STUDY OF DENTIFRICE ABRASIVITY DETERMINATION BY THE RADIOMETRIC TECHNIQUE

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ABSTRACT

In order to evaluate abrasivity of dentifrices, experimental conditions were established for tooth specimen irradiation in the nuclear reactor, for brushing irradiated teeth with a brushing machine and for counting ^{32}P radiation transfered to the dentifrice samples. Abrasivity results presented a good precision. Correlation between the abrasivity and the mean particle size of dentifrice components was also studied.

INTRODUCTION

There are in Brazil many brands and a great variety of dentifrices since their formulas are frequently modified because of improved scientific knowledge or marketing factor.

However, there is not much information concerning abrasivity of dentifrices. Detailed formulations of abrasive systems are exclusive of manufacturers and are often not available in literature. It is known only that dentifrices are mixtures of different inorganic and organic compounds and usually dental abrasives include carbonates, silicas, phosphates, aluminas, zirconium silicate and organic abrasives such as polystyrene, polymethylmethacrylate and polyvinylacetate [1].

The purpose of this research is to develop a reliable technique for assessing dentifrice abrasivity. The development of an appropriate technique to evaluate the abrasiveness of the dentifrices in Brazil is of great interest since at the moment these determinations are performed abroad.

The determinations of abrasivity will allow useful information to industries, dentists, entities or associations and to the general public.

For toothpaste manufacturers, the abrasivity data are important to quality control of their products and to select raw materials used in their production.

For dentists, patients frequently ask their dentists or clinicians which toothpaste they would use since differences exist between the available toothpastes. In order to give this kind of advice, dentists should be familiar with the relative abrasivity of dentifrices and the most frequently tests used to determine these data. Besides this when the abrasion (wear on tooth) is diagnosed, dentists can minimize further progression by eliminating causative factors and by recommending less abrasive toothpastes. This is only possible if the abrasion values of available dentifrices are known.

For general public, abrasivity data are relevant to avoid the use of excessively abrasive pastes that can cause wear to teeth and restoring materials. Also people can select pastes objectively and not only based, for example, on taste.

Also for entities or associations interested in reducing tooth decay problems within the population as well as in standardization of the techniques for dentifrice abrasiveness evaluation, this work is of great significance.

A review of methods to determine the relative abrasivity of dentifrices and prophylaxis pastes was presented by Barbakow et al.[2]. These methods are known as radiotracer or radiometric technique, surface profile test or Talysurf procedure, weight loss technique, helium-neon gas laser method and laser diffusometric method.

Various methods have been developed over the years, but the methods of greatest interest today are the radiometric and surface profile methods. Two procedures are widely used in the radiometric method: one published by Hefferren [3] and adopted by American Dental Association (ADA) of the USA and other by British Standards Institution (BSI) [4] . From the surface profile method, the Talysurf procedure [5] is the most widely used.

The British radiometric procedure differs from the ADA procedure essentially in the type of reference material used for determining relative abrasivity. The procedure studied in this work was based on the paper presented by Hefferren [3].

EXPERIMENTAL

The radiometric procedure established in this work to determine dentifrice abrasivity consists of the following steps:

Selection and Preparation of Teeth. Dentin and enamel from extracted permanent human (or bovine) teeth were the substrates to be abraded in the test. Single rooted teeth, without caries and translucent areas were selected. After extraction teeth were stored in neutralized 4% formaldehyde solution and they were provided by Faculdade de Odontologia Ribeirão Preto-USP. Teeth were cut separating the enamel from dentin (root) and samples of about 14 mm long and at least 2 mm wide at the narrower end were obtained.

