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Evaluation of the precision in the dentifrice abrasivity measurements obtained by a radiotracer method

I. M. C. Camargo, C. C. C. Zoppe, M. Saiki,* M. B. A. Vasconcellos

Instituto de Pesquisas Energéticas e Nucleares, Supervisão de Radioquimica, Caixa Postal 11 049, CEP 05422-970, São Paulo, SP, Brazil

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The radiotracer method was applied to evaluate the precision of the measurements of abrasivity data obtained for dentifrice samples and for abrasive agents (silica and calcium carbonate). This method consists of measuring ^{32}P transferred to a dentifrice or abrasive slurry when an irradiated dentin is submitted to a brushing simulation. Results obtained for abrasivity indices had good precision with relative standard deviations lower than 11.8%. Comparisons made between our abrasivity index data with those obtained at the Oral Health Research Institute of Indiana University also showed a good agreement.

Introduction

Abrasivity is one of the most important and also most discussed properties of dentifrices because it affects the ability to clean the teeth as well as potential damage it may cause to oral tissues.

Dentifrice formulas are frequently modified due to marketing and improved scientific knowledge factors, and when a new product is introduced it is important to have information about relative abrasivity and safety. Also the evaluation of abrasivity of raw materials such as silica and calcium carbonate is performed because their characteristics (particle shape, hardness, crystal structure and particle size distribution) influence the abrasion process.

On the other hand, the knowledge of precision of the abrasivity results is of great interest since this parameter is used in the comparison and classification of products presenting different abrasivity levels.

Along the years, various methods have been developed in order to quantify dentifrice abrasivity,[1,2] however there have been few studies concerning the quality of the results obtained for abrasivity data. Due to the widespread continuing interest in abrasivity tests, the American Dental Association Health Foundation conducted an International Collaborative Study in order to compare results obtained by different methods.[3] Radiotracer and surface profile methods were of most interest in this study; however, it was designed for all investigators who wished to participate with other methods, as well. In the case of tests in vivo, the comparison of the methods are more difficult because of practical limitations.

In this work, the radiotracer method was applied to analyse abrasivity of dentifrices and abrasive agents (silica and calcium carbonate) utilised in dentifrice manufacturing. This work was based on the method originally described by HEFFERREN.[4] It consists of the measurement of beta activities of ^{32}P transferred to a slurry of a dentifrice or of an abrasive agent when an irradiated dentin is submitted to a brushing simulation. The abrasivity index is the ratio of ^{32}P counting rate obtained for dentifrice and for the reference material.

Experimental

Selection and preparation of teeth

Dentins from extracted permanent human teeth were provided by Dentistry School of Ribeirão Preto, São Paulo University, SP. After extraction these tooth samples were stored in 4% formaldehyde solution and in the laboratory they were cut separating the enamel from dentin (root). Single rooted teeth without caries and translucent areas were selected as substrate to be abraded in the test.

Irradiation of the teeth

Roots of teeth were immersed in formaldehyde solution in plastic vials and irradiated for 1 hour under a thermal neutron flux of 10^{12} $n\cdot cm^{-2}\cdot s^{-1}$ at the IEA-R1 nuclear research reactor. During the irradiation, a part of the ^{31}P present in the hydroxiapatite of the teeth was converted to radioactive ^{32}P via the reaction $^{31}P(n,\gamma)^{32}P$. Immediately after irradiation the tooth samples were removed from the core of the reactor to avoid irradiation with gamma radiation that might damage them. For abrasivity tests, the teeth were handled only after one week of decay time so that short lived radionuclides, mainly ^{24}Na would have decayed.

* E-mail: mitiko@curiango.ipen.br

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Mounting of irradiated teeth

The irradiated teeth were fixed in a mold made with dental methacrylate resin that has an exact dimension to be fitted in a sample holder (reservoir for slurry of products to be tested) of the brushing machine.

Brushing machine

The brushing machine was manufactured at our Institute, and it is equipped with two toothbrushes made of nylon bristles of medium hardness and a stroke counter. A charge of about 150 g was applied on each brush head in order to simulate the manual application of force in actual toothbrushing. Before the first brushing test, the newly mounted teeth were brushed with reference material slurry for 6,000 strokes to reduce irregular patterns of abrasion on their surfaces. Irradiated dentins were brushed with a slurry containing American Dental Association (ADA) reference material (calcium pyrophosphate, from Monsanto Co, St. Louis, MO, USA) and a dentifrice or abrasive agent slurry. The reference material slurry was prepared by mixing 10 g of calcium pyrophosphate reference material and 50 ml of diluent previously prepared using carboximethyl-celulose, glycerin and water, prepared according to HEFFERREN.[4] The dentifrice slurry was prepared by stirring 25 g of the product with 40 ml of distilled water. The silica and calcium carbonate slurry was prepared the same way as the reference material. In each brushing operation a number of 1,500 or 3,000 strokes were applied. One stroke refers to a complete forward-and backward stroke of the brush. Aliquots of 3 ml from each slurry were pipetted on planchets and dried overnight in an oven with air circulation at 60 °C carefully, to avoid cracks in the dried samples.

