



Evaluation of total soluble fluoride concentration in some selected toothpastes in Nigeria

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Abstract

Dental caries remains the most prevalent chronic disease of kids in the world. Fluoride has shown effectiveness in the prevention of caries and giving maximum protection against dental caries while reducing the likelihood of enamel fluorosis. Fluoride is most effective in dental caries prevention when a low level of fluoride is constantly maintained in the oral cavity. Meanwhile, there are some undesirable side effects of too much fluoride exposure. This research involves the determination of the total soluble fluoride concentration of five brands of fluoridated toothpastes using ion-selective electrode (ISE). The different brand of adult toothpastes was purchased from local market/shops in Anyigba, Kogi State. Four of the samples contain sodium fluoride as active ingredient while one of them contains sodium monofluorophosphate. The total soluble fluoride content found for the various samples are as follows; sample A and B 0.066%; C: 0.05%; D: 0.138%; and sample E: 0.101% which are approximately 0.1 % (1000ppm) and 0.14%(1400ppm). These results fell within the standard given by the World Health Organization (WHO) and other agencies.

Keywords: Fluoride, fluorosis, monofluorophosphate, ion-selective electrode, total soluble fluoride

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1. Introduction

Fluoride in many forms is the most prevalent active ingredient in toothpaste to prevent cavities. Fluoride is found in small amounts in plants, animals, and some natural water sources. The additional fluoride in toothpaste has beneficial effects on the formation of dental enamel and bones. Sodium fluoride (NaF) is the most common source of fluoride, but stannous fluoride (SnF_2), olaflur (an organic salt of fluoride), and sodium monofluorophosphate ($\text{Na}_2\text{PO}_3\text{F}$) are also used. Stannous fluoride has demonstrated more effective than sodium fluoride reduces the incidence of dental caries [1] and controlling gingivitis, but causes somewhat more surface stains [2]. Also, the relationship between fluoride and prevention of caries has been analyzed broadly in the previously writings [3].

Recent reports in dental literature shows a decline in the dental caries trend in most developed nations and this is mainly attributable to the use of fluorides in different forms [4]. However, the problem of dental caries still exists as disease of high propensity in many under developed and developing countries of Africa and Asia due to lack of public awareness and motivation, inadequate resources for dental treatments and changing dietary habits [5]. Indeed, the use of fluorides in different forms is still recognized as one of the most successful measures for caries prevention [6]

Fluoride toothpaste is the most important form of fluorides used globally and the most rigorously evaluated vehicle for fluoride use. Since 1940, over 100 clinical trials have assessed the effectiveness of fluoride toothpaste and these trials have confirmed and anti-caries efficacy of fluoride toothpaste. Irrespective of low, normal or high fluoride exposure from other sources, fluoride toothpaste is safe to use [7]. Fluoride is commonly incorporated in toothpastes, gels, and mouthwashes for the prevention of caries [8]

The fluoride compounds present in toothpaste such as NaF, NH_4F and SnF_2 are not compatible with abrasives that contain calcium. By contrast, toothpaste which contains sodium monoflourophosphate, the PO_3F^{2-} component has greater capability with calcium containing abrasives. This is because the fluoride which is present in PO_3F^{2-} is firmly bound to the phosphate and cannot bind to the soluble calcium to form insoluble calcium fluoride [9]. However, the analysis of total and soluble fluoride concentrations various brands of children's toothpastes available in Saudi Arabia indicated variations from the labelled claims of the manufacturers in various brands. Therefore, some of the toothpastes may have doubtful anti-caries effectiveness owing to deficiency of total and soluble [10]. The bioavailability of fluoride in toothpaste depends on the types fluoride compound and abrasive present in the formulation [11].

A recent systematic review of literature evidenced that only toothpaste with 1000 ppm F or more would have anti-caries effect. This has increased the relevance of the requirement that toothpaste should have

soluble fluoride in their formulations. It is therefore necessary to assess the concentration of total soluble or free fluoride in toothpaste in order to evaluate the quality and stability of the toothpaste [12]. Basically, the objective of this research is to ascertain the level of fluoride in toothpaste sold in Nigeria markets.

