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Modified Praseodymium Sensor based on Nitrogen and Sulfur Doped Porous Graphite

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Abstract- Incorporation of N and S containing porous graphite (NSPG) in the composition of carbon paste electrodes (CPEs) can improve their sensing behavior. Result of former studies have indicated the selective interaction of N, N-bis(a-methylsalicylidene) diethylenetriamine (BMT) and Pr^{3+} ions, as opposed to other lanthanide ions. In this light, BMT was incorporated into a mixed matrix of NSPG and graphite powder to obtain a modified Pr^{3+} potentiometric sensor. The results indicated that the nano-composite sensor offers enhanced sensitivity, selectivity; response time; response stability and lifetime in comparison to CPEs modified using multiwall carbon nanotube (MWCNT). The optimal response (i.e. a Nernstian response of 19.6±0.1 mV per decade from 1.0×10^{-7} to 1.0×10^{-2} mol L^{-1}) was observed for a sensor containing 10% NSPG, 15% BMT, 30% IL and 45 % graphite powder.

Keywords- Sensor; Ion-selective electrode; Carbon paste; N, S doped porous graphite; Room temperature ionic liquid; Praseodymium

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1. INTRODUCTION

As a relatively novel group of solvents, ionic liquids (ILs), which can be simply defined as salts that are unusually liquid at low temperatures have different applications. Technically

ILs are ionic compounds that are liquid below 100 °C. ILs offer remarkable properties and are commonly used as solvents in different areas of electrochemistry. These most common examples of these ionic compounds are composed of asymmetrically substituted nitrogen-containing cations such as imidazolium, pyridinium, pyrrolidinium with large organic or inorganic anions like Cl^-, PF_6^-, BF_4^-. The highest electrical conductivity among ILs has been reported for ionic liquids with imidazolium and pyridinium cations (around 1 and 10^{-1} S/m, respectively) [1]. These liquids offer advantages of good solvating properties, considerable conductivity, high boiling points, negligible toxicity, and excellent electrochemical stability [2,3].

One important area, where the advantages of ILs are important, is their role as a binder in the composition of carbon paste electrodes (CPEs) [2-5]. CPEs are electrochemical sensing devices, usually made of a paste formed through admixing graphite powder in a nonconductive mineral oil, which has been reported to suffer certain disadvantages. One key issue with mineral oils is that they have varying compositions because they are obtained from various petroleum-refining products. This has been associated with unpredictable influences on the analytical results obtained using CPEs [6].

On the other hand, ion-selective electrodes (ISEs) constitute a widely used class of analytical devices that are applied in different experiments [7-18] due to advantages of portability, ease of use, high speed, low costs, high sensitivity, and highly reliable response over broad concentration windows as opposed to rivaling methods [19-27]. CPEs are among the different classes of potentiometric ISEs. Carbon paste electrodes have various merits over many other forms of potentiometric ISEs, like PVC-based membrane ISEs, including renewable surfaces, stable response profiles, and negligible Ohmic resistance [3-5]. These sensors are usually composed of a selective species, graphite powder, a modifier and a non-conductive mineral oil.

Based on what was described, and given the results of previous selectivity studies [28], which indicated selective interactions between N,N-bis(a-methylsalicylidene) diethylenetriamine (BMT), Figure 1, and Pr^{3+} ions, this study was aimed at constructing an efficient potentiometric CPE for the analysis of Pr^{3+} ions using BMT as the sensing material, in a matrix further containing RTIL and N, S doped porous graphite.

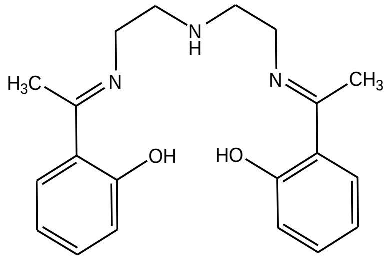

Figure 1. Chemical structure of N,N-bis(a-methylsalicylidene)diethylenetriamine (BMT) [28]

Praseodymium, is present in strong and durable magnets together with neodymium [29]. Mixtures of this element and magnesium are used in alloys used in under-pressure sections of aircraft engines [30]. Like neodymium and magnesium, relatively high amounts (up to 5%) of Pr are also present in Misch Metal compositions. Fluorides of Pr and other lanthanide elements have been used in carbon arcs and projector lights, and praseodymium is also used to create color in different glass samples known as didymium glass, which is used in protective goggles used in welding and glass blowing. Praseodymium silicate crystals are known to create light pulses of as low as a few hundred meters per second, and they are therefore used.in devices requiring this phenomenon [31]. $PrNi_5$ alloys containing nickel and praseodymium are known for their considerable magneto-caloric effects, which make them suitable for use in creating very low temperatures very close to absolute zero [32]. Praseodymium and its oxides are also present in fluoride glass used in single-mode fiber-optical amplifiers [33] and used in solid solutions further containing ceria, or ceria-zirconia [34].

