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Development of poly(3-aminophenylboronic acid) modified graphite rod electrode suitable for fluoride determination

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ABSTRACT

Poly(3-aminophenylboronic acid), (PAPBA) film was formed on the graphite rod surface by potential cycling. The PAPBA-modified graphite rod (PAPBA/GR) electrode prepared in this way was used for potentiometric fluoride determination. The linear calibration range was from 5×10^{-4} to 5×10^{-2} M with the slope of the linear part of the calibration curve of 42.5 mV/log *C*. No interference effect of the most common ions such as sodium, potassium, chloride, nitrate, iodide, calcium, zinc, aluminum, sulfate and sorbitol was observed during electrochemical determination of fluoride. On the other hand, the PAPBA/GR electrode showed not only good sensitivity and selectivity, but also relatively rapid response to changes of analyte concentrations in the range of 20 s. The sensor was successfully applied for fluoride determination in real sample – toothpaste.

1. Introduction

First investigations of ion-selective electrodes had been performed at the beginning of the 19th century [1]. Recently, number of investigations, which are related to the application of ion-selective electrodes, is still increasing since they have number of advantages including good selectivity and sensitivity. Ion-selective electrodes were being used in clinical analysis and in routine control of some ions and biological species especially due to their applicability and selectivity [2,3].

The fluorine, which has the highest oxidation potential among all elements, is mostly found in the form of fluorides. The fluoride and their compounds are mostly used in the synthesis of organic compounds [4], inorganic materials [5,6], drugs [7], enzyme inhibition [8], nuclear fuel reprocessing [9] and polymers [10]. Due to number of applications, the fluoride is getting in contact with the human body, which causes some health problems due to toxicity of soluble fluoride salts at higher concentrations. When the fluoride is ingested together with food, it initially acts locally on the intestinal mucosa, where it forms hydrofluoric acid in the stomach. Thereafter it binds with the calcium and interferes with the activity of various enzymes. Toxicity related effects caused by fluoride ingestion below the lethal limit are dental fluorosis, skeletal fluorosis and rheumatoid arthritis [11–13]. Therefore, fluorine determination is crucial for health purposes. The techniques available for the determination of fluoride in different samples are atomic absorption spectroscopy [14,15], ion chromatography [16], gas chromatography–mass spectrometry [17], capillary zone electrophoresis [18], colorimetric [19,20], fluorometric [21], and ion-selective electrodes [22,23]. Among these techniques, electrochemical determination has been applied more than the others because of its highest sensitivity, selectivity and easy application. As an example, Shinkai et al. have presented the electrochemical determination of fluoride in aqueous media by a redox active ferrocene-boronic acid [24]. Yuchi et al. have reported fluoride determination based on application of napthylboronic acid as an ionophore [25]. Matusevich et al. have prepared miniaturized fluoride selective potentiometric sensor with conductive polymer as an intermediate layer [26]. Gupta et al. have reported electrochemical sensors based on polyvinylchloride membranes for fluoride determination in the presence of several anions as an interfering material [22]. Fluoride ion is involved in the formation of complex with boric acid in aqueous solution and

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it forms fluoroborates of various stoichiometries. It was reported that fluoride ions can also form a tetrahedral phenylfluoroboronat complex with PAPBA [27].

The aim of this work was to prepare a fluoride selective electrode based on the electrochemical polymerization of the 3-aminophenylboronic acid (APBA) monomer on the graphite rod electrode. The selective potentiometric response for fluoride ion using poly(3-aminophenylboronic acid) film electrode displays a nearly Nernstian response, which is suitable for the determination of fluoride ion in real sample – toothpaste.

2. Experimental

2.1. Chemicals

The 3-aminophenylboronic acid was purchased from Aldrich (Steinheim, Germany) and used as received. All commercial chemicals were of analytical grade and were purchased from global suppliers. Stock solutions were prepared with doubly distilled water and kept in the dark. Britton Robinson (BR) buffer solution was prepared by mixing H_3BO_3, CH_3COOH and H_3PO_4. Then the pH was adjusted by addition of NaOH or HCl.

