Community Dental Health (2015) **32,** 170–173
Received 29 September 2014; Accepted 3 April 2015

doi:10.1922/CDH_3518Thakkar04

Fluoride content and labelling of toothpastes marketed in India

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Objective: To enumerate the fluoride content and labelling practices of toothpastes marketed in India and to determine if the content is in accordance with the information provided on the packaging. ***Method:*** In vitro analysis of total and free available fluoride in 5 adult and 5 children's fluoride toothpastes in India using fluoride ion electrode. Information on the tube and carton was checked to assess the labelling. ***Results:*** The mean Total Fluoride concentrations (TF) in the toothpastes labelled 1,000 ppm, 500ppm and 458 ppm were 1,000, 500 and 449mg/L (sd values 3, 5 and 4mg/L) respectively. The toothpastes' Total Soluble Fluoride (TSF) concentration was generally slightly less than the TF concentration. Overall mean percentage of TSF concentration was 94% sd 9%. The mean percentage of TSF concentration in SMFP (sodium monofluorophosphate)/$CaCO_3$ (calcium carbonate) containing toothpastes was 86% sd 16% while that in NaF (sodium fluoride)/Si (silica) based toothpastes was 98% sd 1% and 95% sd 1% in the remaining toothpastes with unknown abrasive. NaF/Si based toothpastes had more TSF concentration than the others ($p<0.05$). All the information required by the Indian regulations was shown on all 10 toothpastes. ***Conclusion:*** The available fluoride content of one of the ten toothpastes was substantially less than the total fluoride content. Although the toothpastes were labelled following the guidelines of the regulatory body of India, 3 of the 10 failed to mention the abrasive present.

Keywords*: dentifrices, product labeling, topical fluorides, India*

Introduction

The main reason for the reduction in dental caries prevalence worldwide has been the use of fluoride toothpaste by populations all over the world (Bratthall *et al.,* 1996; Cury, 2002; Cury *et al.,* 2004). Fluoride toothpaste is one of the important fluoride delivery systems considering the hindrances with other methods of delivery (Frencken *et al.,* 2002; van Loveren *et al.,* 2005). When the oral hygiene practices of the population include use of fluoride toothpastes with a toothbrush, it not only has a caries reducing effect but the toothbrush also exerts a widespread beneficial effect on their periodontal condition (Frencken *et al.,* 2002). However, for the fluoride toothpaste to be effective, it must contain an appropriate concentration of Total Soluble Fluoride (TSF) (Cury *et al.,* 2010). The availability of soluble fluoride is essential in the oral environment to interfere with the caries process thereby reducing enamel demineralisation and enhancing the remineralisation (Cury *et al.,* 2010; Queiroz *et al.,* 2008). Promotion of the use of fluoride toothpaste should go hand in hand with quality control measures on its efficacy. Moreover, fluoride content should be accurately labelled on the tube and the carton.

In India, the Drugs and Cosmetics Act, 1940 (Government of India, 1940, Amendment 2008) gives the labelling guidelines for fluoridated toothpaste including rule 149A for labeling of toothpaste containing fluorides: 1, Fluoride content should not be greater than 1,000 ppm and content of fluoride in ppm shall be given on tube and carton; 2, Date of expiry should be mentioned on the tube and carton. However, compliance with labelling requirements and contents in various brands of fluoride toothpaste across the world varies (Benzian *et al.,* 2012). The regulatory bodies within a country need to ensure that toothpastes comply with their rules. This study was therefore planned to determine the fluoride content and labelling of toothpastes marketed in India.

Materials and Method

The study was an *in-vitro* study designed to determine total and total soluble fluoride available in toothpaste samples. The sample consisted of five adult and five children's fluoride toothpastes marketed in India and purchased from a supermarket in Mangalore city and selected for being within three months of their date of manufacture.

Ethical clearance to conduct the study was obtained from the Institutional Ethics Committee of MCODS, Mangalore (Ref: 13074). Analysis at the Environmental Engineering Laboratory, Civil Engineering Department, National Institute of Technology, Surathkal, Karnataka, India was completed within two months of ethical committee approval being obtained.

The ten toothpastes were labelled A to J for blinding. The toothpaste tubes, with their caps closed, were then carefully squeezed in order to homogenise the contents. The tubes were then opened and the first 3g of toothpaste to emerge from the tube were discarded.

