

# Loss on drying, calcium concentration and pH of fluoride dentifrices

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## Abstract

**Introduction:** Fluoride dentifrices containing calcium carbonate have advantages such as control of dental plaque and progression of dental caries, also contributing to oral hygiene, represent most dentifrices marketed in Brazil. **Aim:** To evaluate the physicochemical properties of seven fluoride dentifrices containing calcium carbonate in relation to hydrogen potential (pH), loss on drying and calcium concentration. **Materials and Methods:** Data collection was performed using the potentiometric method for pH ranges, gravimetric analysis for loss on drying and atomic absorption spectrometry for the concentration of calcium ions. All tests were performed in triplicate and the analysis was performed entirely at random according to one-way analysis of variance at 5% significance level. **Results:** The pH values were alkaline and ranged from 8.67 (Oral-B 123<sup>®</sup>) to 10.03 (Colgate Máxima Proteção Anticáries<sup>®</sup>). The results of loss on drying ranged from 33.81% (Oral-B 123<sup>®</sup>) to 61.13% (Close Up<sup>®</sup>), with significant differences between brands tested. In relation to the calcium content, the highest and lowest concentrations were found in dentifrices Even<sup>®</sup> (155.55 g/kg) and Colgate Ultra Branco<sup>®</sup> (129 g/kg), respectively, with significant difference ( $P < 0.05$ ). **Conclusion:** Fluoride dentifrices analyzed showed alkaline pH and high levels of loss on drying and calcium concentration. However, these physicochemical characteristics differed according to the different brands tested.

**Keywords:** Calcium carbonate, chemistry, dentifrices

## Introduction

The use of dentifrice has emerged as the most common form of oral hygiene in most developed and developing countries. Its use has been associated not only with the purpose of cleaning teeth, but also anticaries and anti-sensitivity action, feeling of freshness and reduction of bad breath.<sup>[1]</sup> There is a wide range of formulations that in addition to adequate brushing, can satisfactorily remove dental biofilm.<sup>[2,3]</sup>

Dentifrices formulated with sodium monofluorophosphate and calcium carbonate present considerable anticaries effect because these formulations show greater benefits resulting from the supply of high levels of calcium into the oral cavity

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and the ability of calcium carbonate particles to neutralize the harmful effect of biofilms.<sup>[4]</sup>

In this sense, fluoridated dentifrices containing calcium carbonate play an active role in the oral biochemistry through a direct action on the processes of demineralization and remineralization of dental tissues.<sup>[5]</sup> The presence of calcium carbonate contributes to dental polishing and stain removal, and this dentifrice composition represents most dentifrices marketed in Brazil.<sup>[6]</sup>

The hydrogen potential (pH) alone is the best parameter to evaluate the erosive potential of dentifrices.<sup>[7]</sup> Thus, according to the International Standard used by the Brazilian Institute of Metrology, Quality and Technology, the pH of dentifrices should range within values considered safe, from 4.5 to 10.5.<sup>[8]</sup> For this, their components must not be present in concentrations that could cause toxic or allergic reactions when in contact with the oral cavity. It is noteworthy that saliva protects enamel up to critical pH not  $< 5.5$ , but dentin is more sensitive and does not bear critical pH lower than 6.5.<sup>[5,8]</sup>

Whereas tooth enamel surface may change due to the action of abrasive agents, since high levels can cause damage to the surface roughness, it is also necessary to evaluate the presence of inorganic contents in dentifrices through physicochemical tests such as loss on drying, which is a test used to determine the amount of volatile substance of any nature eliminated the conditions specified in its monograph.<sup>[9-11]</sup>

It is known that the presence of calcium in the dentifrice formulation leads to the necessity of higher fluorine

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concentration due to the presence of reactions between them.<sup>[5]</sup> In relation to calcium concentrations, literature shows that dentifrices containing carbonate calcium may provide extra benefits by raising the calcium levels and neutralizing the biofilm acidity.<sup>[4]</sup>

Thus, for the correct indication of dentifrices, knowledge of their physicochemical characteristics and composition is necessary, both for use and for purposes of analysis that can contribute to its development through quality control, emphasizing that to perform correct prescription, it must be based on scientific evidence.<sup>[3,12,13]</sup>

Thus, the aim of the present study was to evaluate *in vitro* physicochemical characteristics such as pH, loss on drying and calcium concentration of fluoride dentifrices containing calcium carbonate.

