Original Article

Assessment of Available and Stable Fluoride in Four Widely-Used Toothpastes in the Iranian Market

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Abstract

Objective: Presence of available and stable fluoride in a dentifrice formulation is a major requirement for an anti-caries effect. Although the available fluoride concentration in Iranian dentifrices has been reported in previous studies, there is little information on its stability; which is dependent upon dentifrice formulation. This study was done to assess the fluoride ion concentration and stability in four widely used dentifrices in Iran.

Materials and Methods: In this analytical study, three samples of each brand of dentifrice (Nasim, Pooneh, Crest, and Signal) were purchased. Total fluoride (TF) and total soluble fluoride (TSF) concentrations were determined by ion specific electrodes. Data about TF were analyzed by one-way analysis of variance (ANOVA). Kruskal-Wallis and Mann-Whitney tests were used for nonparametric data (TSF).

Results: All dentifrices had more than 1000 ppm of fluoride ions. TSF in Crest was significantly higher than in other dentifrices ($P<0.0001$) and was over the maximum permitted dose.

Conclusion: The TF concentration in Iranian toothpastes was sufficient to prevent caries.

Key Words: Dentifrice; Fluoride; Dental Caries

Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran (2014; Vol. 11, No. 5)

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Received: 12 March 2014
Accepted :2 August 2014

INTRODUCTION

The effect of fluoride (F) in caries prevention is well known. F-dentifrices are the most widely- accepted anti-caries substances [1]. It has been reported that F-containing toothpaste is the main factor responsible for caries reduction in developed countries [2, 3]. Thus, F-containing dentifrices are suggested for all populations [4].

Solubility of F in its ionic (free) form (F- of NaF) or ionizable form (MFP) ensures its bioavailability and abundance in the mouth. This organized and bioavailable active form of F guarantees anti-caries activity [5, 6].

In order to have a high bioavailability, chemical constituents, type of F and type of abrasive substances in the dentifrice are important [7]. F-dentifrices contain sodium F (NaF), mono fluoro-phosphate (MFP) or their combination. The abrasive substances i.e. aluminum (Al) and Ca ions can reduce the amount of fluoride in the presence of sodium fluoride (NaF) [8]. In MFP molecule, F is conjugated with phosphate by a covalent bond; however, this bond is not stable and can release F ions; which react with Ca ions in Ca-based dentifrices [9]. This reaction results in production of CaF_2; which is insoluble and does not have remineralization [10] or anti-caries effects [11]. It has been proven that in MFP-containing dentifrices, linking of free F ions with Ca-based abrasives can inactivate F ions [8]. Thus, use of insoluble silicon dioxide (SiO2) or heated pyrophosphate as abrasives has been proposed [12]. In order to have an anti-caries effect, a dentifrice should contain at least 1000 ppm of bioavailable F [13, 14]. The minimal amount of free F ions in a dentifrice should not be less than 60% of its total F content [10-15], and this amount should not exceed 1500 ppm [16].

Since a comprehensive study about fluoride availability and stability in widely used dentifrices in Iran is not available, the aim of the present study was to evaluate the available F concentration in 4 widely used dentifrices; which contain different types of fluoride compounds and abrasives.

MATERIALS AND METHODS

Study design: In this analytical study, TF and TSF concentrations were evaluated in four widely-used adult dentifrices in Iran. Table 1 shows the assessment data of the dentifrices. Three dentifrices of each brand were obtained and 2 cm was extracted from each tube. Then each tube was divided into 3 equal parts (top, middle and down) and each part was stored in a plastic box. In order to carry out a blind analysis, a 3-letter code was randomly allocated to each sample. The TF and TSF concentrations were measured under equal laboratory conditions (temperature 20º C). An ion specific electrode was used for all analyses.

Determination of TF and TSF concentrations: The concentration of TF and TSF was determined as explained in previous studies [17].

