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Research Article

Disposable Screen-Printed Microchip Based on Nanoparticles Sensitive Membrane for Potentiometric Determination of Lead

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Realization of screen-printed disposable microchip based on organic membrane sensitive layer highly responsive to lead has been demonstrated for the first time. Fabrication, potentiometric characterization and analytical application of the novel microchip have been reported. A sensitive layer comprises TiO_2 nanoparticles and multiwalled carbon nanotubes "MWCNTs" composite incorporated in PVC membrane has uploaded on the plastic screen-printed microelectrode substrate surface using novel protocol. The new chip provided a linear behavior for Pb^{2+} ions over the lead concentration range of 1×10^{-6}–1×10^{-1} mole L^{-1} with super Nernstian sensitivity (49 mV), relatively long life span (>4 months), and a fast response time (10 s). The advantages showed by the microchip include simple fabrication, small size, mass production, cost effectiveness, and automation and integration feasibility. The realized new microchip has been successfully utilized in the quantification of some lead (II) samples with average recovery of 101.9% and the RDS was <3.

1. Introduction

Potentiometric sensor and devices have received a great attention in the last few years. Recently, major attention has been focused on the miniaturization and patterning of these devices from bulk to microchips scale. Microfabrication methodology of the miniaturized microchips mainly based on the uploading of the membrane sensitive coat on the microsized substrate support. In this context, screen-printing is a well-known technique for realizing the microsensor assemblies on various substrates (i.e., plastic, ceramics, paper, glass, etc.) [1–5]. The advantages provided by the screen-printed electrodes are fast response times, high accuracy, robustness, low cost, require small quantities of reagents, miniaturization, large-scale production, reduced size, disposable, reduced impedance output, and relatively mass production. These analytical devices are especially useful for various medical and environmental measurements carried at remote sites (infield or in situ determination) as well as in the continuous monitoring of the tested species. However, to improve the selectivity of the potentiometric sensors and selective electrodes, much research efforts have been focused on the development in modifying the sensors with nanoparticles based sensitive layer [6–17]. Such materials have unique properties to modify the potentiometric behavior of the sensors due to their high surface to volume ratio [10, 12, 17]. Thus, many attempts have been done by scientist to fabricate plastic based screen-printed microchips using organic membrane sensitive coat. Unfortunately, most of these attempts were failed. The failure of realization of such type of microdevices is due to both of them (sensitive membrane and the chip substrate) are organic matter and consequently, attempting to upload the organic sensitive membrane generally dissolve the organic chip substrate.

On the other hand, the assessment of the toxic metals particularly, lead has recently increased due to their harmful effects to the environment and the human. Lead toxic heavy metal has been widely used in industry for many years. Many health problems result from exposure to high content of lead, namely hemotoxic effects, dysfunction of infants nervous and fetuses, reproductive dysfunction, Alzheimer's disease, gastrointestinal tract alterations, and nephropathies. The activity of heme synthetic enzymes can be inhibited by lead ions and thus anemia can result from lead poisoning. If lead poisoning is left untreated, it may damage the nervous system, kidneys, and the brain [18–29]. The methods in current use for the determination of lead and other toxic elements were reviewed [18–22]. More specifically, the spectrophotometric determinations of lead using flow injection analysis in industrial residual water [23] and in tap water [24] have been demonstrated. The voltammetric detection of lead in hair dye samples [25] and in tap water [26] have also been reported. Flame atomic absorption spectroscopy was applied in the detection of lead [27]. It was reported that, ICP-AES [28] and ICP-MS [29] were used in the quantification of lead. Nevertheless, these methods require sophisticated, expensive machines, tedious calibration and preparation steps, and analysts with extremely high experiences. However, ion selective electrodes and potentiometric sensors particularly, microsensors represent fast and simple analytical tools with high reliability and credibility and mass production. Moreover, microchip sensors are miniaturized devices with small size and consequently provide integration and automation feasibility.