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The fluoride analysis followed that described by Cury *et al* (2010) following a protocol modified from Pearce (1974) with 90-110mg (sd 0.01mg) of toothpaste being weighed out then homogenised in 10.0mL of distilled water. The suspension was prepared and proper mixing was ensured using a stirrer. Duplicates of 0.25mL of the suspension were transferred to sterilised containers for Total Fluoride (TF) analysis. The remaining suspension was transferred to a test tube and centrifuged (3,000g, 10min, r.t.) to remove Ionic Fluoride (IF) bound to the abrasive. Duplicates of 0.25mL of the supernatant were transferred to sterilised plastic containers to determine Total Soluble Fluoride (TSF) concentrations. For all TF and TSF containers, 0.25mL of 2.0M HCl was added, and after 1 h at 45°C, the samples were neutralised with 0.5mL 1.0M NaOH and buffered with 1.0mL of TISAB II (1.0M acetate buffer, pH 5.0, containing 1.0 M NaCl and 0.4% CDTA) (Cury *et al.,* 2010; Ricomini Filho *et al.,* 2012). Analyses were carried out using an ion specific electrode (Orion) coupled to an ion analyser (Orion 940, Orion Research) calibrated with fluoride standards. Analyses were undertaken immediately the toothpaste tubes were opened and all were duplicated.

For each toothpaste the place of manufacture, type of fluoride, fluoride concentration, abrasive agent present in the toothpaste, date of manufacture and date of expiry as shown on the tube or carton was recorded.

Data were entered in the computer and analysed using SPSS v17.0 with the level of significance at $p \leq 0.05$. Group differences in mean fluoride concentrations were analysed using Mann Whitney U test or Kruskal Wallis ANOVA where three groups were involved.

Results

The duplicate analysis of separately prepared samples and subsequent fluoride measurement revealed a duplicate measurement error, $\sqrt{\sum(x_1-x_2)/2n}$, for the total fluoride measurement of 9.2mg/L and 10.2mg/L for total soluble fluoride 10.2mg/L.

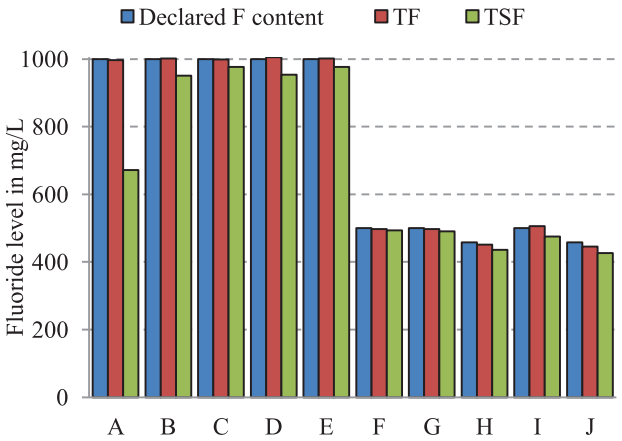

Figure 1. Fluoride content declared on the toothpaste samples, Total Fluoride (TF) concentration and Total Soluble Fluoride (TSF) concentration in adult toothpaste samples A to E and children's toothpaste samples F to J

The ten toothpastes brands analysed in this study included five toothpastes with declared fluoride concentration of 1,000ppm, three toothpastes with 500ppm and two toothpastes with 458ppm. Analysis revealed those groups' mean Total Fluoride concentrations (TF) to be 1,000mg/L sd 3, 500mg/L sd 5 and 449mg/L sd 4 respectively (NB 1ppm=1mg/L). The TF concentration in four toothpastes was found to be slightly more than declared on their labelling – their mean being statistically significantly greater than in the other six (p=0.038).

The Total Soluble Fluoride (TSF) concentration was generally slightly less than the TF concentration in the toothpastes analysed (Figure 1). The mean percentage of TSF concentration in the toothpastes was 94% (sd 9), range 67-99%.

The mean percentage of TSF concentration recorded in the 1,000ppm, 500ppm and 458 ppm toothpastes were 91% (sd 13), 97% (sd 3) and 96% (sd 1) respectively (Table 1). The mean percentage of TSF concentration in adult toothpastes was 91% sd 13 and a similar 97% sd 2 in children's (p=0.42).

