Original Research Article

Quantitative Spectrophotometric analysis of Fluoride content in dentifrice.

Abstract

BACKGROUND - A dentifrice and a toothbrush are universal armamentariums for oral hygiene maintenance. The presence of fluoride in commercial dentifrices aids in dental caries prevention. The fluoride content and compound are mentioned on the packaging along with the time of manufacture and its shelf life.

AIM - To evaluate the fluoride concentration of dentifrices available commercially over a period of time from the date of manufacture.
To compare any variations in the fluoride concentration over time to the content present at manufacture.

METHODOLOGY - This will be an In vitro comparative observational study. Standard dentifrices available Over the Counter will be selected. For each brand of dentifrice, three samples will be randomly chosen and used for fluoride analysis. The fluoride content will be assessed using spectrophotometric analysis, by a single blinded examiner. The test sample was compared to a standard solution, in a calibration curve to determine the fluoride concentration.

Key words: Children, Dentifrice, herbal, spectrophotometer, fluoride

INTRODUCTION -

Maintenance of oral health relies on effective oral hygiene practices. Cleaning the teeth using toothbrush and dentifrice is the most common and widely used oral hygiene practice. Since the introduction in 1945 by the Grand rapid water fluoridation project in Michigan, fluoride has become indispensable in dental caries prevention that resulted in global decline in dental caries.[1] The dentrifices are often the easily available vehicles to deliver fluoride. Its caries reducing property is well proved in not only adults but also children and adolescents.[2,3] Brushing twice daily with a fluoridated toothpaste is an effective and non invasive way to reduce caries incidence.[4]

Ancient Egyptians created first dental cream between 3000 and 5000 BC using pumice, myrrh, powdered oxen hoof ashes, egg shells. Persians used gypsum with burnt shells of snails and oysters along with herbs and honey around 1000 BC. Greeks and Romans around 1000 years later added crushed bones and oyster shells as abrasives to the powder mixture. Romans made it palatable by adding flavouring like charcoal and bark to help with bad breath. People in China and India are known to first used toothpaste around 500BC.[5,6] In 1892 Dr. Washington Sheffield is the first person to put toothpaste in a collapsible tube.[7]

In 1941, fluoride was incorporated in toothpaste by Joseph Muhler, using stannous fluoride as the main anti-cavity ingredient. This toothpaste was marketed as Crest. 1951- In India Binaca, launched by FMCG brand Reckitt Benckiser was the go to oral hygiene brand, Before brands like Pepsodent or Colgate became a household name, in the 1970s.[8]

In 1960, Crest was given the American Dental Association's first seal of acceptance after reviewing published research findings. Herbal toothpastes, become available as an alternative in 1975, including ingredients like peppermint oil, myrrh and plant extracts.[9] In 1987, edible toothpaste were invented by NASA so astronauts could brush their teeth without spitting in a zero-gravity.[10]

Dentifrices contain abrasives including calcium hydrogen phosphates, aluminum hydroxide, calcium carbonate, hydroxyapatite and silica; fluoride in the form of Sodium fluoride, sodium monofluorophosphate or stannous flouride; Detergents like sodium lauryl sulfate, Antibacterial agents like zinc chloride or triclosan; flavouring agents like peppermint, spearmint, and wintergreen; hydrating agent including sorbitol, glycerol, polyethylene glycol, xylitol, and water.[6] It is mandatory for most commercial dentifrices to mention the contents along with the amount of fluoride, on the package. The American academy of pediatric dentistry (AAPD) states that "Using no more than a 'smear' or 'rice-size' amount of fluoridated toothpaste for children less than three years of age may decrease risk of fluorosis. Using no more than a 'pea-size' amount of fluoridated toothpaste is appropriate for children aged three to six."[11] European academy of pediatric dentistry (EAPD)recommends brushing twice per day using a pea-size of 500ppm fluoridated dentifrice for children younger than 24 months. Children between two and six years of age are advised to brush twice daily with a pea-size amount of 1000ppm F toothpaste. Children six years and older are recommended to use 1-2 cm length of 1450ppm F toothpaste twice daily.[12]

Bureau of Indian Standards (BIS), the fluoride concentration in parts per million (ppm) along with other details such as net mass, month, and year

of manufacture and the expiry date has to be mentioned in the label of dentifrice package. As per BIS during the shelf life the product need to meet the requirement of the standard, which is usually from 12 - 24 months. (IS 6356:2001) REVISED 2006.[13] Fluoride present in the dentifrice may be reduced by a number of factors like abrasives, storage time and the surrounding temperature.[14]

This study was designed to evaluate the fluoride concentration of dentifrices available commercially in India using spectophotometry.
To compare variations if any in the fluoride concentration to the content present at manufacture as given on the toothpaste carton.