Table1. Data relevant to the toothpastes studied

Product Name	Manufacturer	Production Date	Expiration Date	Tube Weight	Abrasive	F formulation	Amount of F Declared on Label
Pooneh (3colored)	Paksan-Iran	2011.11	2013.11	80± 1gr	Di hydrated di calcium phosphate	MFP*	1450 ppm
Signal (whitening)	Unilever-India	2011.8	2013.11	76/5gr	Calcium glycerol phosphate -calcium carbonate - hydrated silica	SMFP**	1450 ppm
Crest (3D)	Gross Germ-Germany	2011.12	2014.12	98gr	Hydrated silica	NaF***	1450 ppm
Nasim (conventional)	Paksan-Iran	2012.1	2014.1	80± 1gr	Di calcium phosphate-di hydrated silica	MFP	1450 ppm

*MFP=monofluorophosphate, **SMFP=sodium monofluorophosphate. ***NaF= sodium fluoride

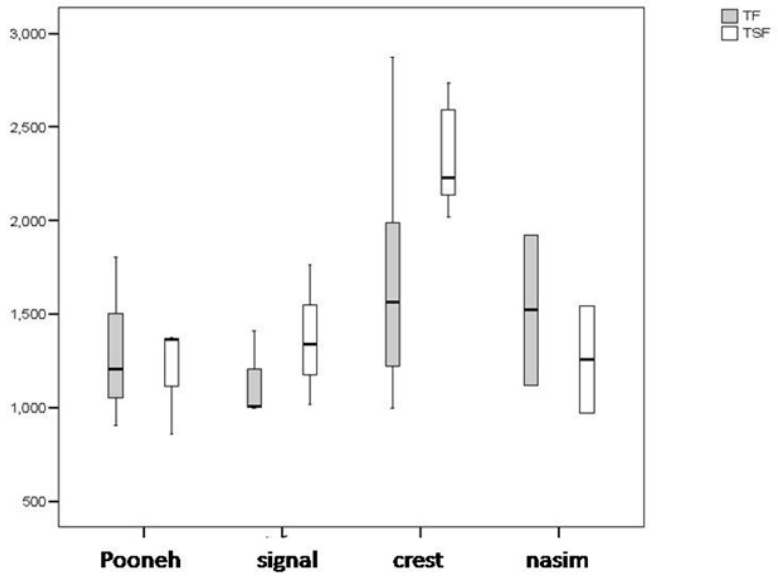

Fig 1. TF and TSF (mean and SD) concentrations in the four evaluated dentifrices (ppm)

Four grams of deionized water (DDW) was used to homogenize 1 gram (±0.01) of dentifrice; and for determination of TSF concentration 0.1 gram (±0.01) of each dentifrice was weighed in scaled plastic centrifuge tubes. All tubes were homogenized in 0.9 g of DDW. After 20 seconds of shaking, a suspension of homogenous solution was produced. In the next step the suspension was centrifuged (3000×g, 10 min, room temperature) and the conjugated fluoride was removed.

Then, the supernatant was transferred to a plastic tube and 9.9 cc DDW was added. Next, 0.25 cc of 2M HCl was added to both TF and TSF samples.

The mixture was located at 50̊c (50 degrees of centigrade) for 10 minutes and neutralized by adding 0.5 ml of 1M NaOH plus 1 ml of TISABII (0.75M Acetate buffer, pH=5 containing 1M NaCl and 0.4% CDTA).

The analysis was performed by using a fluoride selective electrode (Thermo Orion, USA, 9609) and an Ion Meter (Thermo Orion, USA, 720A).

Statistical analysis: The mean and standard deviation of TF and TSF concentrations in dentifrices were calculated and analyzed with SPSS version 11 software. One-way ANOVA was employed to analyze TF concentration in all groups, after confirming the normality of data distribution.

The non-parametric Kruskal-Wallis and Mann-Whitney tests were used for the comparison of TSF between groups. P-value less than 0.05 was considered significant.

RESULTS

In order to perform a true evaluation of TF and TSF in the four mentioned dentifrices, three tubes of each brand were provided and three samples were taken from each tube.

Comparison of the TF concentration between 4 dentifrices: The highest (1605.45 ppm) and the lowest (1136.67 ppm) amount of TF were related to Crest and Signal toothpastes, respectively.

But the difference in this respect between groups was not significant (P= 0.39, Table 2 and Figure1)

Comparison of the TSF concentration between 4 dentifrices: The highest amount of TSF was related to Crest (2326.89 ppm) and the lowest to Pooneh (1188.86 ppm) toothpastes.

The results of Kruskal-Wallis and Mann-Whitney tests revealed that the amount of TSF in Crest dentifrice was significantly more than in other dentifrices (P<0.001, Table 2, Figure 1).