## Materials and Methods

This is an *in vitro* study. Seven different fluoride dentifrice brands containing calcium carbonate and commercially available in Brazil [Table 1] were selected. Data collection was performed at the Laboratory Management and Treatment of Waste (LABGER), located at the Federal University of Campina Grande, Paraíba, Brazil.

## Physicochemical analyses

All analyses were performed in triplicate and the equipment used was previously calibrated according to specifications.

### *pH analysis*

Initially, 5 g of each dentifrice were weighed on a precision scale (BEL Engineering®, Piracicaba, Brazil), followed by dilution with 15 ml of distilled water with the aid of thermal magnetic stirrer (Biomixer®, Ribeirão Preto, SP, Brazil) at constant temperature of 25°C. Then, the pH values were measured using pHTek pH meter (pHTek®, Curitiba, PR, Brazil).<sup>[11]</sup>

### *Loss on drying*

About 5 g of each dentifrice were weighed in porcelain crucibles in a precision scale (BEL Engineering®, Piracicaba, SP, Brazil). Then, the crucibles with dentifrice were taken to the oven (Odontobras®, Ribeirão Preto, SP, Brazil) and heated for 24 h at 100 ± 5°C. After the set time, the samples were weighed again to constant weight as the same weight already obtained, in successive weightings on a precision scale.<sup>[10,11]</sup> Loss on drying values were obtained by Equation (1).

$$PPD = \frac{P_i - P_f}{M_d} \times 100 \quad (1)$$

**Table 1: Composition of dentifrices used in the study**

Dentifrice	Manufacturer	Composition	Batch
Colgate Máxima Proteção Anticáries Menta Refrescante®	Colgate-Palmolive Industrial Ltd.	Calcium carbonate, water, glycerin, sodium laureth sulfate, sodium monofluorophosphate (1450 ppm fluoride), carboxymethyl cellulose, aroma, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, sodium hydroxide	3102BR123K
Colgate Tripla Ação Menta Original®	Colgate-Palmolive Industrial Ltd.	Water, calcium carbonate, sorbitol, sodium lauryl sulphate, sodium monofluorophosphate (1450 ppm fluoride), aroma, carboxymethyl cellulose, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, xanthan gum, sodium hydroxide, CI 74260, CI 7416	3055BR1211*L
Colgate® Ultra Branco™	Colgate-Palmolive Industrial Ltd.	Water, calcium carbonate, sorbitol, alumina, sodium lauryl sulphate, flavor, sodium monofluorophosphate (1450 ppm fluoride) carboxymethyl cellulose, sodium bicarbonate, tetrasodium pyrophosphate, xanthan gum, benzyl alcohol, sodium saccharin, sodium hydroxide	2217BR123A
Close up Triple Menta®	Unilever	Calcium carbonate, water, sorbitol, hydrated silica, sodium lauryl sulfate, sodium monofluorophosphate (1450 ppm fluoride), aroma, carboxymethyl cellulose, potassium citrate, benzyl alcohol, sodium silicate, sodium saccharin, CI 74260, limonene	72123022016
Even Proteção Refrescante Anticáries®	Indústrias Reunidas Raymundo da Fonte S/A	Glycerin, sodium monofluorophosphate (1500 ppm fluoride), sodium saccharine, carboxymethyl cellulose, sorbitol, sodium silicate, tetrasodium pyrophosphate, methylparaben, propylparaben, calcium carbonate, sodium lauryl sulfate, aroma, water	107 806
Oral-B 1-2-3 Menta Suave®	Suavetex Ltd./ Procter and Gamber	Sodium monofluorophosphate (1450 ppm fluoride), calcium carbonate, water, sorbitol, silica, sodium lauryl sulfate, carboxymethyl cellulose, aroma, trisodium phosphate, sodium saccharin, sodium phosphate	30770435A4
Sorriso Dentes Brancos®	Colgate-Palmolive Industrial Ltd.,	Calcium carbonate, water, glycerin, sodium lauryl sulphate, aroma, sodium monofluorophosphate (1450 ppm fluoride), carboxymethyl cellulose, tetrasodium pyrophosphate, sodium bicarbonate, benzyl alcohol, sodium saccharin, sodium hydroxide, limonene	3032BR1221*L