2. Materials and methods

2.1. Apparatus and Reagents.

In order to carry out the experiment, the following reagents and materials were used:

Apparatus

pH meter with fluoride specific ion electrode system, Stirring rod, Timer, Measuring cylinder, Transfer pipette filler, Digital weighing balance, Filter paper, Funnel, 120mls plastic bottles, Hot plate, Oven, Beakers, Volumetric flasks.

Reagents

Sodium fluoride, Potassium chloride, Distilled water, Total ionic strength adjustment buffer (TISAB) solution.

2.2. Sample Collection

Five different brands of adult toothpaste were purchased from a commercial store in Anyigba, Dekina local government, Kogi state. All the toothpaste samples were checked for information provided on the packages. The information on the packages were checked for descriptive names of the fluoride components and its concentration in parts per million (ppm). Descriptive names of abrasives on the packages were recorded. In toothpastes in which both silica and calcium-containing abrasives were declared, calcium abrasives were recorded.

2.3. Preparation of the Samples for Analysis

A toothpaste sample was obtained using a clean, dry and pre-weighed 20ml beaker to which 0.2-0.3g toothpaste was added. A toothpaste sample was prepared as follows:

The beaker plus toothpaste sample was weighed to determine the net weight of the toothpaste sample. The sample was then transferred to a 50ml beaker with a micro spatula or stirring rod and 50ml 0.1M KCl was added. The solution was then boiled gently for 2-3 minutes and then suspension-filtered by gravity filtration using a #1 or #5 filter. After cooling, the solution was transferred quantitatively to a 100ml volumetric flask and made up to volume with 0.1M KCl.

An unknown NaF sample was obtained using a clean 10ml graduating cylinder. A 7.00-10.0ml sample of unknown solution of F⁻ was also supplied. A 5.0ml of the solution was transferred accurately (with a 5ml transfer pipette) and diluted to 100ml in a volumetric flask using the 0.1M KCl.

2.4. Determination of the fluoride concentration

The fluoride concentration in various brands of toothpaste, ion-selective electrodes (ISEs) method was used. The optimal pH range of the electrode is at pH 5.0.

750 ml of 0.10M of KCl was prepared which served the same purpose as the TISAB solution. All dilutions were made using 0.1 M KCl solutions.

2.5. Preparation of Calibration Solution:

A 100ml stock solution containing 1000ppm F⁻ was prepared using reagent grade sodium fluoride dried for 1 hour in the oven. The weighed sodium fluoride (NaF) was dissolved in a 100ml beaker; 50ml of 0.1M KCl was added. The solution was then transferred to a 100ml volumetric flask and filled to the mark with 0.1M KCl and mixed very well. From the stock solution, a 20 ppm F⁻ calibrating solution was prepared. From this 20 ppm F⁻ calibrating solution, by serial dilution, 10, 5, 2.5, and 1.25 ppm calibrating fluoride solutions were prepared by taking 50ml of the prior solution and diluting to 100ml with 0.1M KCl in a 100ml volumetric flask.

2.6. Measurement of F⁻

The measuring system consists of the fluoride and reference electrodes and a pH meter in mV (millivolt) mode.

Approximately 20ml of each solution was transferred into 50ml beakers. The solution was well stirred and the observed emf (in mV) recorded three minutes after the fluoride and reference electrodes were immersed in the solution. This was repeated for all calibrating solutions as well as the sample solutions.

Analysis

A graph of observed emf in mV vs. logarithm of ppm F⁻ was plotted to obtain a calibration curve (Figure 1). The slope of the curve was obtained and used to determine the concentration of total soluble fluoride present in the toothpaste samples using the formula:

$$\%F^- = \frac{(\text{ppm } F^- \text{ from the graph})(0.100L)}{(\text{Mg toothpaste used})} \times 100 \quad (1)$$

Where:

ppm F⁻= part per million of fluoride

Mg= Milligrams of toothpaste

3. Results and discussion

3.1 Composition of Ingredient in Toothpaste

The results on the table 1 indicate the ingredient from the label on each toothpaste samples gotten from the market.