Based on the above-mentioned facts, fabrication, evaluation, and application of the microsensors based on screen-printed are very interesting challenges for many scientists. Realization of different screen-printed modified electrodes were reported [30–39]. More specifically, fabrication, potentiometric characterization, and analytical application of screen-printed microchips were reviewed in the literature. These demonstrated the description of pharmaceutical and biological species analysis [30], Ag/AgCl chloride electrode [31], calibration less pH sensors [32], sodium alkyl sulfates detection [33], printed sensors [34], pesticide detection [35], small molecule sensing [36], carbon electrodes [37], biosensors modified with functional nucleic acids probes [38], and lead cations determination [39], respectively. Furthermore, optimized screen-printed microelectrode have been recently, developed for the determination of xylometazoline [40] and cyclobenzaprine [41].

The current study represents, for the first time, an attempt to fabricate new microchip based on screen-printed substrate using nanostructure organic sensitive membrane coat responsive for lead (II). The nanocomposite material embedded in plasticized PVC membrane was deposited on the surface of the microchip substrate using new, simple, fast, and economic approach. The realized lead (II) screen-printed microchip provides high sensitivity, fast response time, simple design, cheap, and integration and automation applicability. It represents simple, cheap, miniaturized, and mass production tool for lead measurements. The microfabrication, potentiometric evaluation, and analytical application of the new sensor were demonstrated.

2. Material and Methods

2.1. Chemicals and Reagents. All reagents applied—unless otherwise stated—were of analytical reagents assay. Further, deionized distilled water with Aquatron water distiller ($1.0\,\mathrm{M\Omega\,cm^{-1}}$, Bibby Scientific, UK, A4000D) has used in rinsing the glass tools throughout and in the dilution of the solutions as well. All reagents used in characterization study were prepared from chemicals of analytical reagent assay in deionized distilled water. Soluble salts of the metal used were obtained from Riedel-de Haën. A disposable screen-printed substrate microchips (0.25 mm PET, 3/6 mm in diameter working carbon electrode, SPE modified with graphene) were purchased from Suzhou Delta-biotech (Ltd., China) and used as the microelectrode substrate. Powder of titanium (IV) oxide anatase (99.9%, 32 nm) was obtained from Alfa Aesar (GmbH, Germany). Lipophilic additive (potassium tetrakis (4-chlorophenyl) borate) and solvent mediator (2-nitrophenyl octyl ether) were obtained from Sigma–Aldrich (CH-9471 Buchs, Switzerland). Multiwall carbon nanotube (MWCNT purified; purity: >95%, od: 30–50 nm, id: 5–12 nm, and length: 10–20 μm) was obtained from Chengdu organic chemicals company "COCC", China. THF (tetrahydrofuran) and poly (vinyl chloride) carboxylated (high molecular weight, 220,000) were obtained from Riedel-de Haën chemical company (Germany). All characterizations and detection studies were carried out at ambient room temperature.

2.2. Instrumentation. All potentiometric characterization experiments were performed by Jenway pH/mV meter (model 3510) using nanosensitive based microelectrode as indicator electrode responsive for lead (II) in conjunction with reference electrode (double junction, Metrohm Ag/AgCl). The calibration and potentiometric characterization was performed in solutions of pH: 5. The accuracy assessment study was conducted using inductively coupled plasma-optical emission spectrometer (ICP-OES), Thermo Scientific (iCAP 7,000 series, USA) instrument connected to an autosampler (Cetac, ASX-520) and supported by Qtera software. In such study and under the optimized plasma parameters (RF power: 1,150 W, nebulizer gas and auxiliary gas flow: 0.5 L/min), radial mode at 460–379 nm was applied. Further, the surface morphology study of the lead micro microchip was conducted using scanning electron microscope analytical (SEM, JEOL, model JSM-6390 LA). The diameter of the sensitive element nanoparticles was measured by transmutation electron microscope (TEM, JEOL model JEM-1011).

