

## Comparative Evaluation of the pH of Calcium Hydroxide Powder in Contact with Carbon Dioxide (CO<sub>2</sub>)

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This work involved an evaluation of calcium hydroxide powder in the absence and presence of CO<sub>2</sub>. 0.12g of calcium hydroxide powder was used for each of 16 aliquots diluted in 100 mL of deionized water and distributed in 2 samples of 8 aliquots. The indices of pOH, [OH<sup>-</sup>] and [Ca<sup>++</sup>] were obtained by mathematical calculations after determining the pH. The results demonstrated that in the presence of CO<sub>2</sub>, calcium hydroxide showed a marked loss of [OH<sup>-</sup>] and [Ca<sup>++</sup>] in relation to the decrease in pH. However, the high alkaline pH of the calcium hydroxide powder was preserved in the absence of CO<sub>2</sub>, maintaining its reparative and antimicrobial properties.

**Keywords:** *calcium hidroxide, ph evaluation, dioxide carbon.*

### 1. Introduction

Conservative and radical endodontic treatments use calcium hydroxide as a therapeutic support due to its action as a bactericide and inducer of mineralized tissue.

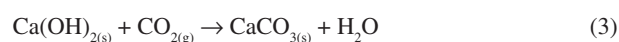
Pure calcium hydroxide was introduced in dentistry by Hermann in 1920<sup>6</sup>, aiming to find a remedy that offered the advantages of a strong antiseptic for the biological treatment of the pulp and for root canal dressings without the attending inconveniences. However, in 1940, it was Rohner<sup>15</sup> who presented the first histological work on human teeth that demonstrated the formation of a mineralized barrier at the root apex after pulpotomy and dressing of the root canals with Calxyl<sup>12</sup>.

Thus, among the substances that have been proposed for the direct coating of dental pulp, calcium hydroxide is undoubtedly the most effective. This drug is therefore the one most widely employed today as an intracanal medication, which, according to the literature, has withstood the tests of both research and time<sup>3,9</sup>.

Thus, from the chemical standpoint, calcium hydroxide is a base also known as an ionic compound, composed of a metal cation and a hydroxide anion. The use of this substance was reinforced because of its ionic dissociation into calcium and hydroxyl ions and the effect of these ions on tissues and microorganisms<sup>10,14</sup>.

Calcium hydroxide comes in the form of a white mass that transforms into oxides under heating. In aqueous solution, its solubility is 1.0 g in 630 mL of water, at a temperature of 25 °C, with solubility decreasing as the temperature rises<sup>8</sup>.

Estrela and Figueiredo<sup>13</sup> state that calcium hydroxide is a white alkaline (pH 12.8) powder with poor solubility in water (solubility of 1.2 g.L<sup>-1</sup> of water at 25 °C). It is a strong base obtained by calcining calcium carbonate until it transforms into calcium oxide (quicklime). Hydration of calcium oxide results in calcium hydroxide, and the reaction between the latter and carbon gas leads to the formation calcium carbonate. These reactions are represented by the following Equations 1, 2 and 3:



Based on the above, Tronstand, Andreassen, Hasselgren, Kristerson and Riis<sup>5</sup> evaluated the alterations in the pH of ape teeth after dressing their root canals with calcium hydroxide, and found that other factors besides pH are important in explaining the reparative effect of calcium hydroxide on inflammatory resorption. They reported that the presence of calcium ions is necessary for the activity of the complement system in the immune reaction and that an abundance of calcium ions activates calcium-dependent ATPase (adenosine triphosphatase), which is associated with the formation of hard tissue. Moreover, they pointed out that, due to its high pH, calcium hydroxide activates alkaline phosphatase and that the optimal

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pH for the activity of alkaline phosphatase, which varies according to the type and concentration of the substrate, the temperature, and the enzyme source, is situated between 8.6 and 10.3.

Alkaline phosphatase is a hydrolytic enzyme that acts by releasing inorganic phosphate from phosphate esters. This enzyme can separate phosphoric esters, releasing phosphate ions. Once released, these ions react with the calcium ions originating from blood circulation, forming a precipitate in the organic matrix, calcium phosphate, which is the molecular unit of hydroxyapatite<sup>4</sup>.

In addition, Estrela, Lopes and Fellipe Jr.<sup>11</sup> explains the action of calcium hydroxide on microorganisms based on a study of the biological effect of pH on enzyme activity. He states that the elevated pH of calcium hydroxide (12.6), influenced by the release of hydroxyl ions, is able to alter the integrity of the cytoplasmic membrane by chemical injury of the organic components and the transport of nutrients, or by destruction of phospholipids or unsaturated fatty acids of the cytoplasmic membrane. This is observed through the process of lipid peroxidation which, in reality, is a saponification reaction.