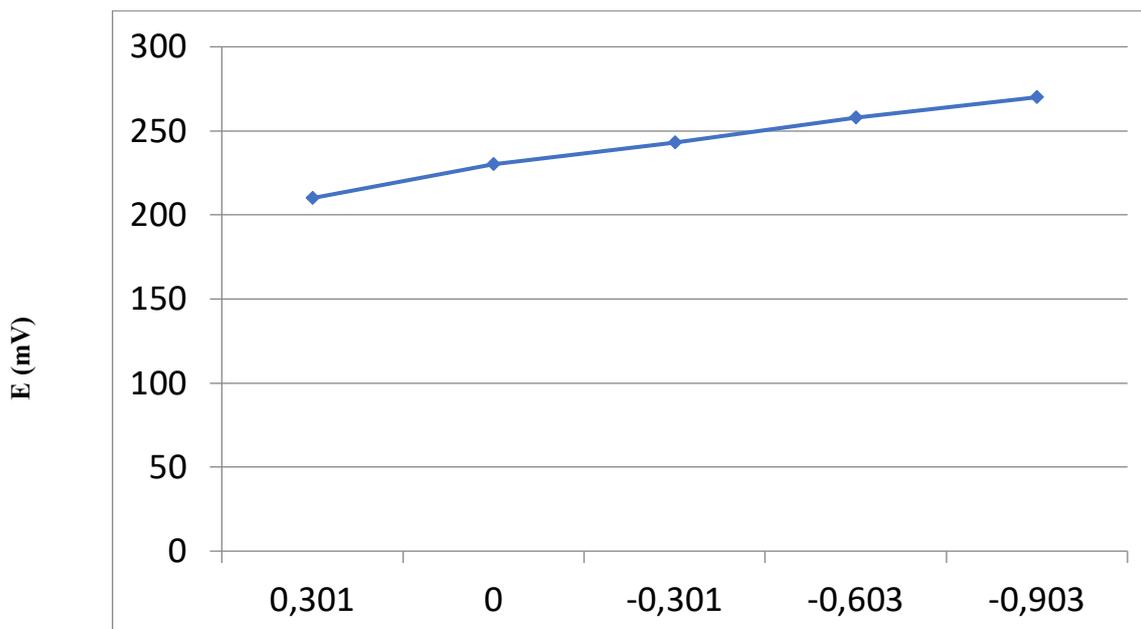


Fig1: plot of observed Emf (mV) against Log ppm F⁻

Log ppm F⁻

Table 1: Toothpaste Composition

Sample	Specified F ⁻ Content (w/w)	Fluoride Source	Ingredients
A	0.145 (%)	Na	sorbitol, aqua, hydrated silica, sodium lauryl sulphate, PEG-32 Aroma, cellulose gum, sodium
B	0.11 (%)	NaF	Sorbitol, aqua, hydrated silica, Sodium Lauryl sulphate, aroma, cellulose gum, tri-Sodium phosphate, sodium phosphate, Sodium saccharin, carbomer, polyethylene, Limonene, CI 77891, CI 42090
C	0.306 (%)	NaF	Sorbitol, aqua, hydrated silica, Sodium Lauryl sulfate, glycerin, PEG-6, flavour, Xanthan gum, sodium saccharin, CI 73360, CI 74160.
D	0.76 (%)	Na ₂ FPO ₃	Sorbitol, hydrated silica, DM water, PEG-1500, Cellulose gum, sodium benzoate, flavour, sodium saccharin, CI 16255
E	0.145(%)	NaF	Sorbitol, aqua, hydrated silica, sodium lauryl sulphate, PEG-32, aroma, cellulose gum, sodium saccharin, Calcium gluconate, CI 19140, CI 42090

Standard solution: 0.1M KCL solution

Stock solution: 2g NaF solution.

3.2. Composition of Fluoride in Toothpaste Samples

The following were obtained as total fluoride in sample analysed. From the ISE experiment carried out, the total soluble fluoride ion concentration of the various toothpaste samples was analysed and calculated. The results of the fluoride analysis of the five toothpastes are presented in the [Table 2](#). The evaluated toothpastes have various types of fluorides and abrasives. Among the five (5) toothpastes evaluated, there were four toothpastes only with NaF, and one contains Na₂FPO₃, as the active ingredient respectively ([Table 1](#)). However, this research tallies with the work of Monica (2020), that the that TSF concentration that is chemically available in toothpastes could be used to estimate how much fluoride would be bio available during brushing [13-16].