METHODOLOGY -

This is a single blinded in - vitro study in a laboratory setting.The Fluoride toothpastes were selected from various pharmaceutical stores in the 2 metropolitan areas that were within 6 months of manufacture. All the toothpastes selected had similar period of expiry of 24 months.

8 different toothpaste brands were selected 3 were for adult use,
2 were herbal, 3 for kids, as explicitly mentioned on the label. All the 3 adult toothpaste samples had available fluoride as 1000 ppm mentioned on the packaging and were labeled as sample A, B, C. Herbal toothpastes labeled D and E with available fluoride of 924 ppm and 1000 ppm respectively. Kids toothpaste labeled F,G and H with available fluoride as 500 ppm, 500 ppm and 458 ppm respectively.
The method used for spectrophotometric analysis carried out was as described by Rice WE et al. (2012).[15] The dentrifices were dispensed

individually in a teflon crucible to measure an amount of 1-1.5 gm using a digital meter.(Figure 1)

Figure 1 : Dentifrice dispensed in a teflon cruicible and measured on a digital meter

Each toothpaste sample after measuring was dispensed in separate crucibles labeled with alphabets and digested with 5 ml of concentrated Hydrochloric acid (HCl) at moderated temperature. All the crucibles were placed on a hot plate in a fume cupboard (Figure 2), for heating for 3–4 h, after which 5 ml of HCl was added again and heated till fuming seized.

Figure 2 : Cruicibles with dentifrice placed on a hot plate

The Teflon crucible was then cooled and rinsed. Distilled water was mixed to the existing dentifrice mixture and was made up to 100 ml in separate beakers. To these 10 ml of SPANDS-Zirconyl acid reagent mixture was added.

For the preparation of a standard fluoride solution, fluoride standards in the range of 0.1–1.40 mgF/L, were divided into 0.2, 0.4, 0.6, 0.8 mg/l. Each of these were prepared by adding appropriate quantities of distilled water to set 50 ml of standard fluoride solution. Ten ml of SPANDS - Zirconyl acid reagent mixture was added to each standard, samples and mixed well. This acid reagent imparts colour to the mixture in the spectrophotometer.

The ultraviolet–visible spectrophotometer was set at zero absorbance with reference solution and the readings of the standard solution were obtained at 570 nm wavelength of visible light. Readings of each of the dentrifice

solutions were obtained at the same wavelength and plotted on a graph with the standard solution as a reference. A calibration curve of microgram fluoride versus absorbance relationship was then plotted. (Figure 3)

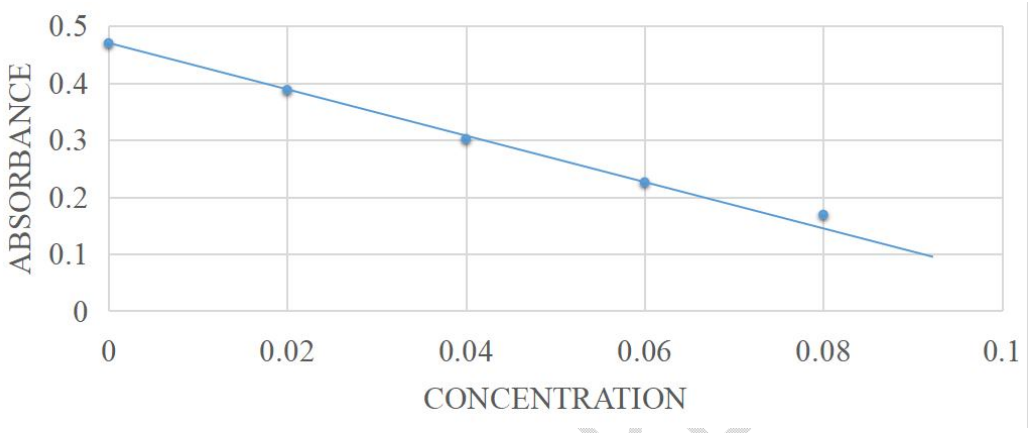

Figure 3: A calibration curve of microgram fluoride versus absorbance relationship

From the calibration curve, the concentration of fluoride present in each sample was obtained in µg which was converted into ppm using the formula -

µg F/sample weight in ml.

RESULTS -

Sodium monofluorophosphate and Sodium fluoride were the most common components in the toothpaste samples selected. 3 samples A,B,C of adult toothpaste had fluoride concentration 1000 ppm as mentioned on the label. The fluoride concentration on analysis had showed lower

fluoride concentration as claimed to be on the label. Sample A had 719.3 ppm, Sample B had 529.3 ppm and sample C had 789.14 ppm of fluoride after spectrophotometric analysis.

Out of 2 herbal toothpaste, sample D had fluoride concentration 1039.62 ppm which was similar or slightly higher to that printed (924 ppm) and sample E had lower which is 651.78 ppm than the printed 1000 ppm.