Where:

PPD = Loss on drying

$P_i$  = Initial weight (porcelain crucible + 5 g dentifrice)

$P_f$  = Final weight (porcelain crucible + 5 g dentifrice after 24 h)

$M_d$  = Dentifrice mass (5 g).

### Calcium concentration

Initially, in separate beakers, 5 g of each dentifrice sample were weighed on a precision scale (BEL Engineering®, Piracicaba, SP, Brazil). Then, acid digestion was performed with 15 ml hydrochloric acid with the aid of a thermal magnetic stirrer (Biomixer®, Ribeirão Preto, SP, Brazil). Soon after, the sample was diluted in 50 ml of distilled water in a 50 mL volumetric flask. The samples were filtered with the aid of a funnel and filter paper for filtering waste from acid digestion.

After filtration, the samples were placed in 50 ml containers. The calcium concentration was read in Perkin-Elmer® atomic absorption spectrophotometer with flame atomization model 5100PC. Analyses were performed using the main calcium resonance line (589.0-nm) and impact pearl. The height of the burner was 8 mm and the air/acetylene mixture was 10.0/2.0 L/min.<sup>[14-16]</sup>

After reading, the values are given in mg/L. For a better visualization of values in the context of its commercial presentation (pasty), values were transformed into mg/kg, as presented by Equation (2).

$$[Ca^+] \text{ mg/kg} = \frac{[Ca^+] \text{ mg/L}}{100} \text{ (2)}$$

Where:

$[Ca^+] \text{ mg/kg}$  = Calcium Concentration in milligrams per kilograms

$[Ca^+] \text{ mg/L}$  = Calcium Concentration in milligrams per liter.

### Data analysis

This study used a completely random factorial design with only one factor (one-way) using the MINITAB® 17 Statistical Software (Minitab Inc., State College, PA, USA) (2014), in which the variability within each dentifrice was randomly explained, that is, by the sum of the effects of several factors (methodological procedures and analysis parameters) that varied randomly. Through the analysis of variance statistical test, significant differences were observed among dentifrices. Thus, the Tukey's test was applied to determine which means were significantly different at 5% significance.

### Results

Table 2 shows the means and standard deviations of each product for pH, loss on drying and calcium concentration tests. The widest pH range was observed for dentifrice Colgate

**Table 2: Physicochemical characteristics of dentifrices (mean, SD and ANOVA with Tukey's test)**

Dentifrice	pH values	Loss on drying %	Calcium concentration g/kg
Colgate Máxima Proteção®	10.03±0.1528 <sup>a</sup>	40.87±0.231C <sup>d</sup>	153.33±11.61 <sup>a</sup>
Colgate Tripla Açã®	9.33±0.0577 <sup>c</sup>	58.14±0.226 <sup>b</sup>	132.63±1.55 <sup>a,b</sup>
Colgate Ultra Branco®	9.53±0.0577 <sup>b</sup>	39.25±0.140 <sup>d</sup>	129.38±6.44 <sup>b</sup>
Close Up®	9.20±0.0000 <sup>c</sup>	61.13±0.144 <sup>a</sup>	140.40±4.80 <sup>a,b</sup>
Even®	9.70±0.0000 <sup>b</sup>	36.80±0.143 <sup>e</sup>	155.55±2.64 <sup>a</sup>
Oral-B®	8.67±0.0577 <sup>d</sup>	33.81±0.214 <sup>f</sup>	152.78±16.51 <sup>a</sup>
Sorriso Dentes Brancos®	9.60±0.0000 <sup>b</sup>	42.94±2.058 <sup>c</sup>	149.95±1.61 <sup>a,b</sup>

