Anal. Bioanal. Electrochem., Vol. 14, No. 8, 2022, 806-817

Analytical &
Bioanalytical
Electrochemistry

2022 by CEE
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Full Paper

Modified potentiometric Sensor for Yb(III) based on Cefixime and Quaternary-doped Graphene

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Received: 17 May 2022 / Received in revised form: 27 July 2022 /
Accepted: 29 July 2022 / Published online: 31 August 2022

Abstract- A carbon-based electrode was modified through incorporating N, S, P and B quaternary-doped graphene (NSPBG) in its composition, and given that former studies indicated a selective interaction between cefixime (CEF) and Yb^{3+} ions in comparison to other lanthanide ions. Therefore, CEF was further incorporated in the CPE composed of a mixed matrix of NSPBG and graphite powder to develop an Yb^{3+} selective potentiometric carbon paste electrode, and evaluations of the modified CPE reflected that high sensitivity, selectivity; short response time and stability, as well as improved lifetime as opposed to CPEs based on the multiwall carbon nanotube (MWCNTs). The optimized electrode with a composition of 7% NSPBG, 13% CEF, 30% IL and 50 % graphite powder, had a Nernstian response of 19.7±0.1 mV per decade over the concentration range of 1.0×10^{-7} to 1.0×10^{-2} M.

Keywords- Sensor; Ion-selective electrode; Carbon paste; N, S, P and B doped graphene; Room temperature ionic liquid; Ytterbium ion

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1. INTRODUCTION

Having an atomic number of 70, ytterbium was discovered by Jean de Marignac in 1878 and got its name from a village in Sweden. The white silvery, soft, ductile element reacts with strong acids quickly and gradually with cold water, and is oxidized by air forming a golden or brown oxide. Ytterbium is present in 3 major minerals namely monazite, euxenite, and xenotime. It forms di- and tri-halides, yet in the majority of its it has a +3-oxidation state. The element and its compound are used to enhance strength, grain refinement and mechanical properties of stainless steel, as well as in various industrial catalysts. Ytterbium is also used in tunable lasers and digital memory devices.

Some general methods for low-level detections of Yb(III) ions in solutions are spectrophotometry, inductively coupled plasma mass spectrometry (ICP-MS) and ICP-AES [1-3], isotope dilution mass spectrometry, neutron activation analysis, X-ray fluorescence spectrometry [4-6], etc. These methods are normally laborious, involving multiple sample operations, or too luxurious for many analytical laboratories. Thus, the development of a simple and direct method for the assay of ytterbium ions in different samples can be a necessity. Sensors based on potentiometric detection offer several advantages such as simple instrumentation, fast response, wide dynamic range, reasonable selectivity and low price.

Ionic liquids (ILs), which are salts that are liquid at low temperatures (usually below 100 °C), can have various applications, given that they enjoy remarkable properties which make them suitable for use as solvents in different areas of like electrochemistry. The commonest ILs are composed of cations like imidazolium, pyridinium, pyrrolidinium (i.e. asymmetrically substituted nitrogen-containing cations) and bulky organic or inorganic anions such as Cl^-, PF_6^-, BF_4^-. Among all ILs those containing imidazolium and pyridinium cations have been reported to offer the maximum electrical conductivity (~1 and 10^{-1} S/m, respectively) [7]. Further properties of these ILS include excellent solvating behaviors, considerable boiling points, low toxicity, and excellent stability against electrochemical [8,9].

A major application of ILs is as binders in the composition of carbon paste electrodes (CPEs) [7-11], that are sensing devices classically based on a paste of graphite powder and nonconductive mineral oils. Mineral oils suffer various disadvantages, especially since they have varying compositions, given that they are commonly obtained from different petroleum-refining products, which makes them susceptible to random effects on the analytical results.

Ion-selective electrodes (ISEs), on the other hand, are a well-known family of analytical instruments used for various applications [12-23] given their portability, ease and high speed of use, moderate costs, good sensitivity reliability over rather wide concentration ranges. Modified CPEs are among the various sub-categories of ISEs, and enjoy various advantages over many of their counterparts such as PVC-based membrane ISEs. Examples of such advantages include easily renewable electrode surfaces, robust response behaviors, and low Ohmic resistance [7-11]. The modification of CPEs is usually performed through incorporating selective species, in the composition of the electrode which is typically made up of a paste of graphite powder, and a mineral oil. In the light of the above, and given the results of former selectivity studies [12], reflecting the selective interactions of cefixime and Yb^{3+}, this study was focused on developing a CEF-based potentiometric CPE for Yb^{3+} further including an RTIL and N, S, P and B doped graphene in its composition.