Irradiation of Teeth. Teeth were made radioactive by exposure to a neutron flux from IEA-R1 nuclear reactor. During the irradiation, a part of ^{31}P present in hydroxiapatite of teeth is converted into ^{32}P by means of the following nuclear reaction:

$$\underset{\text{hydroxiapatite}}{Ca_{10}(^{31}PO_4)_6(OH)_2} + {}^{o}n \longrightarrow$$

$$\longrightarrow \underset{\text{radioactive compound}}{Ca_{10}(^{32}PO_4)_6(OH)_2} + \gamma \quad (1)$$

Mainly phosphorus of the teeth produces sufficiently long lived radioisotope of ^{32}P with half life of 14.3 days in activities sufficient for practical measurements. Irradiation conditions were established and tooth specimens immersed in formaldehyde solution were irradiated in plastic vials during a 1-hour period under thermal neutron flux of $10^{12}n\ cm^{-2}s^{-1}$ and at temperature lower than 40°C. Effect of neutron irradiation upon the hardness of dentin was studied in this paper. Immediately after irradiation the tooth samples were removed from the core of the reactor to avoid irradiation with gamma radiation that can damage the teeth. For abrasivity test teeth were handled only after one week of decay time so that short lived activities, principally from ^{24}Na would have decayed.

Mounting of Teeth. The irradiated enamels or dentins were subsequently mounted in a mold made by dental methacrylate resin. The teeth were fixed using glue in the mold that has an exact dimension to be fitted in the sample holder (reservoir for dentifrice slurry) of the brushing machine.

Brushing Machine. The machine used for our experiments was provided by Brazilian toothpaste industries. This machine allows a toothbrush to pass over teeth mounted in the resin and immersed in a dentifrice slurry. A stroke counter with 125 strokes/min is also provided and the toohbrushes used were those made of nylon bristles of medium hardness and 10 mm long. A pressure on the toothbrush corresponding to a weight of 150g was chosen in this work.

Before the first brushing test, teeth were brushed with reference material slurry for 6 000 strokes in order to reduce irregular patterns of abrasion on the surfaces of newly mounted teeth.

Preparation of Reference Material and Dentifrice Slurries. The reference material slurry was prepared by mixing in a plastic bottle 10g of calcium pyrophosphate reference material and 50 ml of diluent previously prepared using carboxymethylcellulose (CMC) and glycerin.

This reference material is a special lot of calcium pyrophosphate with dentifrice grade which is available from the Monsanto Co, Illinois, USA.

The diluent was prepared by adding 5g of CMC in 50 ml of glicerin heated to 60°C under stirring to obtain a homogeneous mixture. Another 50 ml of heated glycerin was added to this mixture and the stirring was maintained for a total of one hour. Then, 900 ml of distilled water was added and the stirring was continued at room temperature for about 10 hours to obtain a clear solution of diluent. This solution was used one day after its preparation in order to stabilize the viscosity.

The dentifrice slurry was prepared by stirring 25 g of test dentifrice with 40 ml of distilled water.

A number from 1 500 to 3 000 strokes were applied in each brushing operation. Then the radioactive slurries were stirred and an aliquot of 3 ml was pipetted onto separated planchets. All planchets were placed in a Petri dish and the pipetted slurries were allowed to stand for at least 3 h and then they were dried in an oven at 60°C carefully to avoid crackings in the dried samples.

The ^{32}P activities of the dried paste were measured by using a Geiger Muller counting system. The beta radiation of 1.71 MeV ^{32}P with half life of 14.3 days was measured.

Calculations. To calculate the abrasivity, the ^{32}P counting rate obtained for test dentifrice is compared with that one obtained for reference material. These indices are knowm as RDA (Radioactive Dentin Abrasion), when the dentin specimens are used in the test and REA (Radioactive Enamel Abrasion) in the case of the enamels of teeth are used.

A score of 100 for the RDA and of 10 for REA are considered by ADA Committee when calcium pyrophosphate is used as reference material [6]. Then the value of RDA for test paste was calculated using the relation:

$$RDA_{paste} = 100 \times A_p \times f / A_r \quad (2)$$

where

A_p and A_r are the counting rates obtained for paste and reference material, respectively.

f is correction factor. Correction factor was applied because paste and reference material can present different characteristics of self-absorption and backscattering for beta radiation. This value was determined experimentally.

RESULTS AND DISCUSSION

Preliminary studies were carried out to establish adequate conditions and to obtain reliable results.