2.3. Microfabrication of Nanocomposite Based Lead (II) Chip. Deposition of organic membrane sensitive layer on surface of the plastic screen-printed electrode was realized for the first time using simple, fast, and cheap novel technique. In this approach, disposable plastic substrate (screen-printed microchip, Figure 1) was washed by deionized distilled water and left in air to dry before applied as electrode support in all assemblies. As summarized in Table 1, four microelectrode assemblies containing nanocomposite sensitive materials with different ratio were realized and tested as lead (II)

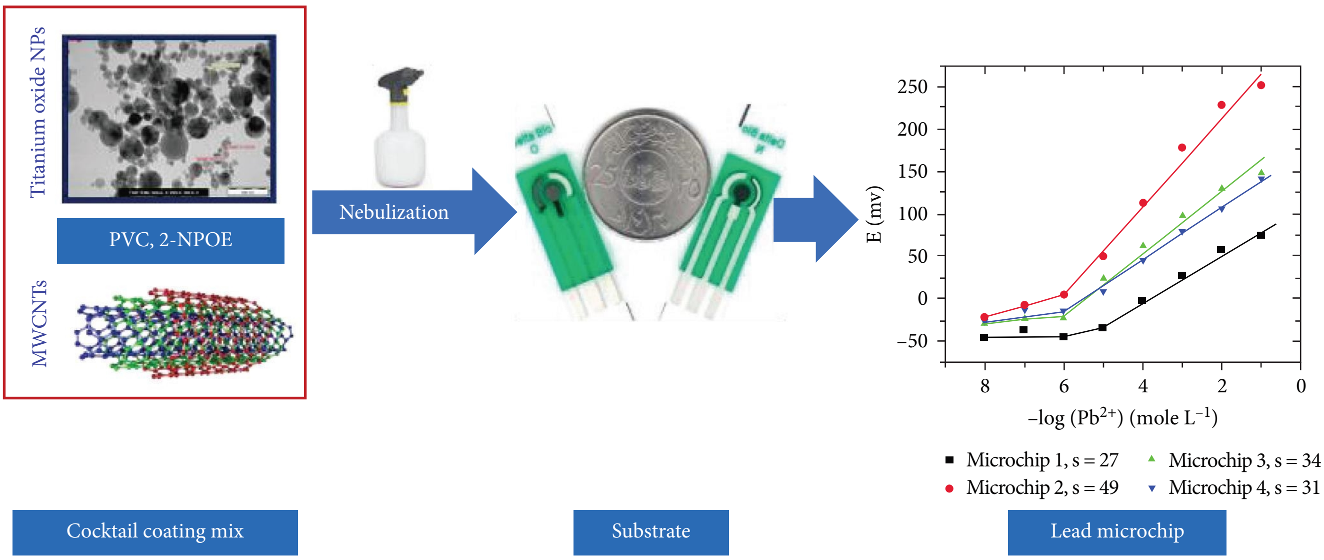

FIGURE 1: Schematic procedure to fabricate of the DSPLM electrode (*t*; 25°C, pH; 5, microchip 2).

TABLE 1: The composition of microelectrode DSPLM assemblies.

No.	Ionophore composite, 14 mg MWCNTs %	Ionophore composite, 14 mg TiO_2 %	Anion excluder, mg	2-NPOE, mg	PVC, mg
1	0	100	6	114	66
2	5	95	6	114	66
3	10	90	6	114	66
4	30	70	6	114	66

microelectrodes. The nanocomposite sensitive materials comprise TiO_2 as ionophore and MWCNTs as modifier. For each assembly, the coating cocktail mixture was realized by thoroughly mixing the ionophore (plasticized composite nanoparticles), anion excluder potassium tetrakis (4-chlorophenyl) borate, poly (vinyl chloride) support and THF solvent in small beaker. The mixture was then transferred into small manual homemade nebulizer and sonicated for 2 hr before being applied as a sensitive membrane layer. In fume hood, few microliter aliquots of the organic membrane coat were nebulized for few seconds onto the surface of screen-printed microelectrode in successive manner. For solvent volatilization, the deposited thin layer coat was then left in air for 3 min. The last two successive steps were repeated for several times until a uniform layer of the organic membrane sensitive layer cover the substrate surface. To spread out the nanoparticles, the coating mixture sensitive material was sonicated for 2 hr before nebulization process and for 3 min between the successive nebulization steps as well. The realized microelectrode assemblies (disposable screen-printed lead microchip, DSPLM) were utilized in the evaluation and applications experiments as lead (II) microchips. Using such new methodology, five microelectrode assemblies were realized and characterized as lead (II) microelectrode.