With regard to the effect of calcium hydroxide on bacterial lipopolysaccharide (LPS), Safavi and Nichols<sup>17</sup>, after an *in vitro* evaluation, concluded that calcium hydroxide hydrolyzes the toxic molecule of Lipid A which is responsible for the harmful effects of the endotoxin<sup>1</sup>.

In 2005, Vianna, Gomes, Sena, Zaia, Ferraz and Souza Filho<sup>18</sup> evaluated the antimicrobial activity of calcium hydroxide associated with different vehicles against endodontic pathogens, suggesting that its antimicrobial property is related both to the formulation of the paste and to microbial susceptibility.

Holland<sup>2</sup> stated that preference should be given to the use of pure calcium hydroxide of pro-analysis quality, stored in an amber jar filled with water, since calcium hydroxide in contact with air transforms into calcium carbonate, a substance that prevents the occurrence of the healing process, and that lime water is very useful for root canal irrigation. Estrela, Sydney, Bammann and Felipe Jr.<sup>10</sup> evaluated the presence of calcium carbonate in different samples of calcium hydroxide, based on the volume neutralization method using the indicators methyl orange and phenolphthalein titrated with chloric acid (0.0109 mol.L<sup>-1</sup>). The samples, which originated from different private endodontal clinics, had been in use for over 2 years. Their results indicated that the percentage of transformation of calcium hydroxide into calcium carbonate in the samples was small, varying from 5 to 11%.

In view of the above, the purpose of this work was to evaluate the pH of samples of calcium hydroxide powders from two different manufacturers, in contact with air.

## 2. Materials and Methods

### 2.1. Reagents

The reagents (calcium hydroxide) were purchased locally and deionized water was used throughout this study to dissolve the samples.

- Calcium Hydroxide P.A. (Manufacturer: Biodinâmica (Sample 1))
- Calcium Hydroxide P.A. (Manufacturer: PROBEM) (Sample 2)

### 2.2. Equipment / materials

- Marte MB – 10 pH meter.
- Amber glass flasks.
- Beakers.

The equipment used in this study is depicted in Figure 1.

Eight aliquots of 0.12 g of each sample were weighed and placed in amber glass flasks for the daily determination of pH, which was done over a period of 15 days. Each day, an aliquot (0.12 g) of each sample was dissolved in 100 mL of deionized water, covered and shaken. After 30 minutes at rest, the pH was read with the pH meter. The eighth aliquot of each sample was stored in an open glass beaker (unprotected from the air) for 15 days, after which each of these aliquots was dissolved, following the above described procedures.

The first two solutions analyzed at the beginning of the experiment (one of each sample) were stored in their own flasks, which remained closed for 7 days, after which the pH was measured again. On the second day of the experiment, the second aliquot of each sample was analyzed and left in the same flask, also for 7 days, followed by a new measurement of the pH.

An evaluation was also made of the transformation of calcium hydroxide into calcium carbonate based on pH readings and on calculation of the pOH and the concentration of [OH<sup>-</sup>] and of [Ca<sup>++</sup>], as shown in Tables 1, 2, 3 and 4. These evaluations involved readings of the pH of all the previously prepared aliquots, which were taken at 24 hour intervals for seven days for each aliquot of each sample. After seven days, readings were also taken of the pH of 2 aliquots that were left in open flasks, as well as of the other aliquots kept in closed flasks. The variation in pH was also measured after 15 days in 2 aliquots of each sample, which were stored in transparent beakers left open to the air.

## 3. Results

The pH values of the analyzed aliquots of the two samples of calcium hydroxide are listed in Tables 1 and 2.

As can be seen from the results listed in Table 1, the variation of the pH of each aliquot of sample 1 was very small. This variation is plotted in the graph in Figure 1.

The data obtained for the aliquots of sample 2 did not differ significantly from those of sample 1, showing a very similar variation, as can be seen in Figure 1.

After 7 days, the solution of the aliquot of sample 1, which was stored in a closed flask on the first day of the experiment, showed a pH of 12.18. Aliquot 2 of the same sample, when dissolved for determination of the pH, remained uncovered for 7 days, after which time the measured pH was 11.84 (Table 3).

