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Abrasiveness evaluation of silica and calcium carbonate used in the production of dentifrices

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Synopsis

Our purpose was to apply a radiometric method to an abrasiveness evaluation in samples of silica and calcium carbonate used as an abrasive in a dentifrice, to help in a prudent selection of materials by dentifrice producers. The results of RDA (radioactive dentin abrasion) abrasiveness indices obtained for these compounds varied from 136 to 19. The relative standard deviations of these RDA results varied from 5.9% to 11.8%, showing a good precision in the method. Also, the results obtained indicated that the abrasiveness indices increase with the particle size of the material. A comparison between different abrasives with similar particle sizes showed that silica presents higher RDA values than calcium carbonate.

INTRODUCTION

Dentifrices are cosmetics used with toothbrushes to clean tooth surfaces to prevent the accumulation of stains and plaques. Therefore, they should present an appropriate abrasiveness to clean the teeth, but without causing wear. Consequently, knowledge of the characteristics of the abrasive agents used for dentifrice manufacturing is of interest to industries in order to obtain appropriate products for perfect oral hygiene without causing wear to the teeth or restorative materials. The abrasives commonly utilized in the dentifrices produced in Brazil are silica and calcium carbonate, and their quantities in dentifrices vary from 30% to 48% in mass (1,2).

Among several articles related to the use of abrasives in the dentifrices, one by Boer *et al.* (3) evaluated the wear caused by abrasives through the method of surface profilometry and verified the correlation between abrasiveness and the particle size of abrasives. Also, these authors obtained different abrasiveness values for distinct abrasives presenting similar particle sizes, that is, the abrasive $Al(OH)_3$ of 7-μm particle size showed greater abrasiveness than that presented by $CaCO_3$ with 8-μm particle size. This difference in abrasiveness may be attributed to the distinct particle hardness of these two agents.

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Kinoshita *et al.* (4) examined several dentifrices by the methods of surface analyzer and scanning electron microscopy. Among the several abrasives ($CaHPO_4 \cdot 2H_2O$, a mix of $CaHPO_4 \cdot 2H_2O$ + $CaHPO_4$, and $CaSiO_3$), $CaSiO_3$ appeared to be more abrasive than the other two. Also, Panzeri *et al.,* (5) examined by scanning electron microscopy the particles of abrasives used in 17 dentifrices and concluded that the majority of their particles present irregular forms and heterogeneous arrangements of the particles.

In this work, the abrasiveness of the samples of silica and calcium carbonate was evaluated by radiometric method. This method consisted of brushing the irradiated teeth (dentine) with an abrasive agent and reference material slurries, one at each time. The beta radioactivity of ^{32}P transferred from the dentine to slurries was measured using a plastic scintillator detector. The abrasiveness index or RDA (radioactive dentine abrasion) is the ratio of ^{32}P counting rates obtained for abrasive and reference material slurries (6).

MATERIALS AND METHODS

MATERIAL SAMPLE

The materials silica and calcium carbonate were provided from industries.

PROCEDURE

The radiometric method applied was based on a paper by Hefferren (6). The experimental conditions were established in order to use the available facilities. This experimental procedure consisted of the following steps:

Selection of the teeth. The substracts to be abraded were roots from extracted permanent human teeth. After extraction, the teeth were stored in 4% formaldehyde solution. They were cleaned by stirring in a domestic detergent solution and then were washed with water and cut, separating the crown from the root.

Irradiation of roots of the teeth. The roots of the teeth immersed in formaldehyde solution were irradiated in plastic vials during a one-hour period under a thermal neutron flux of 10^{12} n/cm^{-2}/s^{-1} at the IEA-R1 nuclear research reactor and in a position where the temperature was lower than 40°C. After irradiation, the tooth samples were removed from the core of the reactor to avoid damage from gamma radiation. During the irradiation a part of ^{31}P present in the hydroxyapatite of teeth was converted to radioactive ^{32}P.

Brushing operation of the teeth. After about one week of decay time, these irradiated teeth were fixed in a mold made by dental methacrylate resin that was fitted in a sample holder (reservoir for slurry) of a brushing machine. The brushing machine was manufactured at IPEN/CNEN-SP and was equipped with two toothbrushes made of nylon bristles of medium hardness and a stroke counter with 125 strokes/min. A pressure corresponding to 150 g could be applied on each toothbrush. Before the first brushing, the irradiated dentins were brushed with a slurry containing reference material of calcium pyrophosphate for 6000 strokes in order to reduce irregular patterns of abrasion on the surfaces of the newly mounted teeth. The number of strokes applied to each toothbrushing operation was 1000.

The slurries of reference material calcium pyrophosphate supplied by Monsanto Co. (St. Louis, MO) or of abrasives were prepared using a mass of 10 g of the material and 50 ml of diluent. In the case of thickeners, the slurries were prepared using a mass of 5 g of the material and 50 ml of diluent because of the large volume of the thickener. The diluent was prepared by adding 5 g of carboxymethylcellulose in 50 ml of glycerin heated to 60°C while stirring to obtain a homogenous mixture. Another 50 ml of heated glycerin was added to the mixture, and then 900 ml of distilled water was added. The stirring was continued at room temperature to obtain a clean solution of diluent.

Each radioactive slurry was stirred, and three aliquots of 3 ml were pipetted onto separated planchets. These slurries were dried in an oven with air circulation, at 60°C, carefully to avoid cracking in the dried samples. The beta radiation of 1.71 MeV ^{32}P (with a half life of 14.3 days) of the dried samples was measured using a plastic scintillator detector.