3. Results and Discussion

Microfabrication of sensitive layer based on organic membrane on plastic screen-printed substrate (PVC-based sensitive layer onto organic chip substrate) was elaborate for the first time using new simple technique. The realized new microelectrode was electrochemically evaluated according to IUPAC regulations as lead (II) potentiometric sensor chip. The advantages provided by the new microelectrode resulted from the combination of the screen-printed microchip substrate with the organic membrane sensitive layer [3, 4]. While the miniaturized chip have the advantages of mass production, small size, simple construction, versatile applications, and cheap, the organic membrane sensitive layers are widely applied in many chemical sensors due to their high sensitivity, selectivity, and simplicity [12, 39]. Further, a new generation of screen-printed microchips based on this new approach will be realized. Huge number of organic membrane-based microelectrodes have been prepared using the new methodology, which is sensitive for biomedical species, drugs, toxic metals, heavy elements, and pollutants [35, 37, 38]. The miniaturized devices modify the automation and integration applicability and therefore, enhance the application of such microchips in network electrodes [12].

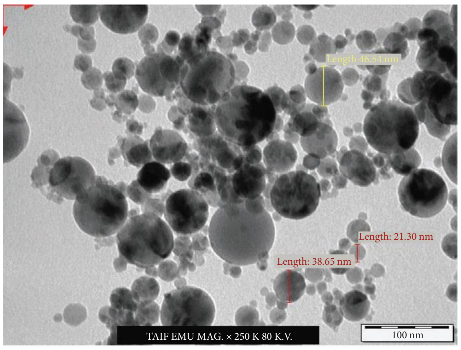

Figure 2: TEM image of TiO_2 nanoparticles.

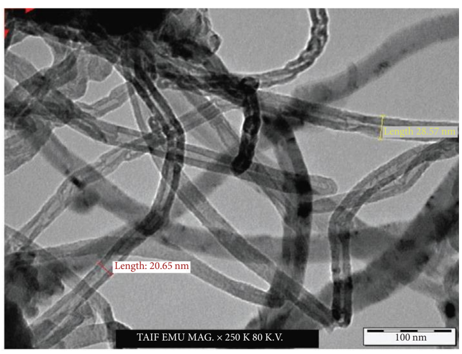

Figure 3: TEM image of carbon nanotube.

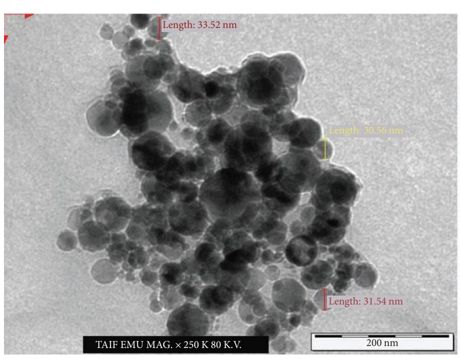

Figure 4: TEM image of TiO_2/MWCNTs composite in PVC membrane (microchip 2).

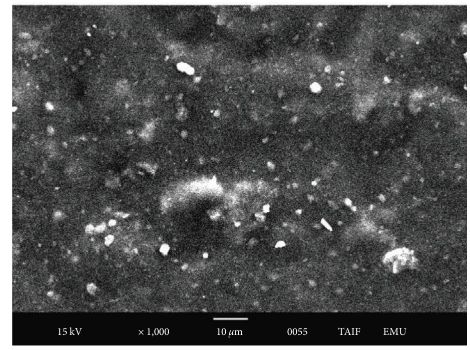

Figure 5: SEM image of TiO_2/MWCNTs composite in PVC membrane (microchip 2).