Aliquots 1 and 2, which were subjected to the same procedures, presented the following results: aliquot 1 (in closed solution), pH of 12.22, and aliquot 2 (in open solution), pH of 11.84 (Table 4).

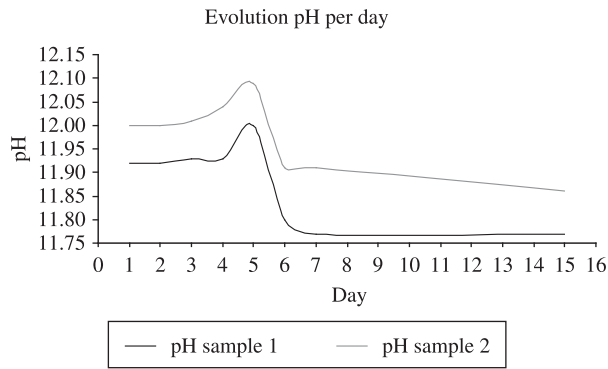
The eighth aliquot of each sample was kept in a glass beaker for 15 days (without protection from light and air). After this period, each aliquot was dissolved, shaken, allowed to rest for 30 minutes, and its pH measured, yielding the results listed in Tables 1 and 2.

- 8<sup>th</sup> aliquot (sample 1).....pH = 11.77.
- 8<sup>th</sup> aliquot (sample 2).....pH = 11.86.

## 4. Discussion

Calcium hydroxide is a white mass, which, according to Silva<sup>8</sup>, transforms into oxides under heating, presenting a solubility of 1.0 g.630 mL in water at room temperature, with this solubility decreasing as the temperature increases. However, in our work, we found that the solubility of the two samples of calcium hydroxide was 1.2 g.1L<sup>-1</sup> of water, which is consistent with the data reported by Estrela and Figueiredo<sup>13</sup>.

A report by Holland (1974) stated that calcium hydroxide transforms into calcium carbonate when in contact with air. In view of this fact, the same author proposed that calcium hydroxide should be stored in an amber glass bottle and steeped in an aqueous solu-



**Figure 1.** Variation of pH in the aliquots of samples 1 and 2

**Table 1.** pH of the aliquots of sample 1.

Sample 1 portions (g)	Day	pH	pOH	[OH <sup>-</sup> ]	[Ca <sup>++</sup> ]
1 - 0.12	1	11.92	2.08	$8.32 \times 10^{-3}$	$4.12 \times 10^{-3}$
2 - 0.12	2	11.92	2.08	$8.32 \times 10^{-3}$	$4.16 \times 10^{-3}$
3 - 0.12	3	11.93	2.07	$8.51 \times 10^{-3}$	$4.25 \times 10^{-3}$
4 - 0.12	4	11.93	2.07	$8.51 \times 10^{-3}$	$4.25 \times 10^{-3}$
5 - 0.12	5	12.00	2.00	$1.00 \times 10^{-2}$	$0.50 \times 10^{-2}$
6 - 0.12	6	11.80	2.20	$6.31 \times 10^{-3}$	$3.15 \times 10^{-3}$
7 - 0.12	7	11.77	2.23	$5.90 \times 10^{-3}$	$2.95 \times 10^{-3}$
8 - 0.12	15	11.77	2.23	$5.90 \times 10^{-3}$	$2.95 \times 10^{-3}$

**Table 2.** pH of the aliquots of sample 2.

Sample 2 aliquots (g)	Day	pH	pOH	[OH <sup>-</sup> ]	[Ca <sup>++</sup> ]
1 - 0.12	1	12.00	2.00	$1.00 \times 10^{-2}$	$0.50 \times 10^{-2}$
2 - 0.12	2	12.00	2.00	$1.00 \times 10^{-2}$	$0.50 \times 10^{-2}$
3 - 0.12	3	12.01	1.99	$1.02 \times 10^{-2}$	$0.51 \times 10^{-2}$
4 - 0.12	4	12.04	1.96	$1.10 \times 10^{-2}$	$0.55 \times 10^{-2}$
5 - 0.12	5	12.09	1.91	$1.23 \times 10^{-2}$	$0.61 \times 10^{-2}$
6 - 0.12	6	11.91	2.09	$8.13 \times 10^{-3}$	$4.06 \times 10^{-3}$
7 - 0.12	7	11.91	2.09	$8.13 \times 10^{-3}$	$4.06 \times 10^{-3}$
8 - 0.12	15	11.86	2.14	$7.24 \times 10^{-3}$	$3.62 \times 10^{-3}$

tion to render it usable. However, using the volume neutralization method, Estrela, Sydney, Bammann and Felipe Jr.<sup>10</sup> found that the transformation of calcium hydroxide into carbonate was small (5 to 11%), based on an evaluation of the presence of calcium carbonate in samples of calcium hydroxide obtained from professionals that had been using it for a period of more than 2 years. Unlike the findings of the latter author, in our work we obtained a variation of 19 to 29% and of 28 to 29% in samples 1 and 2, which were left uncovered and in direct contact with CO<sub>2</sub> for 7 and 15 days, respectively.