Counting and calculations

The beta-particles of 1.71 MeV of ^{32}P with half-life of 14.3 days in the dried samples were measured by using a plastic scintillation detector. The identification of ^{32}P was carried out previously by measuring a sample for different decay times and then determining the half-life. To calculate abrasivity indices, known as radioactive dentin abrasion (RDA), the ^{32}P counting rate obtained for the product to be tested was compared with that obtained for the reference material. A score for 100 for the RDA of calcium pyrophosphate reference material was used in this work, according to the ADA Committee.[4] Then the RDA was calculated using the relation:

$$RDA = 100\, A_p \cdot f / A_r \tag{1}$$

where A_p is the counting rate obtained for dentifrice or abrasive agent, A_r is the counting rate for reference material, f is the correction factor applied in order to compensate for the differing self absorption and backscatterring radiation characteristics inherent of each abrasive. This factor is determined experimentally.

Results and discussion

Table 1 shows the RDA results obtained for different brands of Brazilian dentifrice samples sold in the supermarkets. The relative standard deviations obtained in these analyses varied from 4.8 to 12%. The levels of precision obtained with gel and cream forms of dentifrices were the same. The RDA results obtained for abrasive agents of silica and calcium carbonate presented in Table 2 indicate relative standard deviations varying from 5.9 to 11.8%. Dentifrice slurries also presented difficulty for pipetting due to foam formation, however, this problem did not affect the reproducibility of the results. The uncertainties due to statistical counting error and f correction factors were approximately 1.0 and 3.5%, respectively. Another possible source of errors which could affect the RDA value is the carryover effect in which the removal of dentin by an abrasive influences the following brushing on the same tooth. The potential carryover effect was examined and it was very small in comparison to other effects. This effect was minimized by using a "sandwich" design that consisted of doing a sequence of three consecutive brushings with the slurries of: reference material, dentifrice and again with the reference material. The average of counting rates obtained in brushings with two reference material slurries was considered in the RDA calculation. Also the same dentin could be used in several brushing operations because the dentin removal in the first brushing step did not influence the following brushing on the same dentin.

Table 1. RDA values obtained for different brands of dentifrice samples

Sample No.	Form	n	RDA ± s	s_r, %
1	Gel	8	87 ± 4	4.6
2	Gel	8	77 ± 4	5.2
3	Gel	8	33 ± 4	12.1
4	Cream	8	74 ± 8	10.8
5	Cream	8	66 ± 7	10.6
6	Cream	8	20 ± 1	5.0

n – Number of determinations.
RDA ± s – arithmetic mean and standard deviation.
s_r – Relative standard deviation, (%).

Table 2. RDA values obtained for different abrasives

Sample No.	Abrasives	n	RDA ± s	s_r, %
1	Silica	7	136 ± 8	5.9
2	Silica	8	94 ± 6	6.4
3	Silica	8	85 ± 10	11.8
4	Calcium carbonate	8	54 ± 4	7.4
5	Calcium carbonate	8	24 ± 2	8.3
6	Calcium carbonate	8	19 ± 2	10.5

Table 3. Comparison of RDA results obtained in this work and those obtained at the Indiana University

Dentifrice (form)	This work	Indiana University
A (gel)	72 ± 3 (n=8)	76 ± 14 (n=8)
B (cream)	89 ± 6 (n=11)	95 ± 12 (n=8)
C (gel)	39 ± 4 (n=11)	43 ± 16 (n=8)
D (cream)	59 ± 5 (n=8)	52 ± 9 (n=8)

The radiotracer method applied in this work showed good precision and it can distinguish between products with as little as 10% abrasivity difference when eight replicate determinations are made.

The accuracy of the method was not evaluated since there is no certified product for RDA values. In this work comparison was made between our results with those obtained by the Oral Health Research Institute of Indiana University, USA. These results presented in Table 3 indicate a good agreement.

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