Calculation of abrasiveness indices. To calculate the abrasiveness indices, known as RDA, the ^{32}P counting rate obtained for abrasive material was compared to that obtained for the reference material. A score of 100 for calcium pyrophosphate RDA was considered according to an ADA (American Dental Association) committee (6). Correction factors were also applied in this calculation because different abrasives may present distinct self-absorption and backscatterring radiation characteristics.

Particle size and microscopy analysis of abrasives. The particle size of the silica and calcium carbonate samples was determined by sedigraphic method and their particle forms were examined using scanning electron microscopy at the Metallurgy Department of the IPEN/CNEN-SP.

RESULTS AND DISCUSSION

Table I shows RDA values obtained for six samples of silica and three samples of calcium carbonate, together with their particle sizes determined by sedigraphic method.

The RDA results for raw materials used as abrasive agents (silica 1 and calcium carbonate) presented in Table I varied from 136 to 19. The relative standard deviations of these RDA results, in general, varied from 5.9% to 11.8%, showing a good precision in

Table I
RDA and Particle Size Obtained for Raw Materials, Silica and Calcium Carbonate

	Silica 1 (abrasive)			Silica 2 (thickener)			Calcium carbonate		
Samples	A	B	C	D	E	F	G	H	I
RDA ± s	136 ± 8	94 ± 6	85 ± 10	7 ± 1	6.6 ± 1.0	5.5 ± 2.1	54 ± 4	24 ± 2	19 ± 2
s_r (%)	(5.9)	(6.4)	(11.8)	(14.3)	(15.2)	(38.2)	(7.4)	(8.3)	(10.5)
n	7	8	8	8	5	6	8	8	8
Mean diameter (μm)	4.26	3.21	2.54	1.20	0.31	0.32	3.13	1.77	1.49

RDA ± s: RDA arithmetic mean values and standard deviation.
s_r: relative standard deviations of the RDA values.
n: number of determinations.

the method. The silica 2 substances used as agent thickener presented less satisfactory results, with relative standard deviations varying from 14.3% to 38.2% (Table I). The precision of the thickener results was not so good, probably due to the difficulty in obtaining a homogeneous slurry of thickener during the toothbrushing.

Table I indicates that there may be a relationship between the RDA results and particle size of each type of raw material, that is, the abrasivity indices of $CaCO_3$ and SiO_2 used as abrasive agents increase with the particle size of the material. However, different types of abrasives with similar particle size presented distinct RDA values. As can be seen in the case of the sample, silica B with a particle size of 3.21 μm presented an RDA value about two times higher than that presented by calcium carbonate G with a 3.13 μm size. This result is in agreement with those presented by Boer *et al.* (3).

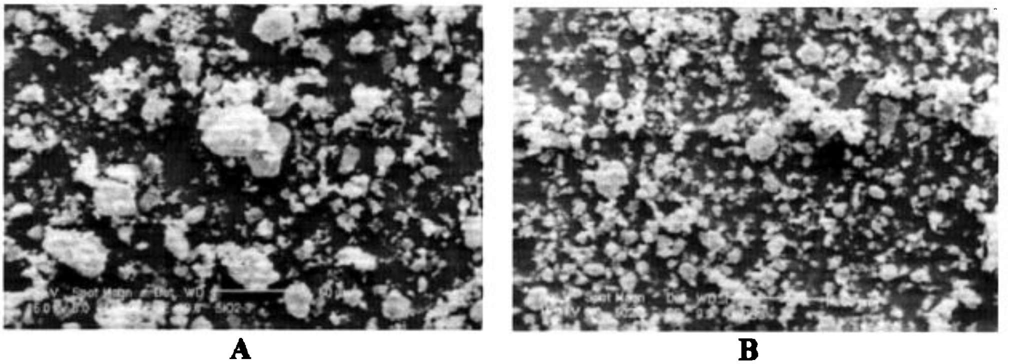
Figure 1 shows the shapes of particles of silica and calcium carbonate obtained by scanning electron microscopy. It can be observed that the particle shapes of the two abrasives are not uniform and that the $CaCO_3$ appears to have more agglomerated particles than SiO_2. According to Navarre (7), the materials constituted of particles with heterogeneous arrangements and irregular forms are more abrasive than those formed with homogeneous arrangements and regular forms.

RDA values for calcium carbonate were also evaluated using different masses of the abrasives in the preparation of slurry with 50 ml of diluent. For 5, 10, 15 and 30 g of $CaCO_3$, the RDA values obtained were 52, 74, 71 and 73, respectively. This preliminary study indicated the increase in RDA values with the mass of $CaCO_3$ until about 10 g. For quantities of $CaCO_3$ higher than 10 g, the RDA values were very close.

According to Roa (1), the quantity of $CaCO_3$ generally used in dentifrice manufacturing corresponds to about 20 g of $CaCO_3$ in the slurry (50 ml) used in our RDA evaluation.

CONCLUSIONS

The determination of RDA values of the abrasives can be utilized for prudent selection of raw materials by dentifrice producers. The radiometric method presented here is simple and fast because it does not require long periods for toothbrushing. Also, the RDA results obtained indicated that the abrasiveness of calcium carbonate and silica compounds increased with the particle sizes of the materials. However, it is important



A B

Figure 1. A: SiO_2 particles (502× magnification). B: $CaCO_3$ particles (447× magnification).

to consider that others factors, such as particle hardness, shape, and distribution, can also affect the abrasiveness of the raw materials.

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