2. EXPERIMENTAL PART

2.1. Reagents and materials

The sulfur trioxide pyridine complex (STPC), camphor, KOH, and HCl (37%), used in preparing NSPG were obtained from Sigma-Aldrich Sigma- Aldrich. 1–2 μm graphite powder and high-purity paraffin oil were obtained from Merk and Aldrich respectively. 1-n-butyl-3-methylimidazolium tetrafluoroborate ($[bmim]BF_4$) and chloride and nitrate salts used to prepare different solutions were obtained from Merck Co. N,N-bis(a-methylsalicylidene) diethylenetriamine (BMT) was synthesized as explained before [28].

2.2. Synthesis of N, S co-doped porous graphite

Chemical vapor deposition (CVD) at 900–1050 °C was used for 2–50 min to prepare porous graphene (PG) in ambient pressure using MgO nanoparticles as a catalyst, and a 4:1 flow of methane and hydrogen as the carbon source and the carrier gas. The produced PG was next purified using 18% HCl (at 30°C) for 3 hours and then washed with deionized water until neutral pH was reached. The cleaned product was eventually dried at 100°C in an oven. To prepare the N,S co-doped graphene electrocatalyst, a solution including 100 mg graphene in 100 ml ethanol was prepared using an ultrasonic bath for 30 min. Then, Sulfur trioxide pyridine complex solution was added into the first solution under stirring for 5 h in room temperature and then dried at 60°C, grounded and pyrolyzed at 900 C for 2 h in N_2 atmosphere [35,36].

2.2. Apparatus

The analytical cell included the CPE as a Pr^{3+} sensor and an Ag/AgCl reference electrode (Azar electrode, Iran) as a reference electrode which was connected to a millivoltmeter, forming the following cell assembly:

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

2.3. Electrode Preparation

The typical procedure for preparing the CPE involved admixing desired amounts of BMT (ionophore), graphite powder, 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4), and NSPG. The obtained pastes were then packed in 3cm long glass tubes (5 mm i.d.) while avoiding the formation of possible air gaps, which can create considerable electrical resistance and rendering the electrodes useless. Next, a copper wire was inserted into the tube and in the paste and the exterior surface of the CPE was cleaned using soft abrasive paper. The resulting CPE electrode was eventually inserted in a 1.0×10^{-3} mol L^{-1} $PrCl_3$ solution and kept under this condition for two days for conditioning [3-5].

3. RESULTS AND DISCUSSION

3.1. SEM and XPS results

The surface morphology analysis of the prepared N and S co-doped graphene samples is investigated using FESEM (Figure 2), which shows a similar structure to the graphene structure according to our last works [35-37].

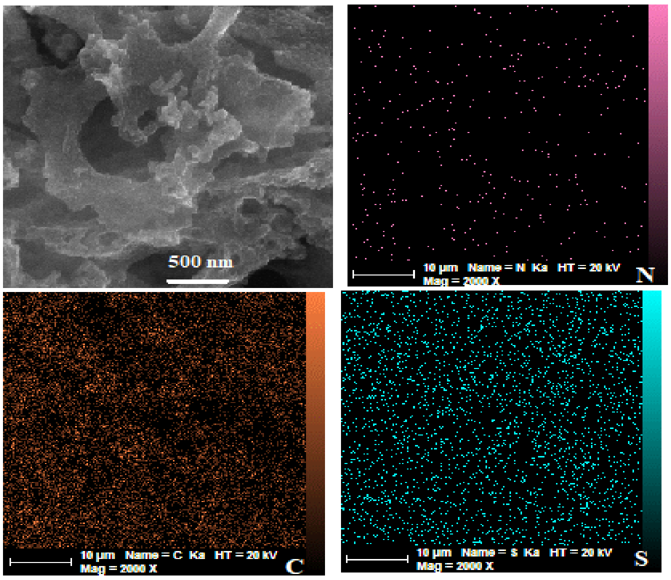

Figure 2. FESEM micrographs of prepared doped graphene and EDX mapping analysis of Carbon, sulfur and nitrogen

Moreover, the elemental mapping distribution analysis shows the homogenous and uniform dispersion of nitrogen and sulfur elements in the carbon structure of samples, which indicates the successful heteroatoms doping into graphene lattice [35,38].

