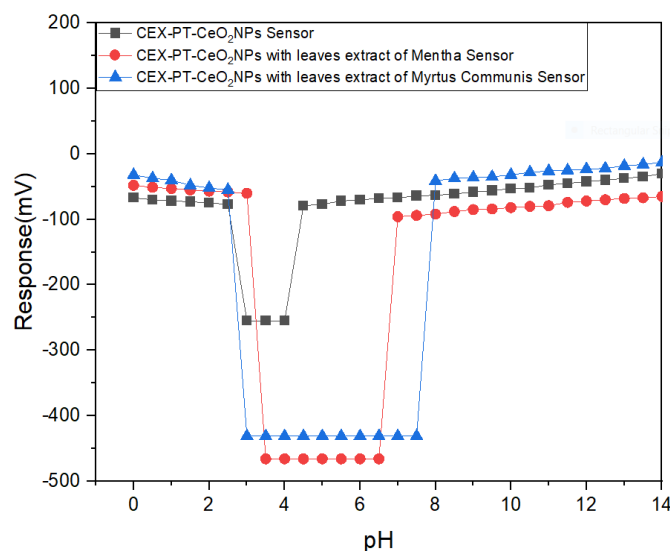


Figure 11. pH values of CEX-PT-CeO₂ NPs, CEX-PT-CeO₂ NPs with leaves extract of *Myrtus communis* and CEX-PT-CeO₂ NPs with leaves extract of *Mentha* coated wire electrode

3.7. Selectivity of Sensors

A different arrangement strategy was utilized to test the selectivity coefficients of the proposed CEX sensors toward different kinds of excipients, normal cations, anions, and a few pharmacologically related composites [27,28].

Table 2. Selectivity coefficient values for CEX-PT-CeO₂ NPs coated wire electrode

Ion interference	CEX-PT-CeO ₂ NPs with leaves extract of <i>Myrtus Communis</i> coated wire electrode	CEX-PT-CeO ₂ NPs NPs with leaves extract of <i>Mentha</i> coated wire electrode
K ⁺	0.6298×10 ⁻¹	0.2716×10 ⁻¹
Na ⁺	0.7771×10 ⁻¹	0.3495×10 ⁻¹
CO ₃ ⁻	0.9588×10 ⁻¹	0.3609×10 ⁻¹
NO ₃ ⁻	0.8452×10 ⁻¹	0.1766×10 ⁻¹
Mg ²⁺	4.0164×10 ⁻⁴	1.9023×10 ⁻⁴
Zn ²⁺	5.6885×10 ⁻⁴	2.2508×10 ⁻⁴
Al ³⁺	5.5804×10 ⁻⁶	6.0529×10 ⁻⁶
Fe ³⁺	9.6371×10 ⁻⁶	4.3240×10 ⁻⁶
Glucose	1.3269×10 ⁻⁶	6.7998×10 ⁻⁶
Starch	1.5698×10 ⁻⁶	4.1056×10 ⁻⁹

Within the sight of normal cations, anions, and a few pharmacologically related composites, the CEX test arrangement was found to affect the assurance of CEX-PT-CeO₂ NPs, CEX-PT-CeO₂ NPs containing leaves concentrate of *Myrtus communis*, and CEX-PT-CeO₂ NPs containing leaves concentrate of *Mentha*, as displayed in Table 2. Matching the areas of lipophilic destinations in the two contending species on the washing arrangement side and those in the receptor of the particle pair is important for the essential system of selectivity.

3.8. Quantification of Cephalexin monohydrate

The proposed electrodes' direct potentiometric determination of CEX in its pure form yielded mean recoveries of (97.00±1.20) for the calibration method and (98.29±1.38) for the standard addition method, as shown in Table 3. The antilog (E/S) of CEX-PT-CeO₂ NPs containing a *Myrtus communis* leaves extract and a *Mentha*-coated wire electrode at 1.0×10⁻³ mol.L⁻¹. Cephalexin monohydrate is depicted in Figures 12 and 13.

Table 3. The statistical results for CEX-PT-CeO₂ NPs coated wire electrode, CEX-PT-CeO₂ NPs coated wire electrode containing an extract of *Myrtus communis* leaves, and CEX-PT-CeO₂ NPs coated wire electrode containing an extract of *Mentha* leaves

Type of wire-coated electrodes	Concentration(M)				
	Sample	Response by potentiometric method			
		Direct	SAM	SAMS	Titration
CEX-PT-CeO ₂ NPs with leaves extract of <i>Myrtus communis</i>	1×10 ⁻³	0.9700×10 ⁻³	0.9829×10 ⁻³	0.9823×10 ⁻³	0.9793×10 ⁻³
	RSD %	1.20	1.38	-	-
	Rec %	97.00	98.29	98.23	97.93
	RE%	-3.00	-3.71	-1.77	-2.07
CEX-PT-CeO ₂ NPs with leaves extract of <i>Mentha</i>	1×10 ⁻³	0.9966×10 ⁻³	0.9847×10 ⁻³	0.9801×10 ⁻³	0.9829×10 ⁻³
	RSD %	0.70	0.68	-	-
	Rec %	99.66	98.47	98.01	98.29
	RE%	-0.34	-1.53	-1.99	-1.71