2.2. Instrumentations

All electrochemical measurements were performed using Autolab PGSTAT 30 Potentiostat/Galvanostat (Utrecht, Netherlands) operated by the GPES software Eco Chemie (Utrecht, Netherlands). Graphite rod (GR) electrode (3.0 mm diameter, 99.999%, low density) with the active surface area of 7.0 mm^2 was purchased from Sigma-Aldrich (Berlin) and used as working electrode while Ag/AgCl in saturated KCl ($Ag/AgCl/KCl_{sat}$) as the reference and platinum wire was used as the auxiliary electrode. All electrochemical experiments were performed at room temperature (25 °C).

2.3. Preparation of PAPBA film electrode

Poly(3-aminophenylboronic acid) (PAPBA) film was formed on the carbon electrode surface in the cell containing 0.2 M HCl solution, 50 mM NaCl, and 40 mM APBA by potential scanning from -0.1 to 1.1 V *vs* $Ag/AgCl/KCl_{sat}$ at the scan rate of 100 mV s^{-1} by several cycles. The number of cycles applied to the electrodes permitted the control of the thickness of the film, which had an effect on the electrochemical properties of the surface during applications. For this reason this process was optimized. The films were then undoped by applying -0.1 V potential to the working electrode for one minute in the same solution as used for the synthesis. The electrode was taken from the synthesis cell and then it was rinsed with water. For all synthesized films it is important to finish the last potential scan at the final potential value of -0.1 V. Hence prior to the potentiometric measurement step the film was left in reduced state.

2.4. Potentiometric measurements

Potentiometric measurements were carried out in two-electrode cell configuration. The GR electrode modified by PAPBA film was used as the working electrode, $Ag/AgCl/KCl_{sat}$ was employed as the reference electrode. In order to present an accurate, reproducible, and sensitive analytical method for the determination of fluoride ion concentrations and to determine the effect of different parameters such as polymer film thickness and pH, optimization experiments were performed. Potentiometric signal dependence on fluoride concentration was studied by the PAPBA/GR electrode *vs* $Ag/AgCl/KCl_{sat}$ and potentiometric responses *vs* fluoride concentration were plotted. In addition, the performance of the PAPBA/GR electrode was evaluated by the determination of selectivity, stability and reproducibility. Selectivity of the PAPBA/GR electrode was studied by the determination of fluoride in the presence of sodium, potassium, chloride, nitrate, iodide, calcium, zinc, aluminum, sulfate ions and sorbitol. For this aim, the fixed interference method (FIM) was applied [28,29]. Selectivity coefficients ($K_{F,I}$) were evaluated potentiometrically in solutions containing a fixed concentration (5.3×10^{-4} M) of interfering ions and varying concentrations of fluoride ions. The potential values were evaluated and the results were discussed *vs* the logarithm of the activity of the fluoride ion in the presence of fixed concentration of interfering ions. The intersection of the extrapolated linear portions of this plot indicated the activity of fluoride, which was used for calculation of $K_{F,I}$ from the Nikolsky–Eisenman equation [30]. Activities were calculated according to the Debye–Hückel method.

The stability experiments were performed for one week with the PAPBA/GR electrode in order to evaluate the sensitivity of potential responses of modified electrode for potentiometric determination of fluoride. The repeatability of the proposed electrode was tested with the same PAPBA/GR electrode for three times by successive determination of 5×10^{-3} M of fluoride ions. The application of the modified electrode for the potentiometric determination of fluoride in toothpaste was realized. Toothpaste sample containing sodium fluoride was purchased from market in Vilnius, Lithuania. 0.052 g toothpaste (0.32% fluoride) was weighed and transferred into an electrochemical cell containing 5.0 mL of BR buffer solution, pH 3.0 and then this solution was stirred vigorously for 20 min. In order to perform an analytical application for the PAPBA/GR electrode, the standard addition method was applied.