2. EXPERIMENTAL PART

2.1. Reagents and materials

All general chemicals were analytical grades and used without any further purification. Ionic liquid, 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4) and other chemicals were obtained from Merck Co. Distilled deionized water was used for solution preparations and dilutions.

2.2. Synthesis of quaternary-doped graphene sample

To prepare the N, S, P and B quaternary-doped graphene sample, 1 g of dried and ground homogeneous grape leaf powder was carbonized at 300° and mixed with graphene (2:1). The powder was ground using ball-mill for 1 h and pyrolyzed under flowing N_2 atmosphere at 900°C for 2h to prepare a quaternary-doped graphene [24-29].

2.3. Apparatus

The potentiometric cell used for the application of the modified CPE further included an Ag/AgCl reference electrode (Azar electrode, Iran) forming the cell assembly below:

CPE | Sample Yb^{3+} ion solution | Ag/AgCl–KCl (satd.)

2.4. Preparing the Electrodes

The CPEs were prepared through admixing desired quantities of CEF, graphite powder, 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4), and NSPBG to form a paste, which was next packed into a 3cm long glass tubes (5 mm i.d.). The packing was performed carefully to avoid the trapping of air gaps. Then a Cu wire was inserted into the paste from the empty side of the tube and the outer surface of the resulting CPE was cleaned using soft abrasive paper. The developed CPE was finally put in a 1.0×10^{-3} mol L^{-1} $YbCl_3$ solution for 48 hours for conditioning, prior to use.

3. RESULTS AND DISCUSSION

3.1. SEM, XPS and Raman results

The surface morphology analysis of the quaternary-doped graphene sample using FESEM (Figure 1) showed similar structure with graphene according to our other works [24-26].

Furthermore, the elemental mapping distribution analysis indicated the uniform and homogenous dispersion of N, S, P and B elements in the carbon structure of prepared sample, confirming the successful doping of heteroatoms into the graphene lattice [24-29].

In addition, X-ray photoelectron spectroscopy (XPS) was used to investigate the chemical nature of the prepared sample. The high resolution XPS N 1s peak (Figure 2) was deconvoluted into three peaks at around 398.9, 399.8 and 402.5 eV, attributed to pyridinic N, C-N-B and oxidized N bonds, respectively. Moreover, the deconvolution analysis of high-resolution spectrum S 2p peak (Figure 2) shows the peak around 163.2, 163.8, 165.0 and 168.8 which correspond to S-H, C-S_n-C, C=S, sulfoxide and other oxidized forms in the graphene lattice. The deconvolution of high resolution XSP of P 2p (Figure 2c) shows two peaks 133.6 and 132.6 eV, which are related to P-O and P-C, respectively. The deconvolution of high resolution XSP of B 1s (Figure 2d) shows three peaks 189.2, 191.1 and 193 eV, attributed to B-C-N, B-C and B-O bonds. The XPS results show that nitrogen, sulfur, phosphorus and boron successfully is incorporated into the graphene structure [24-29].