Comparisons were made based on the fluoride compound and abrasive used in the toothpaste. The mean percentage of TSF concentration at 98% (sd 1, n=4) was greatest in the NaF (sodium fluoride)/Si (silica) based toothpastes (Mann Whitney U test, $p<0.05$) than in the SMFP (sodium monofluorophosphate)/$CaCO_3$ (calcium carbonate) toothpastes' 86% (sd 16, n=3) and SMFP toothpastes with no named abrasive's 95% (sd 1, n=3). There was a difference (p=0.03, Kruskal Wallis test assuming non-normal distribution of data) in the TSF concentration observed among these three groups (Table 2).

All toothpastes were labelled either on the carton or tube with all the necessary information required by national regulations though three did not mention the abrasive present. The details of the labelling practices are presented in Table 3.

Table 1. Total Soluble Fluoride concentration (TSF) in the toothpastes by stated concentration and intended age market

Total Fluoride concentration printed on toothpaste	*Total Soluble Fluoride (TSF) concentration observed on analysis*			
	n	*% mean*	*sd*	*(range)*
1,000 ppm	5	91	13	(67, 97)
500 ppm	3	97	3	(94, 99)
458 ppm	2	96	1	(96, 97)
Adult toothpaste	5	91	13	(67, 98)
Children's toothpastes	5	97	2	(94, 99)
Overall	10	94	9	(67, 99)

Table 2. Total Soluble Fluoride concentration (TSF) in the toothpastes with different fluoride compounds and abrasives

Fluoride compound and abrasive used	*% Total Soluble Fluoride (TSF) concentration observed on analysis*			
	n	*% mean*	*sd*	*(range)*
SMFP/$CaCO_3$	3	86	16	(67, 95)
NaF/Si	4	98	1	(95, 99)
SMFP*	3	95	1	(94, 97)

*The abrasive present in the toothpaste is not mentioned
Significant difference between groups, p=0.03, Kruskal Wallis test

Table 3. Information labelled on the toothpaste samples and results of fluoride analysis.

Sample, Toothpaste name	*Manufacturer/ Distributor*	*Fluoride Compound*	*Abrasive used*	*Manufacturing Date and Place*	*Expiry Date*	*ppm F**	*TF in mg/L*	*TSF in mg/L*	*TSF/TF*
A, Anchor White All round Protection	Anchor Health & Beauty Care	SMFP	$CaCO_3$	August, 2013 Haridwar, Uttarakhand	Aug. 2015	1,000	997	672	67%
B, Colgate Strong Teeth with Cavity Protection	Colgate - Palmolive	SMFP	$CaCO_3$	August ,2013 Goa	Aug. 2015	1,000	1,002	951	95%
C, Closeup Deep Action	Hindustan Unilever	NaF	Hydrated Silica	July, 2013 Haridwar, Uttarakhand	July 2015	1,000	999	977	98%
D, Sensodyne Rapid Relief	GlaxoSmith-Kline	NaF	Hydrated Silica	July, 2013 Silvassa, Dadra & Nagar Haveli	June 2015	1,000	1,005	954	95%
E, Pepsodent Germicheck	Hindustan Unilever	SMFP	$CaCO_3$	July, 2013 Daman	June 2015	1,000	1,002	977	98%
F, Colgate Toothpaste for Kids	Colgate - Palmolive	NaF	Silica	August, 2013 Goa	July 2015	500	497	493	99%
G, PepsodentKids Toothpaste	Hindustan Unilever	Na F	Silica	July, 2013 Haridwar, Uttarakhand	June 2015	500	497	491	99%
H, Cheerio Gel	Dr. Reddy's Laboratories	SMFP	Not stated	July, 2013 Baddi, Himachal Pradesh	June 2015	458	451	436	97%
I, Kidodent	Indoco Remedies Ltd.	SMFP	Not stated	August, 2013 Baddi, Himachal Pradesh	July 2015	500	506	476	94%
J, Pediflor Oral Gel	Group Pharma-ceuticals Limited	SMFP	Not stated	July, 2013 Malur, Karnataka	Dec. 2015	458	446	427	96%

* Concentration of fluoride mentioned on the toothpaste in ppm

Discussion

All the ten toothpastes analysed in this study stated on the tube or carton their total fluoride levels (ppm) and their actual total fluoride content levels differed by less than 3% from that level. The TF concentration in two children's toothpastes were 1-2½% higher than their stated level while all adult toothpastes were within ½% of their 1,000 ppm level. TF concentrations matching local standards has been reported in other studies (Benzian *et al*., 2012; Carrera *et al*., 2012; Conde *et al*., 2003; Cury *et al*., 2010; Giacaman *et al*., 2013; Hashizume *et al*., 2003).