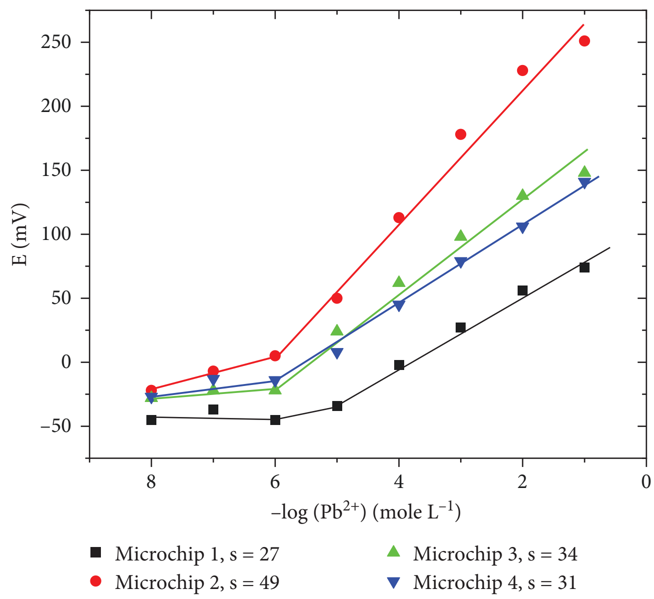

Figure 6: Calibration curves of DSPLM electrodes (microchip assemblies; 1–4, *t*; 25°C, pH; 5, sensitivity ± 1).

3.1. Characterization of the Nanocomposite Sensitive Material. The particles size of TiO_2, MWCNTs, and the nanocomposite sensitive material applied for microchip assembly 2 was measured using TEM and the results obtained were presented in Figures 2–4, respectively. Although the nanoparticles are not monodispersed, the results showed that, the diameters of the particles are relatively uniform and in the nanoscale having a size in average of ∼30 nm.

A typical scanning electron microscopy (SEM) picture of the TiO_2 nanoparticles/carbon nanotubes composite after embedded in organic membrane and uploaded on the screen-printed substrate surface applied for microchip assembly 2 is presented in Figure 5. As clearly seen, the nanoparticles of the composite sensitive material are uniform distributed on the screen-printed microchip substrate surface with some bright aggregations of the polymeric support. The good sensing properties obtained by the microchip was attributed to the homogenously distributed nanoparticles of the sensitive material coat contribute to [36].

3.2. Potentiometric Assessment of the DSPLM Electrode. Four assemblies of the screen-printed microchip based on organic membrane electrode with the same portions of PVC support, solvent mediator, and anion excluder but with different sensitive material compositions (Table 1) have been microfabricated and electrochemically evaluated as Pb^{2+} microelectrodes according to IUPAC regulations. The potentiometric calibration graphs of the four microchip assemblies (1–4) have been presented in Figure 6. The screen-printed electrode based solely on TiO_2 without MWCNTs (microchip assembly number 1) offers a near Nernstian behavior for Pb^{2+} ions with a sensitivity of 27 mV/concentration decade (Figure 6). Incorporating the MWCNTs to the sensitive element (5% MWCNTs, 95% TiO_2, microchip assembly number 2) significantly enhances

Table 2: Response parameters of the DSPLM electrode (t; 25°C, pH; 5, microchip assembly 2).

Parameter	Microelectrode assembly 2
Slope, mV/decade	49 ± 1
Linear range, mole L^{-1}	10^{-6}–10^{-1}
Detection limit, mole L^{-1}	5×10^{-7}
Response time, s	10
Life span, months	>4
pH range	4–6

the sensitivity of the microelectrode (slope, 49 mV/concentration decade). Screen-printed electrode based on this sensitive material (microchip assembly number 2) provide super Nernstian sensitivity towards Pb^{2+} (Figure 6). The increase in the sensitivity upon addition of carbon nanotube is attributed to the interesting and unique properties of the nanostructured materials induced by the high surface to volume ratio. Such materials provide excellent ion-exchange properties and superior electrical conductivity [12, 36, 39].