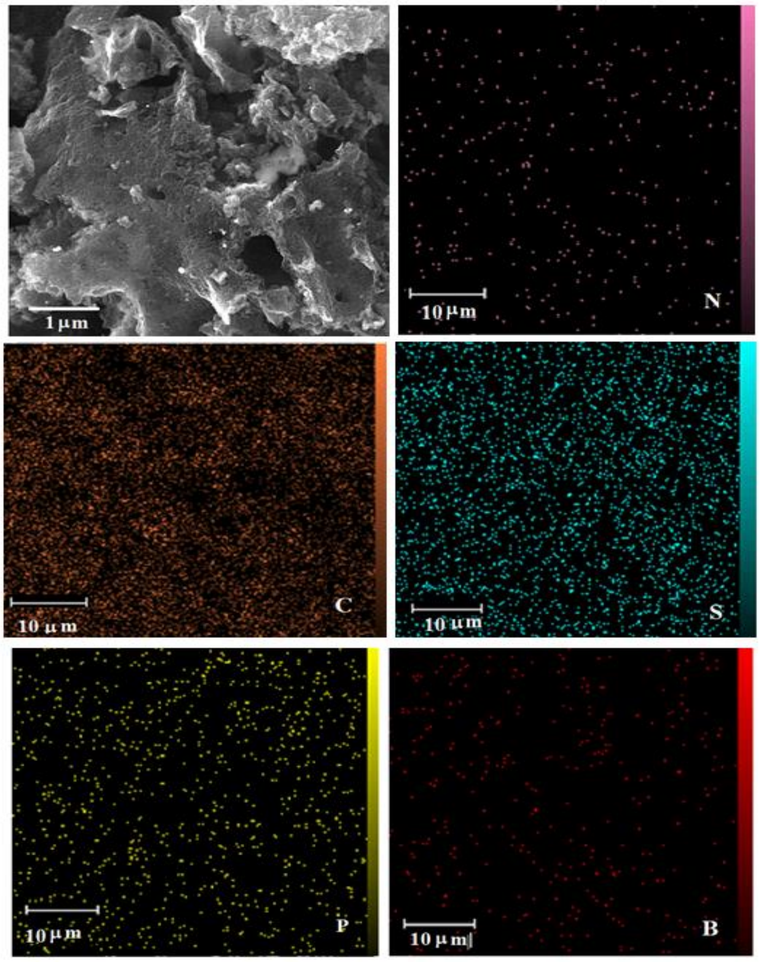

Figure 1. FESEM and EDX mapping analysis of C, N, S, P and B element of prepared doped graphene

Moreover, defects and doping level in the graphene structure were investigated using Raman spectroscopy. The I_D/I_G ratio shows carbon structure disordering that the higher value indicates more defects in the graphene structure and heteroatom doping. The I_D/I_G value of heteroatom-doped sample in the Figure 3 (1.17) was higher than that of for graphene (0.96), indicating more disordered structures and defects confirming doping of heteroatom in the graphene structure [24,28,29].

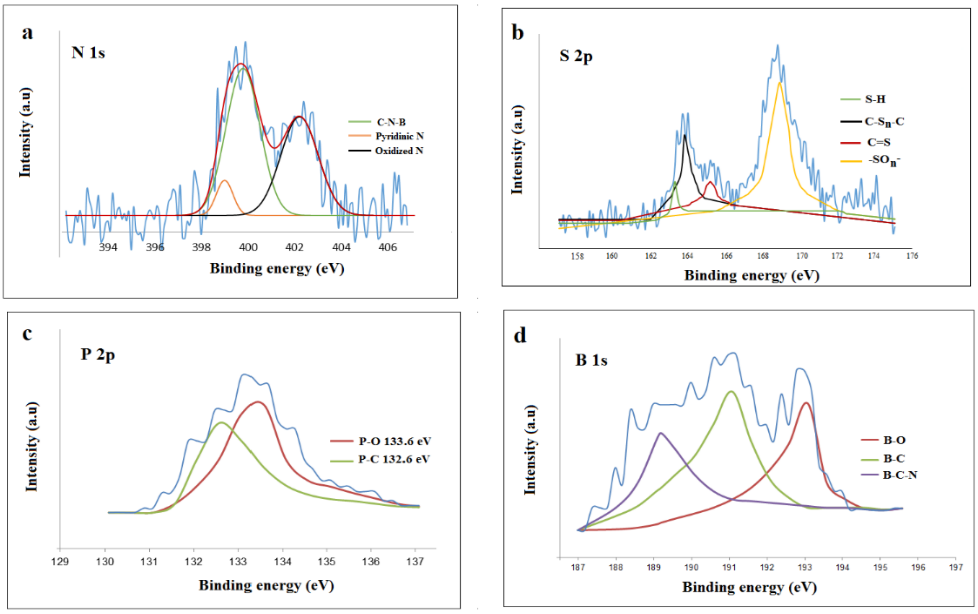

Figure 2. The XPS high resolution spectra of prepared sample, (a) N 1s, (b) S 2p, (c) P 2p, (d) B1s