In addition, the XPS results show the elemental composition of the doped sample and confirm the heteroatoms doping in the structure (Figure 3-a). According to the high-resolution spectra of the nitrogen atom (Figure 3-b), three main peaks can be observed, which the peaks around 398.2, 400.2, and 403.2 eV can be attributed to pyridinic, pyrrolic, and oxidized types of nitrogen atoms, respectively. The high-resolution spectra of the sulfur atom (Figure 3-c) show peaks around 163.2, 163.67, 165.00, and 168.53 eV of the S 2p are evidence of the presence of SH, $C-S_n-C$ and C=S and other oxidized forms in the graphene structure [35,39,40].

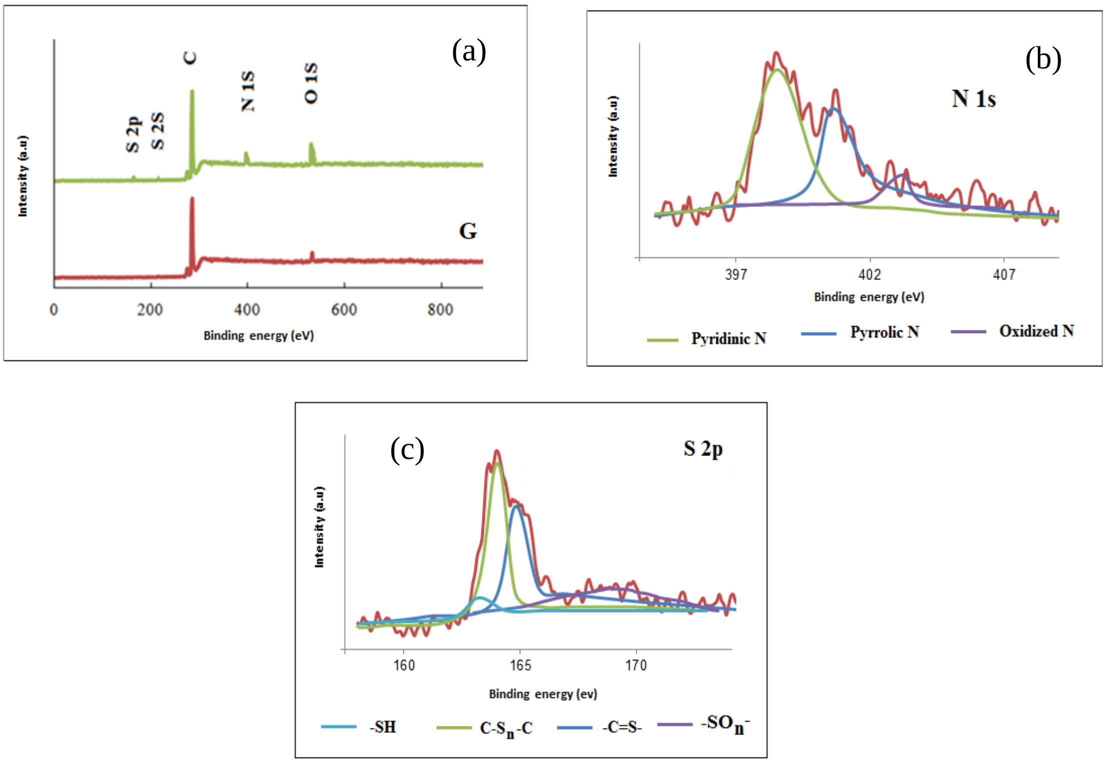

Figure 3. XPS Survey for PG and doped-PG samples (a), the high-resolution XPS spectra of N 1s (b) and S 2p (c)

3.2. Composition of the CPE

The ionophore present in the composition of a potentiometric ISE has a key role in the selectivity profile of the resulting electrode [21-25]. In the light of reports on the selectivity behavior of BMT [28], the compound was considered as a selective ionophore for Pr^{3+} ion, and it was hence used in preparing various modified and unmodified CPEs, the results of analyses based on which can be seen in Table 1.

