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Analysis of fluoride content in salmon using a fluoride-selective electrode

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Abstract: Fluoride is a valuable element for dental health. Therefore, trace fluoride analysis is particularly important. A fluoride-selective electrode has been developed to determine fluoride levels. This electrode was prepared using a fluoride-containing ionophore and sparingly soluble sulfur salts in specific ratios. This electrode showed an analytical slope of approximately 30 mV for fluoride concentrations. Factors affecting the sensitivity of this electrode were investigated to determine optimum conditions. Fluoride analysis was performed on two different salmon samples from the Agricultural Credit Cooperative store. The fluoride levels in the same samples were measured using an ion chromatography device. The results obtained were found to be consistent.

Keywords: Salmon, fluoride, selective electrode, potentiometry.

Introduction

Fluoride is an important element in maintaining dental health. Dental health deteriorates in lack or excess of fluoride. The change in the amount of fluoride is mostly due to the water and food consumed. It has been determined that the amount of fluoride changes as a result of environmental pollution of water and chemicals used in food production, and therefore dental health deteriorates [1-5].

When fluorine is dissolved in water, levels down to 0.5 mg L^{-1} may be toxic to fish, in particular those living in "soft waters". The toxicity of fluorine decreases with increasing hardness ($CaCO_3$ mg L^{-1}) and increasing chlorine concentration, and decreasing temperature [6]. Feeding trials carried out with fish in fresh or brackish water using krill as feedstuff increases bone fluorine content [7-9]. However, a recent study where adult Atlantic salmon were fed krill diets in seawater environments found no increase in bone fluorine in spite of krill replacing up to 30% of fish meal [10].

Determining the amount of fluoride also becomes extremely important. Fluoride determination was made by many methods. These methods include FT-IR spectroscopy [11], Raman spectroscopy [12], ion chromatography [13] and ICP-OES methods [14]. Analyzes performed with these methods are expensive and have many interference effects. In this study, analysis of the amount of fluoride was performed using a fluoride selective electrode. Analytical performance values of the electrode were measured and its sensitivity was determined to be high. The amounts of fluoride in 2 different salmon samples were determined with a fluoride selective electrode.

Materials and methods

Materials and Reagents

Calcium fluoride (Sigma), used as an ionophore, Ag(I)S and CuS were commercially obtained. Anion and cation solutions (Merck) were obtained from solid salts. All solutions were prepared using twice purified water.

Instruments

An external Ag/AgCl reference electrode with a Jenway 3040 pH/ion meter was used for potential measurement. Potential values were measured at room temperature.

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Preparation of fluoride selective electrode

A certain amount of the salt or salt mixture of Ag(I)S, CuS and CaF_2 (10–15 mg) was taken and pellets were made by holding first under a pressure of 1940 atm for 1min, then pressure was increased to 7760 atm by adding each time 1940 atm for 1min. The pellets of 7mmdiameter were sealed with epoxy resin (0.7 g epoxy and 0.9 ghardener). To obtain a good sealing the epoxy resin on the edge of the tubing had to wait for about 10 min and then the pellet was sealed. One day after the pellet was sealed a silver wire is connected. For this purpose, the inside of the glass tube was filled to about 1 cm with a mixture of 0.5 g graphite powder and epoxy resin. The electrode prepared had to wait for about 2 days so that the resin can get dry. The surface of the electrode is washed and then polished with a soft paper [15].

Preparation of salmon samples

Two salmon samples were taken from the agricultural credit cooperative store. The salmon samples were cut into small pieces. They were incubated in 5 ml of TISAB II (Total Ionic Strength Adjustment Buffer) for 24 hours. The amount of fluoride that passed into the solution was measured using an electrode.

Results and discussion

Sensitivity of fluoride electrode

The sensitivity of electrode prepared for fluoride ion was measured. For this purpose, in the presence of 0.1 M $NaNO_3$ solution, the fluoride concentration in the solution was increased by 10 times from $1x10^{-6}$ M to 0.1 M fluoride concentration and the changes in the potential were recorded. At this point, the slope of the electrode was calculated as 30 ± 0.1 mV. Electrode sensitivity is given in figure 1.