Selection of Appropriate Conditions for Irradiation of the Teeth. This study was carried out since long irradiations with high neutron flux or in a reactor position with high temperature can cause damage of the teeth changing their hardness and turning them brittle.

Results presented in Table 1 show a slight reduction of hardness with the irradiation, however within limit of error and it can be considered that there is no change.

Influence of the Number of Toothbrushing Strokes. Table 2 shows the results of the ^{32}P counting rate for different number of toothbrushing strokes applied on two dentin specimens.

Table 1. Effect of Neutron Irradiation upon Hardness of Bovine and Human Dentin

Conditions: Time of Irradiation = 1h
Neutron Flux = 10^{12} n cm^{-2} s^{-1}
Temperature < $40^{o}C$

	Bovine	Human
Before Irradiation	539 ± 40	581 ± 28
After Irradiation	463 ± 52	511 ± 39

The results are mean and standard deviation from 15 determinations. Results obtained using Hoppler apparatus.

Table 2. Influence of the Number of Tooth-brushing Strokes on Dentin Wear

Experiment no. 1 (dentin 1)		Experiment no. 2 (dentin 2)	
No. of Strokes	R (*) (cpm)	No. of Strokes	R (cpm)
3 000	11 558	1 000	1 647
6 000	21 960	2 000	2 925
10 000	38 702	5 000	6 771
15 000	58 003	8 000	10 378
20 000	79 505		

(*) - R indicates rate counting of ^{32}P obtained in toothpaste.

These results show the nearly linear increase of ^{32}P counting rate (or of the wear of teeth) versus number of strokes. A number between 1 500 to 3 000 strokes was chosen to be employed in our studies. Also these results show the possibility of using a same specimen for several runs.

Influence of Toothbrush Pressure on the Abrasiveness. Results of this study presented in Table 3 indicate that the increase in toothbrushing pressure results in the increase of the dentin abrasion. Besides this high brush pressure caused practical problems. Teeth can unglue from the resin mold.

Table 3. Influence of Toothbrush Pressure on Dentin Abrasion

Weight on Brush (g)	Mean Rate Counting of ^{32}P (cpm)
63	3 652 ∓ 184
150	5 517 ∓ 190
301	6 204 ∓ 540

Determination of the Correction Factor f This factor was determined using the same experimental conditions that those used in the RDA determinations, excepting the operation of toothbrushing. Instead of brushing, an aliquot of radioactive solution containing ^{32}P was added to the slurries. This radioactive solution was obtained by dissolving chips of irradiated teeth in the reactor. The following results of f were obtained for two different dentifrices: 1.05 ∓ 0.08 for dental cream and 1.24 ∓ 0.04 for dental gel. These results indicate that the correction for self-absorption and for backscaterring of abrasives for beta counting is necessary to calculate RDA or REA values.

RDA Determinations in Dentifrices. These results presented in Table 5 indicate a good precision of the method with relative standard deviations, generally, lower than 10%.

Table 5. RDA Determinations in Dentifrices Sold in São Paulo Supermarkets

Dentifrice Brand	Mean ∓ s
A	9.2 ∓ 0.4
B	29 ∓ 3
C	55 ∓ 5
D	122 ∓ 15

Influence of Mean Particle Size of Dentifrice Components on the Abrasivity. Results presented in Table 6 show that the RDA values depend on the size particle of dentifrice components indicating that a same abrasive can produce dentifrices with different grades of abrasivity.

Table 6. Influence of mean particle size of components of dentifrice on the RDA values

Dentifrice	Mean Diameter (um)	RDA
A1	5.09	119 ∓ 5
B1	3.07	75 ∓ 9
C1	2.65	50 ∓ 11

CONCLUSION

These preliminary studies allowed the establishment of the experimental conditions for the evaluation of abrasiveness of dentifrices by radiometric technique. This technique is simple and relatively rapid when compared with other methods since it does not require long periods for toothbrushings. The study of the accuracy of our results as well as the determination of REA values will be the next step of this work.

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