3. Results and discussion

3.1. Polymer film formation

Typical repeated cyclic voltammograms (CVs) of APBA in 0.2 M HCl including 50 mM NaCl, presented at the scan rate of 100 mV s^{-1} at the GR electrode are presented in Fig. 1. The relative increase in current values of oxidation and reduction peaks indicates the film formation. In addition to this, it was also observed that the oxidation peak potential shifts to a more

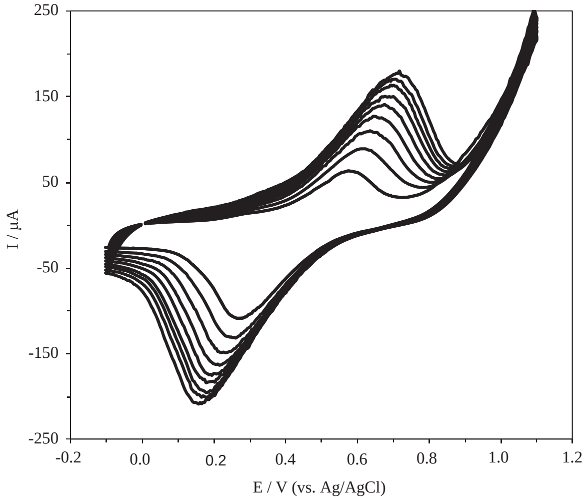


Fig. 1. Cyclic voltammograms registered during the electropolymerization of the APBA in 0.2 M HCl containing 50 mM NaCl and 40 mM APBA at the scan rate of 100 mV s^{-1} *vs* $Ag/AgCl/KCl_{sat}$.

positive value, while the reduction peak potential shifts to a more negative value, which proves the formation of the conducting film.

3.2. Optimization of the potentiometric determination of fluoride ion at the PAPBA/GR electrode

The thickness of the PAPBA film was evaluated by the cycles registered *via* electrochemical polymerization of APBA on the GR electrode (Fig. 2). The best response was registered with the PAPBA/GR electrode modified with PAPBA layer by 40 potential cycles. On the other hand, the signals of PAPBA/GR electrode, which was modified with PAPBA layer by 30 potential cycles, had the similar current values with that of the PAPBA/GR electrode modified by 40 potential cycles even in the lower fluoride concentrations. However its linear concentration range was narrower than that for the electrode formed by 40 potential cycles. For this reason, the electrode modified by 40 potential cycles was selected for further evaluations. The variations in the sensitivity are mainly influenced by different diffusional limitations, different electrochemical capacitances and different resistances of PAPBA layers of different thickness. This conclusion could be supported with one of our previously reported research, which is based on the ellipsometric measurements of polyphenol modified carbon surfaces [31]. In the related study, it was confirmed that the thickness of the film on the surface was dependent on the concentration of the polymerized phenolic structures. Similarly to the related study, in present research, it could be concluded that more homogenous and the thickest polymeric layer was obtained applying 40 potential cycles. It was thought that some areas of electrode could be covered not properly by PAPBA layer and/or pinholes could be formed in this polymeric layer for the PAPBA/GR electrode modified by 30 potential cycles. As mentioned in another study the PAPBA film thickness was important to the sensitivity of modified electrode, which was applied in electrochemical determination iodide [32].On the basis of these examples, it can be summarized that the thickness of the film could have some effects on the electrochemical responses of the modified electrodes.

The influence of pH on the fluoride determination was examined in the pH range from 1.0 to 6.0. The results are presented in Fig. 3. The lowest response was observed at high pH values, which could be influenced by the presence of the negatively charged boronic acid moiety generated during dissociation as shown in Eq. (1) [33]. These negative charges repel the fluoride anions from the GR electrode surface.