However, further increasing of the MWCNTs content in the composite sensitive material (microchip assemblies number 3, 4) gradually decreases the microelectrode sensitivity towards Pb^{2+} ions (Figure 6) due to the decreasing of the TiO_2 sensing element percentage. Screen-printed electrodes containing 10% and 30% MWCNTs (electrode number 3, 4) provide Nernstian response with sensitivity of 31 and 34 mV/concentration decade, respectively (Figure 6). For comparison, the potentiometric calibration graphs of the four assemblies were presented in Figure 6. As can be seen, microelectrode comprises 5% MWCNTs and 95% TiO_2 in its sensitive membrane (microchip assembly number 2) provides highest sensitivity with a slope of 49 mV/concentration decade. Thus, this microchip assembly has been selected for the rest of the evaluation and application experiments. The potentiometric response parameters of the realized microchips assembly number 2 are summarized in Table 2.

The dynamic response time of the microsensor (assembly 2) was assessed by monitoring the time spent to obtain the constant potential after successive dipping the electrode assembly in a series of Pb^{2+} solutions each having a tenfold increase in concentration from 10^{-6}–10^{-1} mole L^{-1}. The results obtained (Figure 7) showed that the nanocomposite based microelectrode provides a very fast response time (10 s) in the linear range. Further, the frequent calibration of the microchip reveal that it shows long life span (>4 months).

The influence of the pH of the test solution on the potentiometric response of the elaborated Pb^{2+} microelectrode (assembly 2) was assessed for two different concentrations (10^{-3} and 10^{-4} mole L^{-1}). Small aliquots of diluted HNO_3 and NaOH were used to vary the pH of the investigated solutions. The potential change of the microchip (mV) was presented versus the pH of the investigated solutions and the data obtained are depicted in Figure 8. The results indicate that, the potential of the microchips is constant and not affected by the pH changes in the pH range from 4 to 6

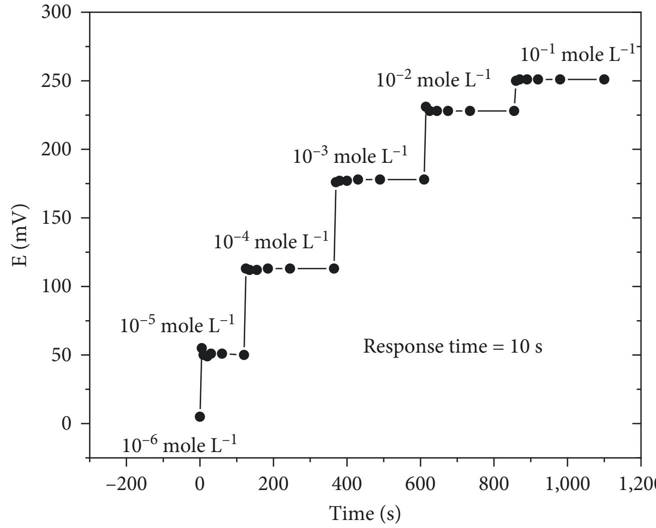

Figure 7: Dynamic potentiometric response of DSPLM electrode (microchip assembly 2, t; 25°C, pH; 5, sensitivity $\pm$ 1).