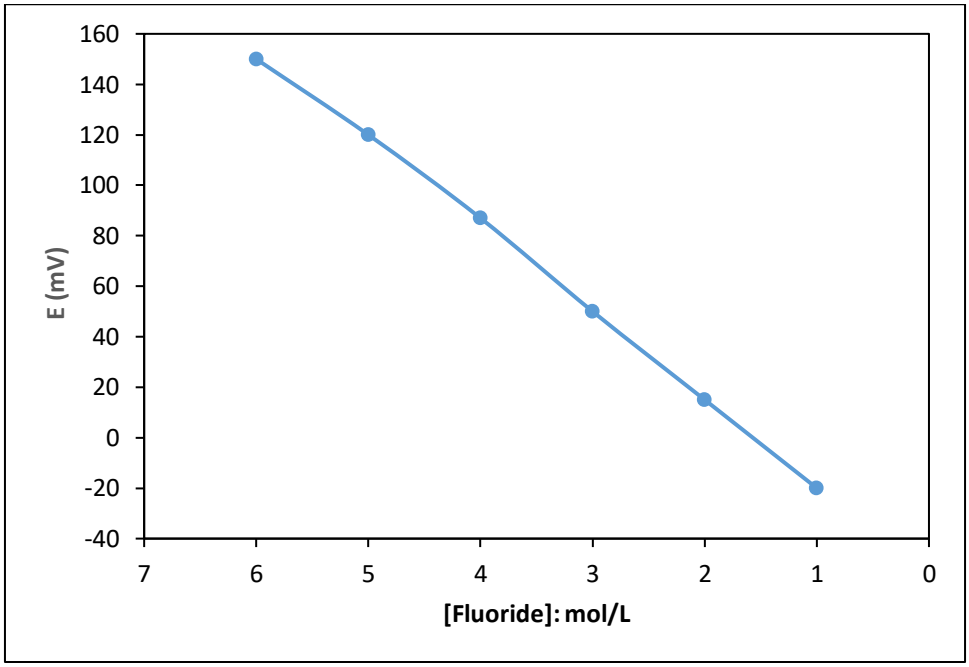

Figure 1. Calibration chart of fluoride electrode

Analytical performance of fluoride electrode

Samples containing known amounts of fluoride, which are necessary to determine the amount of fluoride that may be present in the tooth sample, were analysed with the fluoride selective electrode we prepared. Results were calculated as the mean of 4 experiments based on a 95% confidence interval. Analytical performance values of the electrode are given in Table 1.

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Table 1. Analytical performance values of fluoride electrode

Amount of Fluoride added: µg/L	Measurement $\bar{x} \pm ts / \sqrt{N}$	Recovery, %
10	9,1 ± 0,1	91
25	24,2 ± 0,3	97
50	48,5 ± 0,7	97
100	98,6 ± 4,5	99

(CI 95%, N=4 $\bar{x}$: Arithmetic mean, s: Standard deviation, t: Student's t-test)

Determination of fluoride in salmon samples

Fluoride content in two different freshwater salmon samples was measured using both our specially designed fluoride-selective electrode and ion chromatography in our central laboratory. The results, as the average of four experiments with a 95% confidence interval, are presented in Table 2.

Table 2. Comparison of the amount of fluoride in salmon samples with both methods

Salmon samples	Electrode with found [Fluoride]: µg/L	Ion Chromatography with found [Fluoride]: µg/L
1	10 ± 2	9
2	35 ± 0,2	36

(CI 95%, N=4 $\bar{x}$: Arithmetic mean, s: Standard deviation, t: Student's t-test)

Conclusion

It was measured that the fluoride selective electrode, using calcium fluoride as the ionophore and prepared from slightly soluble sulfur salts, was sensitive to fluoride concentration with a slope of approximately 30 mV. It was determined that the analytical performance of the electrode was high against synthetically added fluoride concentrations. Especially the % recovery rates of 97-99% confirm this. Then, 2 different salmon samples were kept in TISAB II solution for 24 hours and fluoride analyzes were measured both by electrode and ion chromatography. It was determined that the results were compatible with each other. Using fluoride electrodes in salmon samples was found to be extremely practical and easy to analyze.

Acknowledgments

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Conflict of Interest

The authors declare that they have no competing interests.

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