There can be discrepancies observed at local manufacturing units of global toothpastes brands. If strict quality control procedures are not followed locally then toothpastes' TSF concentrations can vary. Manufacturers may use different ingredients in the manufacture of toothpastes, primarily to reduce manufacturing costs but such a change, especially with abrasives, may affect the toothpastes' free fluoride concentrations (Benzian *et al.,* 2012). The mean TSF concentration was slightly greater in NaF/Si based toothpastes than the others analysed as observed previously (Benzian *et al.,* 2012, Carrera *et al*., 2012; Conde *et al*., 2003, Cury *et al*., 2010, Ricomini Filho *et al.,* 2012). For fluoride toothpastes to be effective, the added fluoride should not be chemically bound to other ingredients, especially to calcium in abrasives. In most developing countries the majority of toothpastes have calcium carbonate as the abrasive (van Loveren *et al.,* 2005), which may inactivate some of the fluoride present (Conde *et al.,* 2003; Cury *et al*., 2010). The fluoride sources in toothpastes with sodium fluoride, ammonium fluoride and stannous fluoride are not compatible with calcium-containing abrasives. The fluoride sources in SMFP toothpastes, being firmly bound to the phosphate component, cannot bind to soluble calcium to form insoluble calcium fluoride so SMFP toothpastes have greater compatibility with calcium containing abrasives (Benzian *et al.,* 2012). During the storage of toothpastes containing SMFP/$CaCO_3$, some fluoride is inactivated by the abrasive content. To overcome this problem and ensure availability of free fluoride in use, many manufacturers add excess fluoride (more than the required ppm) in the toothpastes (Cury *et al.,* 2010).

It has been observed in previous studies that both Total Fluoride (TF) and Total Soluble Fluoride (TSF) concentrations decreased in the toothpastes over a period of time as compared to analysis of fresh samples (Conde *et al.,* 2003; Hashizume *et al.,* 2003; van Loveren *et al.,* 2005). In the present study, only fresh samples of toothpastes were used for analysis to avoid this effect by ensuring that the manufacturing dates of all toothpastes selected are within three months of the month of analysis.

Any observed discrepancy in the values of the fluoride concentrations in the toothpastes may be due to the problem of counterfeit products. Counterfeit products and medicines are a growing problem at the global level in both developed and developing nations though the true extent of the problem is not known. A WHO (2011) guideline provides information on measures that should be taken by the stakeholders and interested parties to combat counterfeiting of products (WHO, 2011). Effective action should be taken in India and elsewhere in line with these guidelines.

There is no regulation of the actual total free available fluoride in the toothpastes in many countries (Benzian *et al.,* 2012; Cury *et al.,* 2010; van Loveren *et al.,* 2005). However, the United States Food and Drug Administration (US FDA, 1995) state that toothpastes with NaF as the fluoride source should have ≥650 ppm and those with SMFP as the fluoride source should have ≥800 ppm available fluoride. ISO Standard 11609 (2010) requires toothpastes to declare and accurately label their total fluoride content, but not their free available fluoride content. National and international guidelines might usefully be reframed to include levels of total free available fluoride in toothpastes and its labelling on tubes or boxes as this quantity is associated with the anti-caries efficacy of the toothpastes.

To ensure proper delivery of fluoride through toothpastes, proper quality control, standard setting and compliance with standards are required. Three toothpastes in this study did not detail the abrasive they contained and had no list of ingredients on their boxes. So there is no requirement to list non-hazardous ingredients (e.g. abrasives) despite this being potentially desirable on account of their possible effect on free fluoride levels.

Only in brand A was a short-fall found in the toothpaste's total soluble fluoride concentration. This discrepancy may its anti-caries effect. Though all the toothpastes analysed met rule 149A of the Indian labelling requirement, poor practice was observed in three toothpastes which failed to mention the type of abrasive used and detail other ingredients.

As the quality control of toothpastes cannot be guaranteed, consumers in India may benefit from the simple recommendations suggested by Benzian *et al.* (2012): check the expiry date of toothpastes and purchase those with a recent manufacturing date; select a silica-based fluoride toothpaste (in preference to sodium monofluorophosphate toothpastes) with no calcium-containing abrasive as it is more likely to have an effective anti-caries action.

The authorities in India should consider the introduction of regular quality monitoring of fluoride toothpastes to strengthen compliance with existing regulations.

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