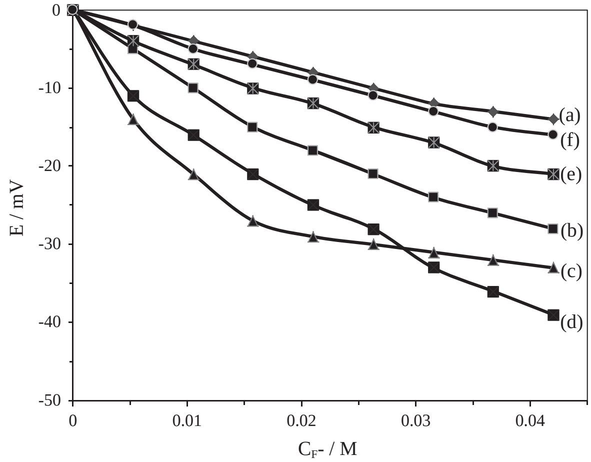


Fig. 2. The effect of the polymer film thickness on the signal registered during fluoride determination: (a) 10, (b) 20, (c) 30, (d) 40, (e) 50, and (f) 60 cycles, pH 4.0.

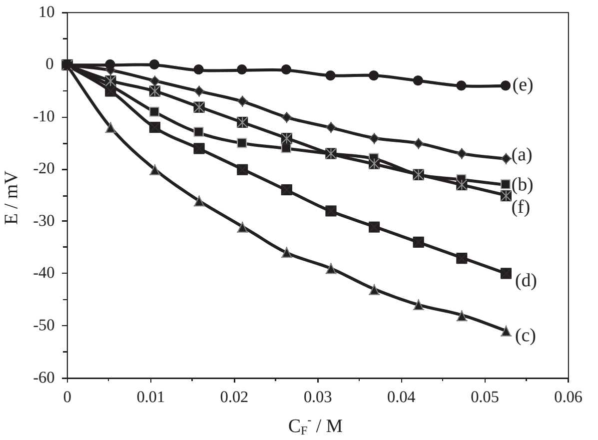
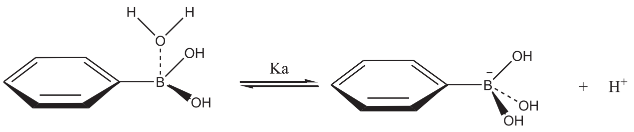


Fig. 3. The effect of the pH on the fluoride determination: (a) pH 1.0, (b) pH 2.0, (c) pH 3.0, (d) pH 4.0, (e) pH 5.0, and (f) pH 6.0, 40 cycles.



(1)

At low pH values, which are characterized by highest concentration of hydrogen ion, fluoride ions are associating with hydrogen ions instead of reaction with boronic acid on the PAPBA/GR electrode surface. Due to the limited HF dissociation into fluoride and hydrogen ions the efficiency of the interaction between PAPBA and fluoride ions is decreasing. During several investigations, it was obtained that the PAPBA electrode showed ‘Nernstian behavior’ at pH 3.0, therefore this pH value was selected as the most optimal for the fluoride determination.

In the present study, formation constant (K_f) of F^-BO was obtained by fitting potentiometric responses *vs* fluoride concentration [34];the formation constant was found as 1.5×10^6 $dm^6\ mol^{-2}$. The formation constant calculated in our research is higher then these reported for phenylboronic acid and naphthylboronic acid, which are 1.01×10^4 and $1.08 \times 10^4\ dm^6\ mol^{-2}$, respectively [35]. On the other hand, in acidic solution, H^+ ions will react with fluoride ions to form HF, which has relatively low ionization constant (7.2×10^{-4}) in dilute solutions. This increase in acid strength can be explained by the increase of dimer concentration with the increase of pH value from 1.5 to 3.0, the organometallic compounds are able to form a covalent bond with the fluoride ion and they also have an excellent selectivity towards fluoride [36].