Comparative evaluation of the other dentifrices: Pooneh-Signal (P=0.233), Pooneh-Nasim (P=0.823) and Signal-Nasim (P=0.727) showed no significant difference regarding this variable. Comparison of both TF and TSF concentrations with limits set by the International Standard Institute revealed that all understudy dentifrices followed these standards except for Crest.

It is notable that the amount of TF (1605.45 ppm) and TSF (2326.89 ppm) in Crest was above the maximum standard limit (1500 ppm).

DISCUSSION

Using fluoridated dentifrices is the most common method of dental caries prevention; it has caused a reduction in the prevalence of dental caries in all countries [18]. Two essential requirements need to be met in a fluoridated dentifrice namely availability and stability of F. The abrasives used in dentifrices play an essential role in inactivating F ions [17, 19-20]. The inactivation of F may lead to formation of a low soluble product with decreased anti-caries effect [21].

In our study, the main abrasive was silica in all dentifrices except for Pooneh; which contained a Ca-based abrasive. Signal and Nasim contained Ca-based abrasives in addition to Silica (Table 1).

TSF concentration was the highest in Crest (which only contains silica abrasive) and the lowest in Pooneh (which only contains a Ca-based abrasive). The fact that TSF concentration in Ca-containing dentifrices was less than the amount declared by their manufacturers indicates that Ca-based abrasives decrease F stability. In agreement with our findings, Filho et al, [17] and Cury et al. [21] showed that incompatibility between the abrasive agent (usually $CaCO_3$) and F type (usually MFP) leads to lower concentrations of TSF.

A study by Condeh et al. showed that silica-based dentifrices either in combination with MFP or NaF had a greater amount of soluble F/TSF. These results verify our findings.

On the other hand, the higher concentration of TSF in Crest led us to suppose that with regard to F availability, NaF is better than MFP and SMFP; albeit this advantage of Crest may not be solely due to the presence of NaF, and the compatibility between NaF and silica may also play a role in this regard. NaF-containing dentifrices are often formulated with silica.

Similar findings were obtained by previous studies [22]. However, others showed no significant difference between MFP and NaF [23]. In another study by Arnold et al, [24] F availability and the remineralization effect of SMFP were shown to be greater than those of NaF. In our study, we found that TF and TSF concentrations in Nasim, Pooneh and Signal dentifrices were within the standard limit (1000-1500 ppm); but these concentrations were slightly higher than the maximum permitted limit in Crest.

Table 2. The mean and SD of TF and TSF in evaluated dentifrices (ppm)

Product Name	TF Mean (±SD)	TSF Mean (±SD)
Pooneh	1252.00 (±336.5)[a]	1188.86 (±154.3)[b]
Signal	1136.67 (±236.7)[a]	1305.72 (±235.4)[b]
Crest	1605.45 (±646.3)[a]	2326.89 (±213.6)[c]
Nasim	1364.66 (±377.3)[a]	1388.11 (±567.7)[b]
P value	0.39	< 0.001

Thus, all mentioned dentifrices had optimal anti-caries effect and none of them except for Crest can lead to F overdose.

In contrast, Hassanzadeh et al [25] in 2004 demonstrated that F ion concentration in most of the Iranian and some of the foreign made dentifrices was less than the required threshold for having anti-caries effect. But the amount of soluble F ion in Crest and Signal was above this threshold.

One of the limitations of our study was the small number of evaluated dentifrices which were different both in type of abrasive and F compound; thus, separate evaluation of the effects of different types of abrasives and F compounds on F stability was not feasible.

Two factors may be responsible for the superiority of TSF over TF in some dentifrices in this study and other similar surveys [21] namely 1) Presence of non-homogenized parts of a dentifrice due to separate sampling from different parts of the tube and 2) A multistage laboratory system causing more confounding factors.

With regard to the constraint of such studies and the small number of studies in this field, further surveys with larger sample size and more precise laboratory techniques are proposed.

CONCLUSION

The minimum amount of soluble fluoride required for anti-caries effect was available in the dentifrices evaluated in this study.

Furthermore, the stable form of F had a higher concentration in silica/NaF-containing dentifrice i.e. Crest compared to the Ca/ MFP containing dentifrices (Pooneh, Nasim, and Signal).

ACKNOWLEDGMENTS

The authors would like to thank Torabinejad Dental Research Center for helpful cooperation, and also Dr S. Khazaei, DMD, for his scientific assistance.

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