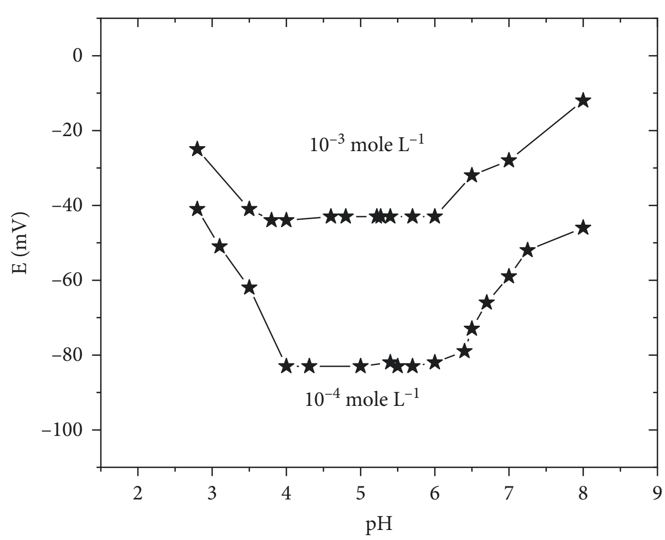

Figure 8: Influence of the pH on the potentiometric response of DSPLM electrode (microchip assembly 2, t; 25°C, sensitivity $\pm$ 1).

and consequently confirm the applicability of the microelectrode in this particular pH range. Thus, the characterization and applications studies were conducted in this pH range. The obtained high potential at lower pH values may be attributed to the interferences of higher concentration of hydronium ion, while the high concentration of hydroxyl ion deteriorates the membrane coat [39].

The utility of the realized microchip (assembly 2) to detect the primary ion in the presence of some interfering ions was tested by successive measurements of the potentiometric response of the microchip in (1×10^{-3} mol L^{-1}) of the primary ion and the tested interfering ions. Consequently, the selectivity was determined by assessing the selectivity coefficient ($K_{A,B}^{pot}$) using the separate solution method (SSM). The results obtained (Table 3) reveal that the microchip provides reasonable selectivity towards Pb^{2+} in the presence of

Table 3: Selectivity coefficient values of the DSPLM electrode (microchip assembly 2, *t*; 25°C, pH; 5, [M]; 1×10^{-3} mol L^{-1}).

Ion	$K_{A,B}^{pot}$
Pb^{2+}	1
Li^{1+}	1.7×10^{-2}
Cr^{3+}	5.2×10^{-7}
NH_4^+	1.1×10^{-2}
Mg^{2+}	1.6×10^{-5}
Cd^{2+}	1.4×10^{-5}
Fe^{3+}	7.9×10^{-6}
Ca^{2+}	1.6×10^{-4}
Na^+	1.6×10^{-2}

Table 4: Determination of Pb^{2+} using the realized DSPLM electrode (microchip assembly 2, *t*; 25°C, pH; 5, RDS; <3)*.

No.	Microchips, ppm	ICP, ppm	Recovery, %
1	0.21	0.21	100
2	1.65	1.65	100
3	20.72	19.76	104.8
4	103.60	100.6	102.9
Average recovery			101.9

*The results are based on three replicate measurements.

the tested monovalent, divalent, and trivalent interfering cations.

3.3. Analytical Application. To investigate the credibility and reliability of the elaborated organic membrane based screen-printed Pb^{2+} microelectrode (assembly 2), the chip was successfully used in the detection of Pb^{2+} in some simulated samples. Such samples were also quantified using ICP-OES for comparison. The data obtained were summarized in Table 4. As seen, the concentration values given by the microfabricated chip indicate a satisfactory good agreement with those obtained with the independent ICP-AES method and the average recovery of the study was 101.9% and the RDS was <3.

4. Conclusions

Microfabrication, potentiometric evaluation, and analytical application of novel organic membrane based screen-printed chip responsive for lead (II) have been demonstrated. The elaborated disposable microchip has been realized, for the first time, using a newly developed nebulization methodology. The fabricated microsensor provides good selectivity, high sensitivity, long life, fast response time, and automation and integration applicability. The microchip has been successfully applied in the quantification of Pb^{2+} with satisfactory accuracy (101.9%) and precision (RDS < 3).

Data Availability

The data used to support the findings of this study are included within the article.

Disclosure

We declare that, the data was registered as a US patent and consequently cited in the manuscript [39] according to the international ethical scientific regulations (https://www.global-engage.com/life-science/publications-and-patents).

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Authors' Contributions

All the authors contributed significantly to this work.

Acknowledgments

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