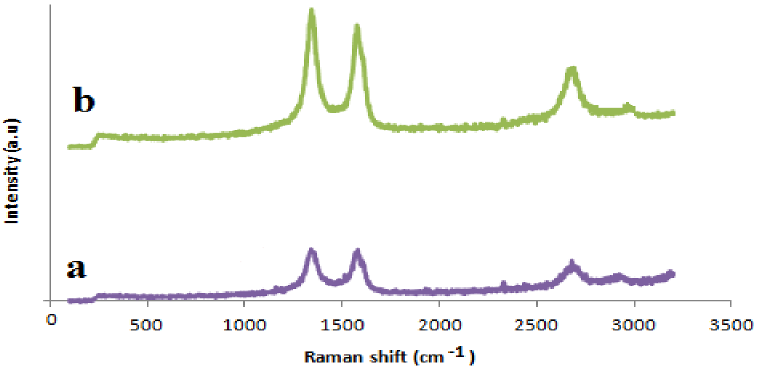

Figure 3. Raman spectra of (a) Graphene, (b) doped sample

3.2. Composition of the CPE

The selectivity of an ion selective electrode determines its selectivity behavior [30-37], and since former experiments have shown that CEF has selectivity towards Yb^{3+}, and CEF was used in preparing a modified CPEs, and the results are presented in Table 1.

Table 1. The various compositions of the modified CPEs constructed

Electrode No.	Binder	CEF	Graphite Powder	NSPBG	Slope mV per decade of concentration
1	15%-Paraffin oil	4%	81%	0%	11.1±0.3
2	15%-Paraffin oil	6%	79%	0%	11.9±0.2
3	15%-Paraffin oil	10%	75%	0%	13.2±0.4
4	15%-Paraffin oil	13%	72%	0%	14.2±0.4
5	15%- Paraffin oil	17%	68%	0%	14.3±05
6	15%- [bmim]BF_4	13%	72%	0%	16.3±0.4
7	20%- [bmim]BF_4	13%	67%	0%	16.7±0.4
8	30%- [bmim]BF_4	13%	57%	0%	16.9±0.2
9	35%- [bmim]BF_4	13%	52%	0%	16.9±0.3
10	30%- [bmim]BF_4	13%	54%	3%	18.6±0.1
11	30%- [bmim]BF_4	13%	52%	5%	19.3±0.2
12	30%- [bmim]BF_4	13%	50%	7%	19.7±0.3
13	30%- [bmim]BF_4	13%	48%	9%	19.7±0.2

Among the compositions not containing NSPBG, the CPE containing 15% wt. of paraffin oil, 70% wt. of graphite powder, and 13% wt. of CEF (no. 4) had the best response (i.e. a sub-Nernstian slope of ~14.2 mV per decade), and even higher amounts of CEF (17% wt.) left no influence on the response of the CPE (no. 5), and therefore 13% wt of CEF was considered the optimal amount of the ionophore for constructing the CPE. In a next step the paraffin oil was replaced with an equal amount (i.e. 20% wt.) of 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4), which enhanced the response of the CPE to 15.3±0.1 mV per decade of concentration (no. 6). Interestingly increasing the RTIL quantity up to 30% wt. (no. 8) further enhanced the sensitivity of the electrode to 16.9±0.2 mV per decade of concentration, which was attributed to the improved extraction of ions into the CPE matrix. This is especially important for ions with high charge densities, due to the fact that the dielectric constant of RTILs is much higher than that of the paraffin oil.

The next step was adding various amounts of NSPBG to the CPE composition which substantially improved the electrode response. Incorporating 3%, 5%, and 7% wt. of NSPBG in the electrode composition (no. 10, 11 and 12 respectively) greatly enhanced the electrode response from 16.9±0.3 to 19.7±0.1 mV per decade of concentration, but this improvement did not go on after 7% wt, as indicated by composition 13 containing 9% wt. of NSPBG. This behavior was also attributed to the better conductivity in the presence of NSPBG which was further reflected by the enhancement of the dynamic measurement range, and response time of the sensor. Considering the results a electrode containing 30% wt. of [bmim]BF_4, 13% wt. of CEF, 50% wt. of graphite powder, and 7% wt. of NSPBG (no. 12) was considered as the best electrode and the rest of the studies were performed using this electrode.

3.3. Linear measurement range and detection limit

The results obtained using the optimal electrode for the analysis of a set of Yb^{3+} solutions with concentrations ranging from 1.0×10^{-8}-1.0×10^{-1} mol L^{-1} was potted as the calibration curve of the developed CPE (Figure 4). The results showed that the response is linear in the range of 1.0×10^{-7} to 1.0×10^{-2} mol $L^{-1}$with a detection limit of 7.5×10^{-8} mol L^{-1}. The detection limit was determined through extrapolating the linear parts of the calibration plot at low concentrations [38-40].