Table 2: Measured Fluoride Content in samples

Sample	F ⁻ Content Found (w/w) IN (%)	Sample (mg)	Volume of solution (ml)
A	0.066	250	100
B	0.05	250	100
C	0.138	250	100
D	1.101	250	100
E	0.066	250	100

Table 3: Fluoride in Toothpaste Composition Specification on Sample Package

Toothpaste Sample	Concentration in (ppm)	Concentration in (%)	Fluoride Source
A	1450	0.45	NaF
B	1100	0.11	NaF
C	3060	0.306	NaF
D	7600	0.76	Na ₂ FPO ₃
E	1450	0.145	NaF

The five toothpaste samples analysed fell within the standard specified value according to world Health Organization (WHO), which is between the ranges of 1000-1500 ppm.

The value of the total soluble fluoride obtained is given in [Tables 2](#). From these results, for example, the total soluble fluoride present in sample A and sample E toothpaste is 0.1% equivalent to 1000ppm which is within the recommended range but below the specified fluoride content by the manufacturer while

sample B was discovered to have 0.1% (1000ppm). Sample C had 0.14% (1400ppm) fluoride content. Therefore, the five samples of toothpastes analysed have fluoride content within the recommended values, although the values were slightly different from the specified fluoride content by the various manufacturers (Tables 2 and 3) this also in agreement with MbarakaNdoile (2020) findings in toothpaste analyzed in Tanzania.

3.3. Calibration Curve Readings

The Readings obtained for serial standard and samples are given below. The calibration curve was obtained using the values in column three (3) and four (4) of Tables 4. From the curve obtained, a slope 60.0 was obtained in relation to the value $-59 \pm 4\text{mV}$ which indicates normal curve.

The fluoride ion concentration in the toothpaste expressed as % F⁻ was determined using the equation below and the value 0.12% (1200ppm) was obtained.

Percentage (%) fluoride:

$$\%F = \frac{(\text{ppm } F^- \text{ from the graph}) (0.100L)}{\text{Mg of toothpaste used}} \times 100 \quad (2)$$

Where:

ppm F⁻= part per million of fluoride

Mg= Milligrams of toothpaste

Tables 4: Serial Standard for Calibration Curve

S/N		Volume of Working Standard Solution (ml)	Diluted to Total Volume (ml)	standard of F ⁻ Concentration (mg/L)
1		20	100	2.0
2		10	100	1.00
3		5.0	100	0.500
4		2.50	100	0.250
5		1.25	100	0.125

Tables 5: Calibration Curve Measurements

Series of Standard	F ⁻ (Mg/L)	Log [F ⁻]	E/mV
2.0	2.000	0.301	210
1.0	1.000	0.000	230
0.5	0.500	-0.300	243
0.25	0.250	-0.603	258
0.125	0.125	-0.903	270

Conclusion

As a whole, fluoride is one of the most beneficial micro nutrients for our body but in excess it can be hazardous to our health in many ways. Therefore, to get the beneficial effects of fluoride, people must be careful before using highly fluoridated toothpastes and other sources. Since young infants and children under age two years can swallow most of the toothpaste when brushing, parents should have to be careful.

From the result of the research carried out, it was found that the five samples of toothpastes analysed met the required concentrations of fluoride as recommended by WHO.

Fluoride is most effective in dental caries prevention when a low level of fluoride is constantly maintained in the oral cavity. Meanwhile, there are some undesirable side effects of excessive fluoride exposure.

Government should monitor fluoride concentration in different sources of drinking water and ground water and thorough fluoride map should be available to the public.

To ensure whether people need fluoride supplements or not, government should include the International/WHO guidelines in a circular form to prevent health problems due to deficiency or excess fluoride exposure. The guidelines must deliver an evidence-based summary of current research and fact to enlighten best practices in the use of fluoride containing materials for the safety and security of public health. In addition with several international guidelines and recommendations on the usage of fluoride toothpaste, we recommend the use of toothpaste containing 1000 ppm or more fluoride in children for caries prevention [17-19].

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