*n=5

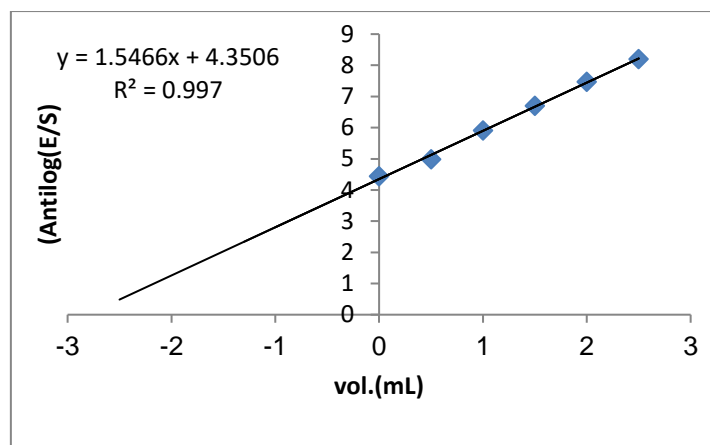


Figure 12. Antilog (E/S) of CEX-PT-CeO₂ NPs with leaves extract of *Myrtus communis* coated wire electrode at 1.0×10^{-3} mol.L⁻¹ of Cephalexin monohydrate

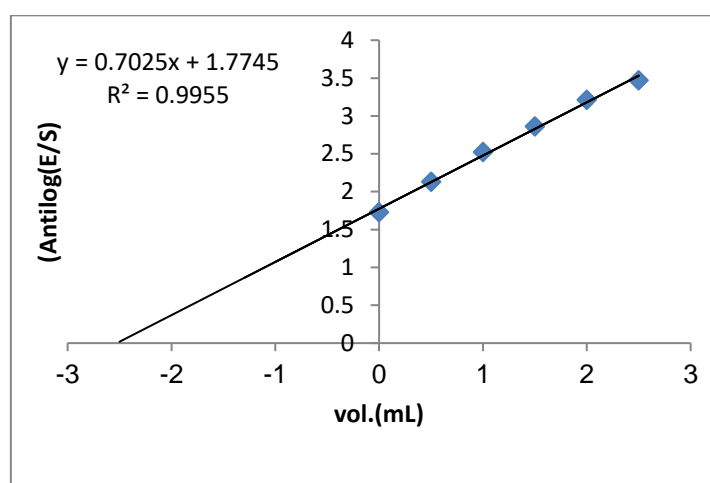


Figure 13. Antilog (E/S) of CEX-PT-CeO₂ NPs with leaves extract of *Mentha* coated wire electrode at 1.0×10^{-3} mol.L⁻¹ of Cephalexin monohydrate

3.8. The Analytical uses of CEX in pharmaceutical capsules

A direct method at 1.0×10^{-3} mol.L⁻¹ was utilized to decide CEX in drug items. Of CEX-containing NPs made of CEX-PT-CeO₂, NPs made of CEX-PT-CeO₂ containing *Myrtus communis* leaves concentrate, and NPs made of CEX-PT-CeO₂ containing *Mentha* leaves extricate-covered wire terminals. For the two sorts of covered wire sensors, the typical recuperation for CEX in cases was around 97.16 percent. CEX-PT-CeO₂ NPs covered with *Mentha*-covered wire electrodes and CEX-PT-CeO₂ NPs implanted with *Myrtus communis* leaves removed had RSD rates of 1.34 and 0.69, individually. Table 4. shows the aftereffects of the immediate technique for deciding (CFX) utilizing CEX-PT-CeO₂ NPs covered with *Mentha* leaves removed and CEX-PT-CeO₂ NPs covered with *Myrtus communis* leaves separated.

Table 4. Aftereffects of direct strategy for CEX-PT-CeO₂ NPs, CEX-PT-CeO₂ NPs with leaves concentrate of *Myrtus communis* and CEX-PT-CeO₂ NPs with leaves concentrate of *Mentha* covered wire electrodes for assurance of (CEX)

Type of coated wire electrodes	Conc. Of CEX(prepared) (Capsules/mg)	Conc.of CEX(found) (Capsules/mg)	RSD%	Rec%
CEX-PT-CeO ₂ with leaves extract of <i>Myrtus communis</i>	1.0×10 ⁻³	0.9666×10 ⁻³	1.34	96.66
CEX-PT-CeO ₂ with leaves extract of <i>Mentha</i>	1.0×10 ⁻³	0.9833×10 ⁻³	0.69	98.33

4. CONCLUSION

To decide CEX in its unadulterated structure as well as in drug definitions, the proposed potentiometric techniques in view of the development of different kinds of particular anodes with CeO₂ NPs might give valuable logical attributes. The proposed strategy's high exactness and accuracy should be visible in its high recuperations and low relative standard deviations. Also, the technique's minimal expense, straightforwardness, and convenience make it a superb apparatus for routine CEX assurance in quality control research centers.

Acknowledgments

This project was upheld by the Department of Chemistry, College of Science, Tikrit University, and Department of Chemistry, College of Science, Al-Nahrain University.

Declarations of interest

The authors declare no conflict of interest in this reported work.

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