3.3. Potentiometric measurements at the PAPBA/GR electrode

Electrochemical measurements of the PAPBA/GR electrode were carried out in the range of 5×10^{-5}–5×10^{-2} M of fluoride due to optimal response in this range (Fig. 4). The slope of the linear part of the calibration curve was 42.5 mV/log C ($R^2=0.98$) in 5×10^{-4}–5×10^{-2} M fluoride concentration range. The calibration curve for this electrode is shown in the inset of Fig. 4.

Although the linearity between 5×10^{-4} and 5×10^{-2} M is observed in Fig. 4, the linearity of the curve at lower concentrations than 5×10^{-3} M shows a behavior similar to Nernstian one. During the evaluation of the graph presented in Fig. 5 the slope of the curve is approximately equal to 51 mV/log C (Eq. (2)). Under the experimental conditions determined above, calibration graphs were constructed and the equation for the calibration curve for the

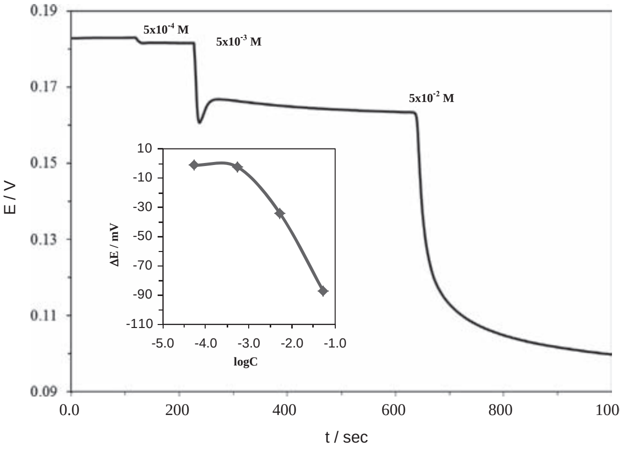


Fig. 4. Potentiogram of the PAPBA/GR electrode *vs* different concentration (5×10^{-2}–5×10^{-4} M) of fluoride. Inset: calibration curve of the PAPBA/GR electrode.

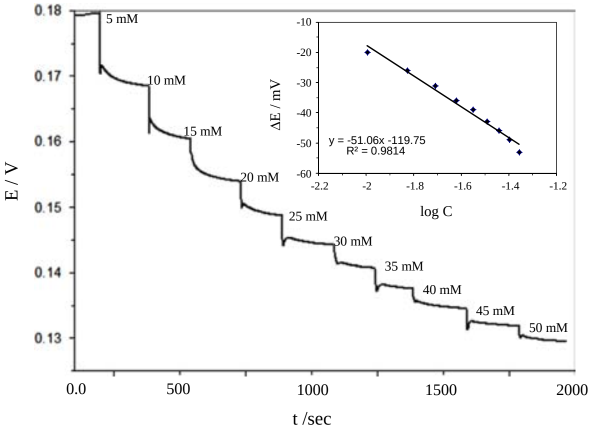


Fig. 5. Potentiogram of the PAPBA/GR electrode *vs* different concentration (5×10^{-2}–5×10^{-3} M) of fluoride. Inset: calibration curve of the PAPBA/GR electrode.

Table 1
Selectivity coefficients for various interfering ions, determined potentiometrically.

Ion	$-\log K_{F^-,j}$
Cl^-	5.97
SO_4^{2-}	6.58
NO_3^-	6.10
Na^+	5.78
K^+	6.09
I^-	5.85
Al^{3+}	3.50
Zn^{2+}	4.10
Ca^{2+}	4.05

Table 2
The comparison with the PAPBA electrode to commercial fluoride electrode.

