

Fluoride Measurements in Toothpastes using Acidic Phosphatase: Reproducibility of Results in a Three-Laboratory-Study

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Background and Aim of the Study

Some years ago the fluoride content in toothpastes from different Asian countries was measured by using Acidic Phosphatase (AcP) for the measurement of potentially bioavailable fluoride in dentifrices (van Loveren et al. 2005). This way of measurement tries to follow the fact that MFP is hydrolyzed enzymatically in the oral cavity by bacterial phosphatase (Pearce 1983). But so far, no data is available concerning the reproducibility of this method when applied in different laboratories. The main aim of this study was to assess the reproducibility of fluoride measurements in MFP toothpastes from the Philippines by applying the same protocols and involving three different laboratories. A second aim was to compare the reproducibility of fluoride measurements in NaF toothpastes.

MATERIALS AND METHODS

Laboratory: The preparation of samples and F-measurements were performed in three laboratories (Food and Drug Administration Manila, Philippines; Department of Preventive Dentistry, ACTA, Amsterdam, The Netherlands; Department of Conservative Dentistry, Heidelberg University Dental School, Heidelberg, Germany). The samples were measured independently and without any knowledge of the results of the other laboratories.

Tested Toothpastes: 19 different brands of toothpastes were bought in 2014 in supermarkets in Manila, Philippines. According to the labels, 7 of them contained Sodium Fluoride (NaF), 8 Sodium Monofluorophosphate (NaMFP), 2 a mix of MFP/NaF, 1 a mix of Sodium Fluoride and Bis(hydroxyethyl)aminopropyl – N – hydroxyethyl – octadecylamine – dihydrofluoride and one two different types of fluoride as an ingredient without specifying the exact chemical formula.

Fluoride Declaration: The information about F-type and F-concentration was taken from the package label. If F-content was indicated in % w/w, ppm was recalculated.

Preparation of samples: From each sample 2 g were taken and diluted with distilled water to a 1:3 dilution. The slurry was centrifuged and 0.1 ml of the supernatant was used to prepare a 1:20 dilution. 4U of AcP was dissolved in NaOAc buffer (0.5 ml/trial; pH 4.8) and the sample (0.5 ml) was digested for 24h at room temperature. NaF containing samples were prepared the same way except using plain buffer without enzyme. Prior to the F-measurement 1 ml TISAB II was added to each sample.

Toothpastes containing MFP + NaF and the toothpaste with no specified type of fluoride were prepared like plain MFP pastes. The mixed NaF/Amine toothpaste was prepared like NaF pastes.

Fluoride measurements were performed with a F-specific electrode. (Manila: Orion 9609 BNWP; Heidelberg: Mettler Toledo DX 219-F; Amsterdam: Hach ISE25F-9)

Calibration curve: a standard MFP and a standard NaF solution was diluted in 5 steps. These solutions were used to calibrate the F-specific electrode.

Data calculation: F-concentration was determined in ppm for 3 samples of each brand of toothpaste and these figures were used to calculate a mean value.

Measurement Results of Fluoride Content

Product Name	Manila [ppm]		Heidelberg [ppm]		Amsterdam [ppm]		labeled Fluoride [ppm]	Mean Difference labeled - measured Fluoride [ppm]
	MFP	NaF	MFP	NaF	MFP	NaF		
Colgate Fresh Confidence		1007		1099		1078	1000	-61
Pyodontyl Plus		930		1051		915	1000	35
Aquafresh triple protection		1348		1386		1271	1450	115
Sensodyne		988		1104		1150	1000	-81
Armani Hammer		1163		1313		1355	1100	-177
Systema Tooth and Gum Care		948		986		1095	no data	no data
CloseUp Fire Freeze		1298		1525		1482	1450	15
Lacalut Sensitive**		199		205		201	1476	1274
Colgate Maximum Cavity Protection	1032		1022		1017		1450	426
Hapee Night Mint	1577		1619		1494		1500	-63
GLEE Fluoride Toothpaste	511		538		319		no data	no data
Beam Active	114		66		68		no data	no data
Pepsodent	1347		1356		1375		no data	no data
UNIQUE	1440		1544		1631		1500	-38
Pearlie White	944		1011		1205		1050	-3
Unique	1541		1485		1345		1500	43
Kutitap Healthy Fresh White	666		569		424		1000	447
Fluocaril Original***	903		996		979		1000	41
Freshmint	946		917		1017		1000	40

* Mixed NaF/MFP-Toothpastes, ** Mixed NaF/Amin-F-Toothpastes, *** no type of fluoride labeled

Results, Table 1: Fluoride concentration measured in 19 toothpastes purchased in Manila, Philippines. Results are presented for each of the involved laboratories separately. The second last column presents the declared F-concentration on the label and the last column presents the difference between the labeled and the mean of the measured F-concentration.

	MFP [ppm]			NaF [ppm]		
	Manila	Heidelberg	Amsterdam	Manila	Heidelberg	Amsterdam
mean	1002	1011	989	985	1084	1068
SD	457	482	511	355	400	392
range	114-1541	66-1619	68-1631	199-1348	205-1386	201-1482
	H ₀ : 0.037 > 0.05			H ₀ : 1.145 > 0.05		

Results, Table 2: Mean values, standard deviations and range of fluoride concentrations measured in the toothpastes mentioned in table 1. These values are presented for the 3 laboratories involved in the study separately. Kruskal-Wallis Test was used with df = 2 and α = 0.05

DISCUSSION AND CONCLUSION

It is encouraging to see that the mean values for the fluoride concentration measured in NaF toothpastes did not differ statistically significantly between the 3 laboratories involved in this study although all laboratories used different technical equipment and in part chemicals from different producers. Looking at the values measured in the three laboratories for each toothpaste included in this study the difference between the highest and lowest value was in the range of 6 and 227 ppm.

It is also encouraging to see that the mean values for the fluoride concentration measured in MFP toothpastes did not differ statistically significantly between the 3 laboratories because the use of AcP is much more sensitive to mistakes. To the best knowledge of the authors this is the first study which investigated the reproducibility of F-measurements in MFP toothpastes with the AcP method. Furthermore, it is striking to see that some MFP toothpastes measured with the AcP method contained more fluoride than labeled, in one case even more than 1500 ppm.

The quality control of toothpastes marketed in Philippines clearly needs improvement because in several brands no fluoride concentration was labeled. Furthermore, in some NaF toothpastes the F-concentration was higher than labeled. This was also the case in some MFP-toothpastes which is surprising because it is agreed that AcP does not dissolve the complete amount of fluoride which is contained in the MFP-toothpaste. In addition, in one NaF toothpaste and in several MFP toothpastes the measured F-concentration was extremely low.

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