Evaluation of fluoride bioavailability in toothpastes

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Objectives: The aims of this study were to determine the total fluoride concentration and bioavailable fluoride concentration in different toothpastes, based on a newly suggested method by the International Organization for Standardization (ISO), and to compare the measured concentrations with the concentrations written on the packaging.

Methods: The concentrations of total fluoride (TF) and bioavailable fluoride (BF) were measured in six toothpastes. For the TF measurement, 1 g of each toothpaste was mixed with dipotassium hydrogen phosphate (K2HPO4), and hydrogen chloride (HCl) was placed. After 24 hours, the samples were centrifuged and total ionic strength adjustment buffer (TISAB) solution was added. For the BF measurement, the toothpaste was mixed with K2HPO4 for only 1 minute. The samples were centrifuged, and then HCl was placed and allowed to stand for 24 hours. The TISAB solution was added subsequently. The concentration of fluoride ions was measured using a fluoride ion-selective electrode and calculated against a standard curve.

Results: The six toothpastes were composed of different fluoride compounds and abrasives. The measured TF concentration ranged from 624.99 ppm to 1,353.00 ppm, and the similarity to the declared fluoride concentration ranged from 53.48% to 93.31%. The measured BF concentration ranged from 587.61 ppm to 1,360.05 ppm, and the similarity to the expected fluoride concentration ranged from 41.97% to 93.80%. Two samples were clearly separated when the samples were centrifuged, whereas the remaining four samples had unclear supernatants. The clearly separated toothpastes (i.e., toothpastes 5 and 6) had BF concentrations that were similar to or lower than the declared fluoride concentrations and the measured TF concentrations. However, the unclearly separated toothpastes showed inconsistent relationships between the measured TF and BF concentrations.

Conclusions: The measured TF and BF concentrations of the six toothpastes did not reach the expected fluoride concentration. This finding resulted from the different compositions and forms of the toothpastes. Therefore, the properties of toothpastes need to be considered when measuring their fluoride concentrations.

Key Words: Fluoride, Toothpaste, Bioavailability, Standards
Introduction

The large reduction in number of dental caries in the last few decades is considered to be due to the widespread use of both systemic and topical fluorides\(^1\). As the beneficial effect of topical fluoride has been proven through number of studies\(^2,3\), there was a huge improvement on the topical fluorides, with toothpaste still remaining as the most widespread and significant form of fluoride\(^4,5\). There was a Cochrane systematic review that also emphasized the role of fluoride toothpaste in anti-caries activity, yet it stated that the concentration of fluoride in toothpastes needs to be 1,000 ppm or more in order to be effective\(^6\).

On the other hand, some of early studies on the fluoride toothpastes failed to prove the efficacy of the toothpastes against caries without finding the exact mechanism\(^7,8\). However, as more number of investigations were done on fluoride toothpastes, it was found that for the toothpastes to be effective against caries, the fluorides need to be in soluble form and that the anti-caries effect could be reduced by insoluble salts formed between the free fluoride ions and the abrasives contained in the toothpastes\(^9,10\). Thus, it is crucial for the toothpastes to contain sufficient concentration of not only the total fluoride but also the soluble fluoride to be effective in prevention of caries.

However, the concentration of soluble fluoride can be lower than that of the total fluoride which is labeled on the packaging. In Korea, there were several attempts to measure the total fluoride and the soluble fluoride of the commercialized toothpastes in Korea, and they found that the majority of the analyzed toothpastes contained soluble fluoride close to 1,000 ppm\(^11,12\). Considering the maximum permitted fluoride content in toothpaste used to be 1,000 ppm when these studies were performed, it can be assumed that most of the fluoride toothpastes commercialized in Korea contained soluble fluoride in close proximity of the labeled fluoride. Nevertheless, there was a previous study which determined total and free fluoride from the toothpastes purchased in low-income countries, and they reported that in 25% of the toothpastes, <55% of the declared fluoride was in free fluoride form\(^10\). Therefore, in order to ensure the efficacy of fluoride toothpastes, there is a need of quality control measures that test toothpastes by measuring both total fluoride and soluble fluoride.

There are various measurement methods for fluoride in toothpastes. As one of the examples, International Organization for Standardization (ISO), who defines standards for the quality of fluoride toothpaste, describes methods for testing the total fluoride content of toothpastes in its standard 11609\(^13\), yet the guideline for soluble fluoride is still in preparation. Recently, a suggestion for measuring the soluble fluoride was made by the ISO TC 106. The suggested method measures the concentration of soluble fluoride in toothpaste slurry at one minute. As the actual tooth brushing time has been known to be approximately 1 minute\(^14,15\), this method can be considered as the representative method to capture all the bioavailable fluoride species during the 1-minute brushing. However, the fluoride measurement method often used in Korea is the diffusion method, which measures soluble fluoride concentration without considering the actual brushing time. Therefore, there is a need to evaluate the 1-minute fluoride bioavailability also in Korea.

The aims of this study were to establish the total and 1-minute potentially available soluble fluoride concentration of toothpastes that were available in many countries using the newly suggested method and to compare the concentrations with the concentration written on the packaging.

Materials and Methods

1. Fluoride toothpaste samples

Six toothpastes of five brands commercialized in many countries were evaluated (Table 1). Along with the instruction sheet information about the toothpastes such as lot numbers, density and concentrations of fluoride compounds, and expiration dates were provided by the ISO TC 106. The information provided on packaging was also checked for descriptive names of fluoride compounds and abrasives.

2. Preparation of the samples

Two different fluoride concentrations were assessed for each toothpaste: (1) Total fluoride and (2) 1-minute bioavailable fluoride. For both assessments, approximately 1 g of the toothpaste was measured into a tared test tube, and mixed with 0.1 mol/L K\(_2\)HPO\(_4\) (Dusksan, Ansan, Korea) at a 1:3 dilution to create a slurry sample. For each toothpaste, 2 slurry samples were produced: one for determination of Total Fluoride and the other for determination of Bioavailable Fluoride. After 12 slurry samples were produced from six toothpastes, the fluoride preparation was carried on as follows.

2.1. Determination of Total Fluoride (TF)

The six slurry samples were thoroughly vortexed, and 1.0 mol/L HCl of the twice the slurry volume was added to each