Added fluoride (M)	PAPBA electrode (M)	RSD% ($n=3$)	Commercial fluoride electrode (M)	RSD% ($n=3$)
5.3×10^{-3}	6.6×10^{-3}	5.5	5.4×10^{-3}	1.5
10.6×10^{-3}	12.1×10^{-3}	3.9	10.8×10^{-3}	1.7
15.9×10^{-3}	15.2×10^{-3}	7.4	16.3×10^{-3}	1.6

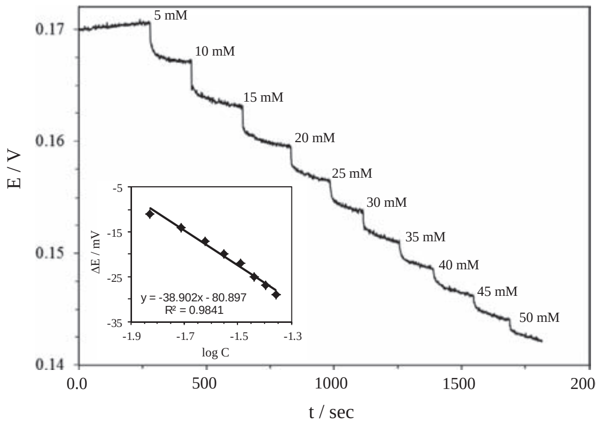


Fig. 6. (a) Potentiogram of the PAPBA/GR electrode recorded for toothpaste containing matrix. (b) Calibration curve of the PAPBA/GR electrode for toothpaste.

determination of fluoride ion was found as follows :

$$E(\text{mV}) = -51.06\log C - 119.75 (R^2 = 0.981) \tag{2}$$

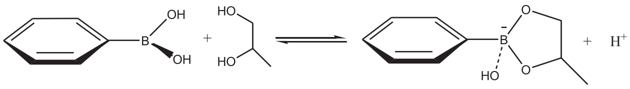
Time required for the potential to reach a constant value is determined as 'response time'. The response time of the sensor based on electrode, which was modified by 40 potential cycles, was 20 s. The lifetime of the electrode exceeded one week, during this period no measurable divergence was observed. The repeatability of the method was evaluated by successive determination of 5×10^{-3} M of fluoride ions and the relative standard deviation (RSD) was calculated as 10.8% ($n=3$).

3.4. *Selectivity of the PAPBA/GR electrode*

The performance of the PAPBA/GR electrode was investigated in the presence of interfering ions. For this aim, the fixed interference method (FIM) was applied [29,30]. The calculated $K_{F^-,j}$ values were small (in the range of 10^{-4}–10^{-7}). Hence the electrode displayed excellent selectivity towards fluoride ion in comparison with other tested ions as shown in Table 1.

The high affinity for fluoride binding by boronic acids was the basis of many selective methods used for quantitative analysis of fluoride anions. The boronic acids are trivalent boron containing organic compounds in which the sp^2 hybridized boron atom possesses a vacant p orbital. Therefore they can act as Lewis acids and form complexes with strong bases like F^- or OH^- [37]. Hence the interaction between boronic acid and ions, which are interfering the fluoride ion determination, is insignificant. Due to advanced selectivity the PAPBA/GR electrode was identified as the selective electrode for fluoride ions. Although we also used PAPBA electrode for iodide determination at pH 1.5 in our previous work [32], but recent study determined that iodide is not interfering the determination of fluoride at pH 3.0.

As known from the literature [38], the complexity of diol groups with aromatic boronic acids produces a stable ester (Eq. (3)). This reaction occurs in the range of pH from 6 to 10. Sorbitol, which contains diol groups, in the toothpaste did not show interference effect at low pH. Some ions such as sodium, potassium, chloride, nitrate, iodide, calcium, zinc, aluminum, and sulfate were also studied and no interference by these ions was observed during electrochemical determination of fluoride even in real sample such as toothpaste.



(3)

To compare PAPBA-modified electrode with commercial fluoride-selective electrode, different concentrations of fluoride were

Table 3
Comparison of PAPBA sensor with other fluoride sensors.