Values showed statistically significant differences ( $P \leq 0.05$ ) among brands, which can be observed vertically, where different letters indicate significant differences among the different dentifrice brands for each test, so means that do not share a letter are significantly different ( $P < 0.05$ ) (one-way ANOVA with Tukey's test). ANOVA: Analysis of variance; SD: Standard deviation

Máxima Proteção® (10.03), with a significant difference from the other brands ( $P < 0.05$ ). On the other hand, the lowest value was found for dentifrice Oral-B® ( $P < 0.05$ ).

Regarding the loss on drying test, the highest values were found for dentifrices Colgate Tripla Açã® (58.14%) and Close Up® (61.13%), and the lowest for Oral-B® (33.81%) and Even® (36.80%). However, all products exhibited different loss on drying values in the statistical analyses ( $P < 0.05$ ).

The highest calcium concentrations were found for dentifrices Even® (155.55 g/kg), Colgate Máxima Proteção® (153.33 g/kg) and Oral-B® (152.78 g/kg), with no significant difference ( $P > 0.05$ ). However, the lowest concentration was found for dentifrice Colgate Ultra Branco® (129.38 g/kg), with differences in relation to the other groups ( $P < 0.05$ ).

### Discussion

Since the introduction of dentifrices, their formulations have developed considerably though their development is not yet complete and still has many challenges to overcome.<sup>[17]</sup> Thus, the evaluation of their physicochemical properties still requires a better understanding of their characteristics.

The pH results of the present study showed significant differences among brands tested; however, all dentifrices were within the safe range (from 4.5 to 10.5).<sup>[8]</sup> Thus, these results showed that the pH of all seven dentifrices was alkaline (pH > 7). It is noteworthy that among brands evaluated, Colgate Ultra Branco® and Sorriso Dentes Brancos®, with bleaching function, also showed alkaline pH.

A previous study analyzed the pH of 15 different brands in triplicate by means of a 6.25% suspension of each

dentifrice.<sup>[9]</sup> Most dentifrices showed alkaline pH; however, three dentifrices showed pH value below neutral. They were: Oral-B Dentes and Gengivas<sup>®</sup> (6.1) Kolynos Ação Total<sup>®</sup> (6.3) and Up Close com Fluor<sup>®</sup> (6.9). In the same study, it was found that dentifrices showed high abrasiveness, which is a feature that could indicate a possible combination of erosive and abrasive effect.<sup>[9]</sup>

Previous studies have evaluated the pH of dental bleaching products, including dentifrices. When evaluating 26 bleaching products, among them nine dentifrices, it was we found that the average pH was acidic (+6.83) and ranged from 4.22 to 8.35, suggesting that such dentifrices had an acidic profile, which is indicative of a possible cause of damage to the enamel surface.<sup>[18]</sup> However, in a similar study with 21 teeth whitening products, among them 7 whitening dentifrices, the average pH of these products was predominantly alkaline ( $7.66 \pm 1.19$ ), ranging from 6.61 to 9.68, highlighting the importance of the correct prescription by dentists.<sup>[19]</sup>

However, in another study with 3 different brands of whitening dentifrices, pH values ranged from 7.87 (Close-Up Whitening<sup>®</sup>) to 10.09 (Sorriso Dentes Brancos<sup>®</sup>), that is, all were alkaline.<sup>[11]</sup> However, in that study, the group that showed the highest pH (Sorriso Dentes Brancos<sup>®</sup>) also presented an extensive change in the enamel surface, although it has been advocated that alkaline pH has the tendency to cause fewer changes to the dental surface, while lower pH values can cause more damage to dental tissues,<sup>[11]</sup> emphasizing the need to characterize the physical and chemical properties of dentifrices for the understanding of their results and effectiveness.