After collecting the data, we found that up to the 5<sup>th</sup> day, all the aliquots of both samples that were kept in open amber flasks showed a discrete increase in pH. After that period, we recorded a slight decrease in the pH of the remaining aliquots. In the aliquots of each sample that were stored in closed flasks, there was a discrete increase in the values of pH, while those that were stored in transparent beakers open to the air showed a slight decrease in pH values after 15 days.

**Table 3.** pH of aliquots 1 and 2 of sample 1 after seven days.

Sample 1 aliquots (g)	Days	pH	pOH	[OH <sup>-</sup> ]	[Ca <sup>++</sup> ]
1 - 0.12	7°	12.18	1.82	$1.51 \times 10^{-2}$	$0.75 \times 10^{-2}$
2 - 0.12	7°	11.84	2.16	$6.91 \times 10^{-3}$	$3.45 \times 10^{-3}$

**Table 4.** pH of the aliquots of samples 1 and 2 after seven days.

Sample 2 aliquots (g)	Day	pH	pOH	[OH <sup>-</sup> ]	[Ca <sup>++</sup> ]
1 - 0.12	7°	12.22	1.78	$1.65 \times 10^{-2}$	$0.83 \times 10^{-2}$
2 - 0.12	7°	11.84	2.16	$6.91 \times 10^{-3}$	$3.45 \times 10^{-3}$

In a study of the changes in pH on the surface of root dentin after dressing with calcium oxide and calcium hydroxide, Minäna, Carnes and Walker<sup>16</sup> state that the pH declined significantly after exposure to CO<sub>2</sub>.

However, our data seem to show better agreement with the statements of Holland<sup>2</sup>, to the effect that when in contact with air, calcium hydroxide transforms into calcium carbonate. Nevertheless, our results indicate that this transformation does not affect the healing properties of this compound for at least 15 days, although these properties can be affected when contact with air occurs over a long period.

Therefore, our results seem to reinforce the findings of Tronstand, Andreassen, Hasselgren, Kristerson and Riis<sup>5</sup> about the alterations in pH in ape teeth after the root canals were dressed with calcium hydroxide. These authors found that the presence of calcium ions is necessary for the activity of the complement system in the immune reaction, and that the abundance of calcium ions activates calcium-dependent ATPase (adenosine triphosphatase), which is associated with the formation of hard tissue. They also stated that calcium hydroxide activates alkaline phosphatase due to its high pH and that the optimal pH for the activity of alkaline phosphatase, which varies according to the type and concentration of substrate, the temperature, and the enzyme source, is around 8.6 to 10.3.

Estrela, Lopes and Fellipe Jr.<sup>11</sup> explains the action of calcium hydroxide on microorganisms based on a study of the biological effect of pH on enzyme activity. According to this author, in response to the release of hydroxyl ions, the elevated pH of calcium hydroxide (12.6) is able to alter the integrity of the cytoplasmic membrane by chemical injury of the organic components and the transport of nutrients, or by destruction of phospholipids or unsaturated fatty acids of the cytoplasmic membrane revealed by the process of lipid peroxidation, which is actually a saponification reaction. However, our findings indicate that the concentration of [OH<sup>-</sup>] and [Ca<sup>++</sup>] is already high up to 15 days, compared with a negligible loss of pH when in contact with CO<sub>2</sub>. This leads us to believe that there is also a decrease in its beneficial properties.

## 5. Conclusions

The analysis of four results lead us to the following conclusions:

- 1) The values of pH in aqueous solutions and in calcium hydroxide powder stored in closed amber flasks showed a discrete increase compared to those stored in transparent beakers open to the air for up to 15 days;
- 2) The values of pH in aqueous solutions and in calcium hydroxide powder stored in open amber flasks showed a discrete decrease

compared to those stored in transparent beakers open to the air for up to 15 days;

- 3) According to the data obtained with our methodology and compared with the pertinent literature, calcium hydroxide maintains its tissue healing and antimicrobial properties provided it is stored away from contact with CO<sub>2</sub>.

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