Electrode	Method	Concentration range (M)	LOD (M)	pH	Response time	References
PVC-based membrane	Potentiometric	1×10^{-2}–1×10^{-7}	1.3×10^{-7}	4.0	11 s	[39]
(Al(III)-TPP) /polymethacrylate polymeric membrane	Potentiometric	1×10^{-1}–1×10^{-7}	1×10^{-5}	3.0	–	[40]
GC/polycarbazole–BF3	CV	1×10^{-3}–4×10^{-4}	–		–	[41]
GC	DPV	5.3×10^{-5}–5.3×10^{-6}	3.16×10^{-6}		–	[42]
A plant tissue (asparagus)-based Carbon Paste	CV	0–1×10^{-3}	2.6×10^{-5}	5.0	60 s	[43]
MPBA/Au	CV	1×10^{-2}–1×10^{-8}	–	4.0	–	[44]
n-DCPD/GCE	CV	2×10^{-5}–5×10^{-7}	1.4×10^{-7}		–	[45]
n-HAP/GCE		5×10^{-5}–1×10^{-7}	6.9×10^{-8}			
LaF3	Potentiometric	5.3×10^{-2}–5.3×10^{-5}	5.3×10^{-8}	5.0–5.5	–	[46]
Commercial electrode	Potentiometric	–	5.3×10^{-7}	5.0–5.5	–	[47]
PAPBA	Potentiometric	5.0×10^{-2}–5.0×10^{-4}		3.0	20 s	Present study

PVC, polyvinyl chloride; Al(III)–TPP, Al(III)-tetraphenyl porphyrins; GC/polycarbazole–BF3, glassy carbon/ polycarbazole–boron; ISE, ion-selective electrode; MPBA/Au, 4-mercaptophenylboronicacid/Gold; n-DCPD/GCE, dicalcium phosphate dihydrate /glassy carbon; n-HAP/GCE, hydroxyapatite/ glassy carbon; CV, cyclic voltammetry; DPV, differential pulse voltammetry.

spiked into water (Table 2). The compatibility between the two electrodes proves the reliability of PAPBA-modified electrode for the determination of fluoride in water samples.

3.5. *The determination of fluoride ions in toothpaste*

In order to evaluate the application for PAPBA/GR electrode, the concentration of fluoride in the toothpaste was determined using the 'standard addition method' (Fig. 6). The concentration of fluoride ion in toothpaste was calculated as 1.8 ± 0.1 mM and it was found as 2.2 ± 0.2 mM ($n=3$) in toothpaste. There was the deviation of about 20% between calculated and detected fluoride concentrations. These results indicated that the PAPBA/GR electrode can be applied to the determination of fluoride ions in real sample matrix.

The determination of fluoride ion has also been studied by other researchers. A number of different modified electrodes and different detection techniques were applied for this determination (Table 3). We believe that PAPBA-modified electrode will be an alternative for fluoride determination with a rapid response time and easy preparation.

4. Conclusion

A new potentiometric sensor for fluoride determination was designed. This sensor enables the determination of fluoride ion in concentration range of 5×10^{-5}–5×10^{-2} M even in the presence of some interferences such as sodium, potassium, chloride, nitrate, iodide, calcium, zinc, aluminum, sulfate ions and sorbitol. The slope of the linear range, which was observed within 5×10^{-3} and 5×10^{-2} M of iodine was 51 mV/log C. The PAPBA/GR electrode was characterized by advanced sensitivity, selectivity and repeatability. The most remarkable advantages of the method proposed in this study are: single step electrode preparation, direct measurement of fluoride ion and easy handling when comparing to other methods such as atomic absorption spectroscopy, chromatography and capillary electrophoresis. Moreover, there is no interference effect by most common ions. The proposed sensor was also successfully applied for the determination of fluoride ions in real sample – toothpaste.

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