Table 1. The optimization of the carbon paste ingredients

Electrode No.	**Binder**	**BMT**	**Graphite Powder**	**NSPG**	**Slope mV per decade of concentration**
1	15%-Paraffin oil	5%	80%	0%	10.2±0.5
2	15%-Paraffin oil	7%	78%	0%	11.3±0.3
3	15%-Paraffin oil	10%	75%	0%	12.6±0.5
4	15%-Paraffin oil	15%	70%	0%	13.8±0.1
5	15%- Paraffin oil	20%	65%	0%	13.9±0.4
6	20%- [bmim]BF_4	15%	65%	0%	15.3±0.1
7	30%- [bmim]BF_4	15%	60%	0%	16.3±0.4
8	30%- [bmim]BF_4	15%	46%	4%	16.9±0.2
9	30%- [bmim]BF_4	15%	48%	7%	17.9±0.3
10	30%- [bmim]BF_4	15%	45%	10%	19.6±0.1
11	30%- [bmim]BF_4	15%	40%	15%	19.6±0.2

In the absence of NSPG, the best response was observed for the CPE containing 15% wt. of paraffin oil, 70% wt. of graphite powder, and 15% wt. of N,N-bis(a-methylsalicylidene)diethylenetriamine (BMT) (no. 4), which was a linear, yet sub-Nernstian slope of ~13.8 mV per decade. Increasing the amount of BMT to 20% wt. did not have a considerable effect on the response of the CPE (no. 5). Subsequently, 15% wt. was considered as the optimal ligand content for the CPE. Interestingly replacing the paraffin oil with an equal amount (i.e. 20 % wt.) of 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4) enhanced the response of the sensor from 13.8±0.1 mV per decade of concentration to 15.3±0.1 (no. 6), and this effect continued up to an RTIL content of 30% wt. (no. 7). This was expected, since the incorporation of the room temperature ionic liquid, instead of the paraffin oil, is known to enhance the extraction of ions into the CPE matrix, especially in the case of ions with high charge densities. This effect is attributed to the greatly higher dielectric constant of RTILs as opposed to paraffin oil.

The next substantial improvement in the response of the CPE, was observed upon adding NSPG. The presence of respectively 4%, 7% and 10% wt. of NSPG (compositions 8, 9 and 10) remarkably increased the response of the electrode from 16.9±0.2 to 19.6±0.1 mV per decade of concentration, but this did not continue by further increasing the NSPG content to 15% wt. This was further attributed to the improved conductivity of the CPE matrix, improving the dynamic measurement range, and response time of the CPE. Subsequently, the CPE composed of 30% wt. of [bmim]BF_4, 15% wt. of BMT, 45% wt. of graphite powder, and 10% wt. of

NSPG (no. 10) was considered as the optimal electrode and further evaluations were performed using this electrode.

3.3. Measurement range and limit of detection

The response profile of the CPE with the optimal response was studied over a wide concentration window of 1.0×10^{-8}-1.0×10^{-1} mol L^{-1} (Figure 4), and its response was found to be linear from 1.0×10^{-7} to 1.0×10^{-2} mol L^{-1}. The detection limit of the sensor was determined to be 8.5×10^{-8} mol L^{-1} by extrapolating the linear sections of the calibration curve at lower concentrations.