Considering the property of loss on drying, the lowest loss was observed for Oral-B 123<sup>®</sup>. However, all brands evaluated showed significant differences. Previous findings evaluating the inorganic content of dentifrices showed that there was a large variation in the total percentage; however, a greater content of abrasives in the formula did not necessarily lead to increased abrasiveness.<sup>[9]</sup> However, it was confirmed that dentifrices containing calcium carbonate were less abrasive.<sup>[9]</sup>

In another loss on drying assessment evaluating three different dentifrices, both physical and chemical properties as their effect on the enamel surface, the following values were found: Sorriso Dentes Brancos<sup>®</sup> (31.91%), Close Up Whitening<sup>®</sup> (44.73%) and Sensodyne Branqueador<sup>®</sup> (65.83%). All values showed significant differences. However, Sorriso Dentes Brancos<sup>®</sup> was the only group that contained calcium carbonate in its composition, and in this specific study, it did not prove to be the least abrasive to the enamel surface.<sup>[11]</sup>

Thus, it is noteworthy that the loss on drying test indicated the amount of solid waste and can be associated with greater abrasiveness since high values represent sign of a possible

change on the enamel roughness. In addition, it could be inferred that for the loss on drying to influence the damage of the enamel surface, it should be considered together with other properties such as ash content, morphology and particle size.<sup>[9,11,20,21]</sup>

In the analysis of the calcium concentration values, the results showed differences among samples. The lowest concentration was found for Colgate Ultra Branco<sup>®</sup>, with significant difference, being thus the dentifrice with the lowest performance on this feature. However, the Tukey's test showed that dentifrices considered of better performance (Colgate Máxima Proteção<sup>®</sup>, Oral-B 123<sup>®</sup> and Even<sup>®</sup>) were statistically equal, showing no significant difference that could interfere in the choice of these products.

The lack of similar studies in literature evaluating the calcium concentration of dentifrices makes comparison with other studies difficult. It is necessary an extension of this type of analysis aiming at standardization that does not interfere with the concentration of fluoride ions and show a better benefit in enamel remineralization, being satisfactory to the consumer.

Thus, when evaluating the three properties studied in this research, the best performance would be of a dentifrice with the following features: pH within the safe range, as observed in all products tested; low average loss on drying, as in product Oral-B 123<sup>®</sup>; and higher calcium concentration, as presented by dentifrices Colgate Máxima Proteção<sup>®</sup>, Oral-B 123<sup>®</sup>, and Even<sup>®</sup>.

However, for being an *in vitro* research, the present study has some limitations, since it does not simulate important natural oral conditions such as buffering capacity of saliva, acquired pellicle formation, concentrations of calcium, phosphate and fluoride ions, dietary habits and other conditions inherent in the oral cavity.<sup>[22]</sup> Nevertheless, *in vitro* studies are widely used to evaluate the physicochemical properties of products such as dentifrices as it has the advantage of providing isolated data of variable of interest, avoiding confounding biases.

Thus, the values found cannot be directly extrapolated to the clinical situation; however, according to the tests performed, all products showed significant differences, which suggest the use of more specified criteria for their prescription. Nevertheless, the authors emphasize the need for further studies, including clinical or *in situ* trials to obtain higher levels of evidence on the evaluation of these properties.

## Conclusion

Fluoride dentifrices with calcium carbonate showed high pH values, loss on drying and calcium concentration. However, these physicochemical characteristics were differentiated according to the dentifrice brands tested.

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