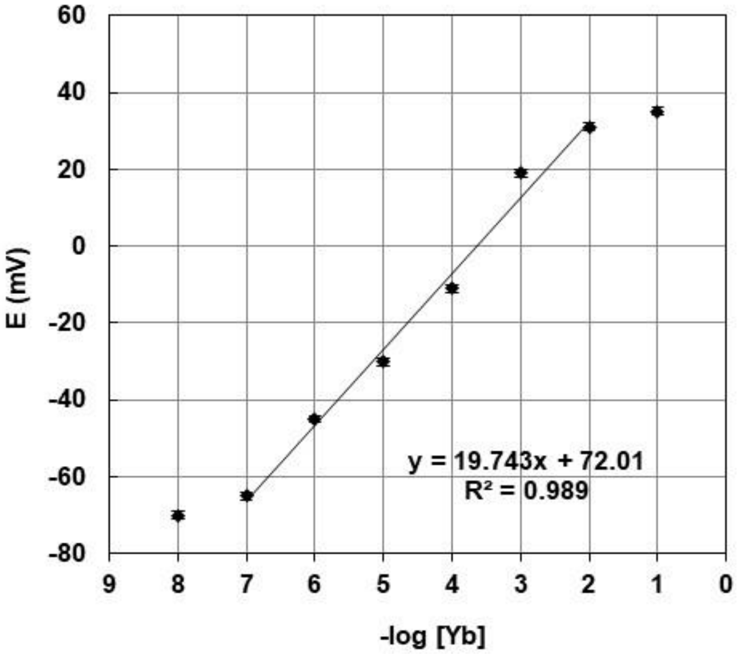

Figure 4. Response profile of the e of the Yb^{3+} sensor with composition no. 10

3.4. Effect of pH

The response behavior of the electrode against varying pH values was monitored through using the electrode in a fixed concentration of Yb^{3+} (1.0×10^{-4} mol L^{-1}) and reading its responses upon varying pH in the range of 2-10. The changes in the pH were made by adding small quantities of concentrated solutions of HNO_3 or NaOH to the test solution to avoid significant changes in the concentration of Yb^{3+} solution. The potential readings were plotted against pH (Figure 5), which indicated that the electrode response is pH-independent in the range of 2.0 to 7.0. This is also a measure of the absence of interference from H^+ or OH^- ions over this range. At pH values over 7.0, however, the electrode potential considerably changes which was attributed to the formation of soluble hydroxyl complexes of Yb^{3+} (i.e. $Yb(OH)^{2+}$and $Yb(OH)_2{}^+$) or insoluble $Yb(OH)_3$. At pH values below 3.0, the changes in the potential response of the electrode were ascribed to the protonation of the donating atoms of CEF, which can also be named considerable interferences on the part of the H^+ ions.

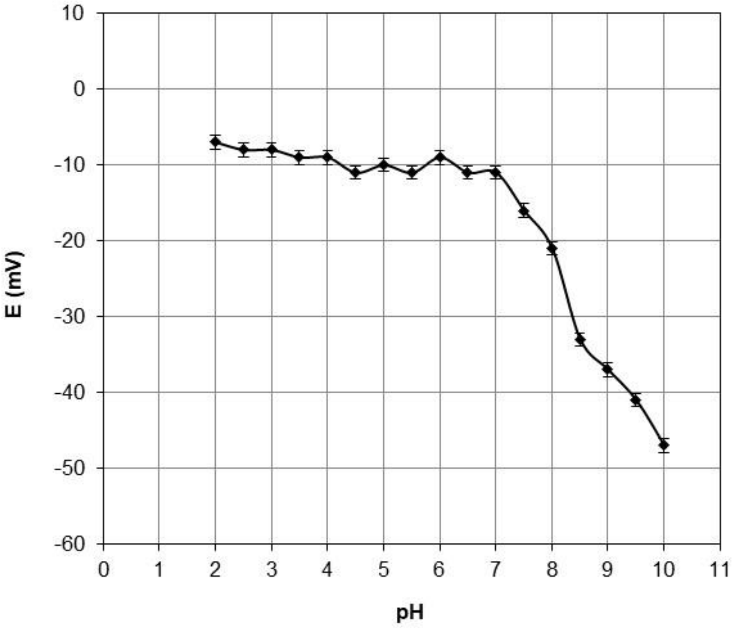

Figure 5. Effect of pH on the response profile of the optimal modified Yb^{3+} CPE in a 1.0×10^{-4} mol L^{-1} solution of Yb^{3+} ion

3.5. Response time

The response time of an ion selective sensors is measured by determining the time required for the sensor to reach a steady potential within ±0.1 mV of its equilibrium potential upon being subjected to a 10-fold change in the concentration of the target ion. This is commonly done by successively immersing an electrode into different solutions of the target ions with 10-fold concentration differences, while the other factors like stirring or the flow rate, matrices of the test solutions are similar in all solutions [33-43]. The experiments performed using the Yb^{3+}-CPE, revealed a response time of less than 15s between 10^{-3}-10^{-2} M, and around 30s with more dilute solutions (10^{-6}-10^{-4} M).