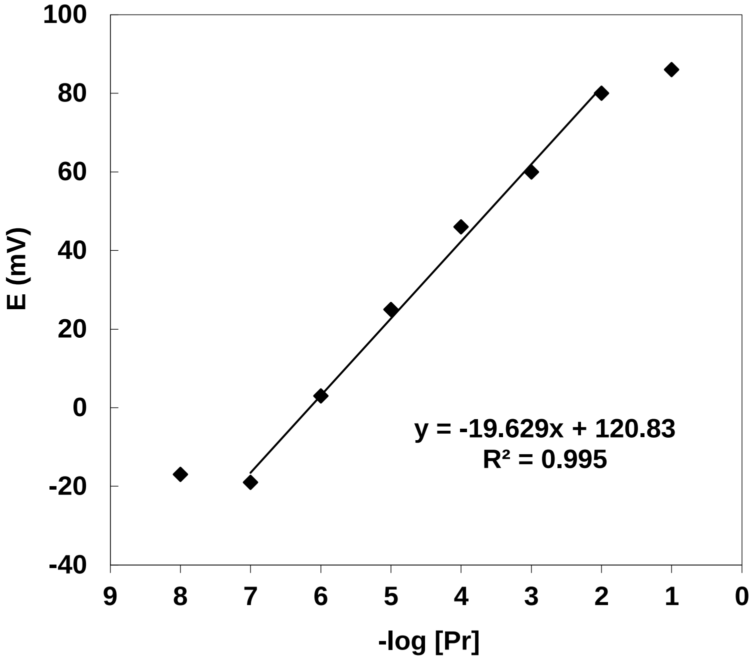

Figure 4. Response profile of the e of the Pr^{3+} sensor with composition no. 10

3.4. pH effect on the electrode response

To monitor the changes in the response of the optimal electrode by altering the pH of the solution, the response of the electrode to a fixed concentration of Pr^{3+} ion was recorded at various pH values in the range of 2-10. The pH of the solution was changed by adding concentrated solutions of HNO_3 or NaOH, to avoid significant dilution of the Pr^{3+} ion solution. A plot of the resulting data is presented in Figure 5, which clearly shows the electrode response was independent of pH over the range of 3.0 to 7.0. This also means a lack of considerable interferences from the H^+ or OH^- ions that are intrinsically present in the solution, in this pH range. The changes in the response of the electrode at pH values over 7.0, were attributed to the formation of soluble hydroxyl complexes of Pr^{3+} (i.e. $Pr(OH)^{2+}$and $Pr(OH)_2^+$) or insoluble $Pr(OH)_3$. Changes in the potential response at pH values below 3.0 were attributed to the interaction of the electrode donor atoms in BMT with H^+ ions, or in other words the considerable interference of the proton ions.

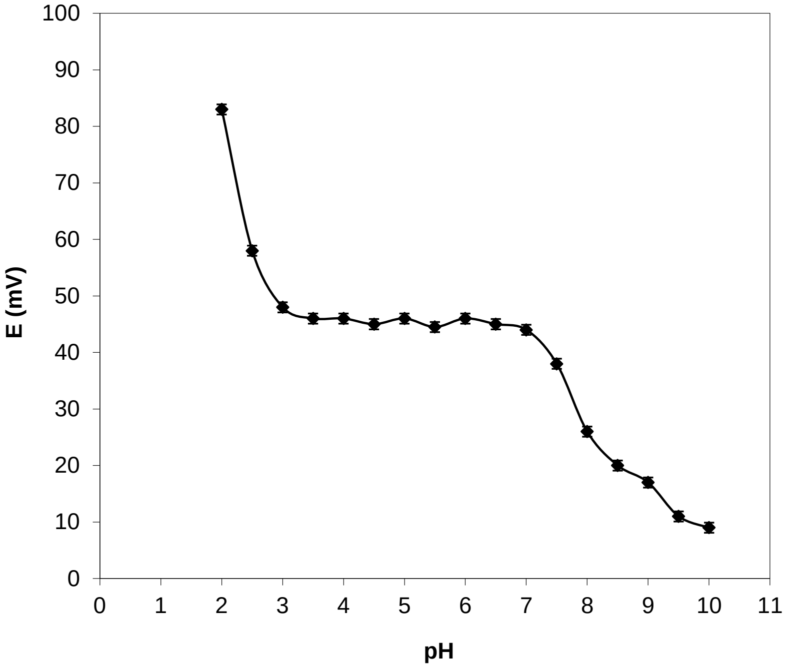

Figure 5. pH effect on the potential response of Pr^{3+} CPE with composition no. 10 in the solution of 1.0×10^{-4} mol L^{-1} of Pr^{3+} ion

3.5. Response time

The response time of electrochemical sensors is determined through determining the average time taken to reach a steady potential within ±0.1 mV of the equilibrium potential of an electrode, after subjecting the electrode to a 10-fold concentration change. The tests are often performed by successive immersions of the electrode into various solutions of the target species. Other experimental conditions like stirring or the flow rate, concentration, test and conditioning solution matrices, any previous applications or preconditioning of the electrode, and temperature can influence the response [14-19]. In the case of the developed Pr^{3+}-selective CPE, the response time was determined to be below 12s in the concentration range of 10^{-3}-10^{-2} M, while it was almost twice this value (i.e. around 25s) when more dilute solutions in the range of 10^{-6}-10^{-4} M were used.