Among 3 toothpastes for kids, sample F had lower concentration of fluoride 305.99 ppm than mentioned on the label (500 ppm). Sample G had fluoride concentration 905.25 ppm that is higher than printed (500 ppm). Sample H had similar amount of fluoride on analysis that is 402.22 to that mentioned on the label (458) ppm.(Table 1)

Table 1: Samples labeled alphabetical along with fluoride concentration available and analysed in ppm.

Sample	Fluoride type	Available fluoride (ppm)	Fluoride analysed (ppm)
	ADULT TOOTHPASTE		
A	Sodium Monofluorophosphate	1000	719.3
B	Sodium Fluoride	1000	529.3
C	Sodium Monofluorophosphate	1000	789.14
	HERBAL TOOTHPASTE		
D	Sodium Fluoride	924	1039.62
E	Sodium Monofluorophosphate	1000	651.78
	KIDS TOOTHPASTE		

F	Sodium Monofluorophosphate	500	305.99
G	Sodium Fluoride	500	905.25
H	Sodium Monofluorophosphate	458	402.22

DISCUSSION -

Fluoridated toothpastes are effective as therapeutic agents for the inactivation or arrest of carious lesions. Dental Council of India (2018) recommends fluoridated toothpaste to be used under parental guidance for children under 6 years of age and optimal recommended fluoride to be 1000ppm - 1500ppm.[16]

Veeresh DJ et al. (2014) assessed the total and soluble fluoride concentration in six selected commercial dentifrices in Davangere. They concluded that the all the fluoride dentifrices had adequate amount of total (930-987 ppm fluoride) and soluble fluoride (916-943 ppm),[17] contrary to the results obtained in this study. Jairoun AA et al, (2021) used Ion Chromatography conductivity analysis to determine the total fluoride content. This study revealed that 22 of the sampled products had a total fluoride concentration below 1000 ppm fluoride. The amount of fluoride did not match the claims and indications given on the label.[18] In our study too all 3 Adult toothpaste samples showed lower fluoride concentration than claimed on the label.

Herbal dentifrice contain Guava-Anti-inflammatory, Neem-Antibacterial, Kalmi-Flavoring agent, Babul-Astringent, and other ingredients like Honey-Sweetening agent, Camphor-Antiseptic, Calcium Carbonate-

Abrasive, Glycerine-Humectant, SLS-Detergent and also use the sodium chloride and distilled water. Some herbal dentifrice formulations also have presence of fluoride in the form of sodium fluoride and sodium monofluorophosphate.[19] We couldn't find any studies where fluoride concentration in herbal toothpaste was analyzed. In this study the values for sample G (1039.62 ppm) comparable to that claimed on the package (1000 ppm). Fluoride concentration in sample H (651.78 ppm) is lower than claimed on the package (1000 ppm).

Borremans M, Van Loco J et al. (2008) conducted a similar study on toothpaste for children and found that the fluoride concentration of dentifrices was close to 500 ppm.[20] Benzain H et al. (2012) had analysed 115 samples and stated that the 85% of the children toothpastes had less total flouride concentration than that claimed on the label, similar to sample H in our study.[21] Farooq I et al. (2018) he analysed three samples of five different commercial brands of children toothpastes for its total fluoride concentration and total soluble fluoride concentration. The measured total flouride in 2 toothpastes was higher than mentioned, which is similar to sample H in this study while 3 were lower than mentioned on label which is similar to sample G in this study.[22] Chavez B et al. (2019) analysed concentration of total flouride and total soluble fluoride in children's dentifrices marketed in the city of Lima, Peru. Three samples of 23 dentifrices were included in the study. The total fluoride concentration in the majority of the fluoride toothpastes matched that shown on the label, which is similar to sample H in this study; except for one which is similar to sample G in this study. [23] Pillai DD, Vijayaraghavan R et al. (2022) in an in-vitro study, evaluated 4 brands of commercially available dentifrices indicated for children. On analysis concluded that all the samples had optimum amount of fluoride (449.5 ±

26.1 ppm.) in comparison to that mentioned on the label.[24] This study had similar results when compared with samples F and H. But sample G (905.25 ppm) has fluoride concentration more than claimed on package (500 ppm).

LIMITATION -

The total soluble fluoride concentration in the dentrifrice has not been determined and compared since the baseline total soluble fluoride is not mentioned on the toothpaste carton.

CONCLUSION -

The protective effect of fluoride in dentifrices against dental caries largely depends on fluoride present in it.

The fluoride content mentioned on the pack and the concentration following a spectrophotometric analysis showed considerable variations.

The results of a quantity lower than mentioned on the label and its result on the anti cariogenic activity needs to be studied.

More studies are required to evaluate the anticariogenic effects of different concentration of fluoride in dentifrice over varied time within the 2 year use of the toothpaste.

COMPETING INTERESTS DISCLAIMER:

Authors have declared that they have no known competing financial interests OR non-financial interests OR personal relationships that could have appeared to influence the work reported in this paper.

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