3.6. Selectivity

The most significant feature of an ion-selective sensor, is its selectivity behavior which is reflected by its selectivity coefficients. These coefficients indicate the sensor’s preference for the target ion, in the presence of other ions which are referred to as interfering ions . In the case of the developed CPE the selectivity coefficients were determined according to the matched potential method (MPM) using a 1.0×10^{-7} mol L^{-1} solution of Yb^{3+} and 1×10^{-4} to 1.0×10^{-1} mol L^{-1} solutions of the interfering ions [40-43], and are presented in Table 2.

According to the data the highest interference was observed in the case Tb^{3+} which has a selectivity coefficient of 7.8×10^{-3}. This indicates the presence of negligible interferences by tested ions.

Table 2. The MPM selectivity coefficients of the developed CPE

Cation	**Selectivity Coefficient**	**Cation**	**Selectivity Coefficient**
Na^+	2.5×10^{-4}	Gd^{3+}	3.2×10^{-3}
K^+	2.3×10^{-4}	Pr^{3+}	6.3×10^{-3}
Nd^{3+}	4.1×10^{-3}	Tb^{3+}	7.8×10^{-3}
Ho^{3+}	$6{,}0\times10^{-4}$	La^{3+}	2.3×10^{-3}
Ca^{2+}	5.1×10^{-4}	Sm^{3+}	5.5×10^{-3}
Cu^{2+}	2.7×10^{-4}	Dy^{3+}	4.8×10^{-3}
Pb^{2+}	2.1×10^{-4}	Lu^{3+}	4.0×10^{-4}
Fe^{3+}	1.9×10^{-4}	Eu^{3+}	2.9×10^{-3}
Ce^{3+}	3.3×10^{-3}		

3.7. Lifetime

Most ion-selective sensors have average lifetimes of 4–10 weeks. After this period sensitivity and/or detection limits of sensors deteriorate.

Table 3. Lifetime of Pr^{3+} -selective CPE

Week	**Slope (mV decade^{-1})**	**DL (mol L^{-1})**
1	19.7±0.4	6.3×10^{-8}
2	19,8±0.3	8.1×10^{-8}
3	19.6±0.2	9.2×10^{-8}
4	19.3±0.3	1.7×10^{-7}
5	19.0±0.3	3.3×10^{-7}
6	18.3±0.2	5.5×10^{-7}
7	18.0±0.3	8.7×10^{-7}
8	18.0±0.4	1.2×10^{-6}
9	17.1±0.2	4.3×10^{-6}
10	15.9±0.2	7.2×10^{-6}
11	15.12±0.2	9.6×10^{-6}
12	12.3±0.5	2.3×10^{-5}

In the case of the developed sensor , the lifetime studies were conduced over a 12-week period, during which specific sensors were used in a daily basis for about two hours. The results indicated that the senosr could be used for about 7 weeks without observing considerable changes in its sensitivity or detection range . After 10 weeks, however, the sensitivty of the sensor changed from from 19.7 to 12.3 mV per decade and the detection limit reached as high as 2.3×10^{-6} mol L^{-1} (Table 3). The deterioration of the beharior of the sensor was attributed to well known phenomena of the loss of ionophore and other material from the electrode due to the repeated use .

4. CONCLUSIONS

The cefixime-based Yb^{3+} -CPE containing N, S, P and B doped graphene (NSPBG), and 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4) had good response to the target species with good selectivity, which was attributed to improved electrical properties of the carbon paste matrix due to the incorporation of NSPBG and the RITL binder in its composition, further to the effects of the ionophore. The electrode had a favorable analytical property i.e. a response slope of 19.7±0.1 mV per decade of concentration, from 1.0×10^{-7} to 1.0×10^{-2} mol L^{-1}, negligible selectivity coefficients of 7.8×10^{-3} or less, and rather a long lifetime of more than 7 weeks.

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