3.6. Selectivity

As the most important property of a sensor, selectivity coefficients illustrate a sensor’s preference toward the target ion when other interfering ions are present in the solutions. The potentiometric selectivity coefficients of the CPE were determined using the matched potential method (MPM) [22-25], and the results are summarized in Table 2. In the tests, a 1.0×10^{-7} mol

L^{-1} solution of Pr^{3+} ion and 1×10^{-4} to 1.0×10^{-1} mol L^{-1} solutions of the interfering cations were used.

Table 2. The matched potential method (MPM) selectivity coefficients the optimal CPE against different interfering ions

Cation	Selectivity Coefficient	Cation	Selectivity Coefficient
Na^+	1.3×10^{-4}	Gd^{3+}	4.2×10^{-3}
K^+	2.0×10^{-4}	Yb^{3+}	6.6×10^{-3}
Nd^{3+}	3.7×10^{-3}	Tb^{3+}	5.7×10^{-3}
Ho^{3+}	8.6×10^{-4}	La^{3+}	1.3×10^{-3}
Ca^{2+}	7.4×10^{-4}	Sm^{3+}	5.0×10^{-3}
Cu^{2+}	2.2×10^{-4}	Dy^{3+}	4.1×10^{-3}
Pb^{2+}	2.5×10^{-4}	Lu^{3+}	7.0×10^{-4}
Fe^{3+}	1.5×10^{-4}	Eu^{3+}	2.0×10^{-3}
Zn^{2+}	4.2×10^{-4}	Ce^{3+}	4.6×10^{-3}
Tm^{3+}	6.4×10^{-3}	Er^{3+}	5.5×10^{-4}

The results in Table 2 clearly show that the maximum interference was in the case of Yb^{3+} ions, with a selectivity coefficient of 6.6×10^{-3}, indicating the negligible influence of the presence of the tested interfering species on the response of the developed CPE.

3.7. Lifetime

On average, most ion-selective sensors have lifetimes ranging from 4–10 weeks, after which the sensitivity (potential slope) or detection limit values deteriorate.

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

Week	Slope (mV decade^{-1})	DL (mol L^{-1})
1	20.1±0.4	6.3×10^{-7}
2	20.0±0.3	8.5×10^{-7}
3	19.9±0.2	9.6×10^{-7}
4	19.7±0.3	1.0×10^{-6}
5	19.5±0.3	1.2×10^{-6}
6	19.4±0.2	1.5×10^{-6}
7	19.2±0.3	2.7×10^{-6}
8	19.0±0.4	3.2×10^{-6}
9	19.1±0.2	4.5×10^{-6}
10	18.9±0.3	5.5×10^{-6}
11	17.2±0.2	8.5×10^{-6}
12	15.4±0.4	1.5×10^{-5}

The lifetime of the developed CPE was monitored over a 15-week period. During this period the sensor was subjected to a daily use of around two hours. based on the results, the electrode could be used for at least 10 weeks without significant changes in its performance. After 10 weeks, the slope of the calibration curve slightly decreased from 19.6 to 14.4 mV per decade and the detection limit increased from 8.5×10^{-8} mol L^{-1} to 5.4×10^{-6} mol L^{-1} (Table 3). As in the case of many other ions selective electrodes, this is attributed to the loss of sensing material from the electrode into the test solutions upon repeatetive use.

4. CONCLUSIONS

The developed Pr^{3+} carbon paste electrode based on N,N-bis (a-methylsalicylidene) diethylenetriamine showed good response to the analyte in the presence of N and S containing porous graphite (NSPG), and 1-n-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4). The selectivity was attributed to the presence of the ligand, while the enhanced behavior was further due to the improved electrical properties of the carbon paste matrix due to the presence of NSPG in the matrix and the room temperature ionic liquid as the binder. The electrode enjoyed good analytical performance (a response slope of 19.6±0.1 mV per decade of concentration over the concentration window of 1.0×10^{-7} to 1.0×10^{-2} mol L^{-1}), very low selectivity coefficients ($<6.6\times10^{-3}$), and rather a long lifetime of over 10 weeks.

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