Calcium Hypochlorite Solutions: Evaluation of Surface Tension and Effect of Different Storage Conditions and Time Periods over pH and Available Chlorine Content

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Abstract

Introduction: The aim of this study was to evaluate the pH and the available chlorine content from sodium hypochlorite (NaOCl) and calcium hypochlorite ($Ca[OCl]_2$) solutions stored in different conditions and time periods and the surface tension of $Ca(OCl)_2$ solutions in comparison with NaOCl. **Methods:** Solutions at 0.5%, 1%, 2.5%, and 5.25% concentrations were prepared. The pH level and the available chlorine content of freshly prepared solutions and solutions stored for 30, 60, and 90 days at 25°C, 4°C, or 37°C were evaluated in a digital pH meter and by titration, respectively. Surface tension was tested using a Du Nouy tensiometer (Sigma 702, Force Tensiometer; Attension, Espoo, Finland). Descriptive and inferential statistical analyses were performed. **Results:** A precipitate formed by 2.5% and 5.25% $Ca(OCl)_2$ solutions was observed. $Ca(OCl)_2$ showed a higher concentration of available chlorine than NaOCl. Both 2.5% and 5.25% NaOCl and $Ca(OCl)_2$ had a decrease in the available chlorine content when compared with freshly prepared solutions; 0.5% and 1% NaOCl tend to have a lower pH compared with 0.5% and 1% $Ca(OCl)_2$. NaOCl, 5.25%, showed higher pH compared with 5.25% $Ca(OCl)_2$. NaOCl and $Ca(OCl)_2$ in 0.5% and 1% concentrations tend to show a reduced pH level, whereas 2.5% and 5.25% solutions showed an increase in pH. The heat contributed to the instability of the solutions. NaOCl showed lower surface tension values than $Ca(OCl)_2$. **Conclusions:** $Ca(OCl)_2$ solutions are extremely alkaline and tend to have more available chlorine content than NaOCl but have a higher surface tension than NaOCl. Regarding the available chlorine content, these solutions tend to be stable to 30 days of storage when kept at 4°C or at 25°C. *(J Endod 2016;42:641–645)*

Key Words

Calcium hypochlorite, chlorine content, pH, sodium hypochlorite, surface tension

Disinfection of the root canal system is complex, especially because of the anatomic factors. Several areas of the root canal remain untouched during chemomechanical preparation (1). Therefore, the use of irrigants and auxiliary chemical solutions should be associated with instruments during endodontic therapy, promoting cleaning and shaping.

Several irrigants have been used during endodontic treatment; sodium hypochlorite (NaOCl) is the most widely used (2). NaOCl is a strong base, with a pH around 11 (3). The strong alkalinity can be associated with its ability to dissolve organic tissues (4), and its properties can be assessed by the amount of active available chlorine (5). The concentration of NaOCl solutions needs to be sufficient to guarantee their antimicrobial and solvent effects.

On the other hand, their chemical instability, enhanced by exposure to heat, light, and air (5), can be considered critical, indicating a reduction in the NaOCl shelf life. Furthermore, NaOCl has a relatively high surface tension (6). This characteristic may limit the penetration of the solution into canal irregularities and deep into dentinal tubules (7). Therefore, studies should be conducted to test irrigants that have similar properties to NaOCl solutions, such as tissue dissolution and antibacterial activity, with an improved stability and low surface tension.

Calcium hypochlorite ($Ca[OCl]_2$) is a white powder used for industrial sterilization, bleaching, and purifying water treatment (8). It is relatively stable and has greater available chlorine compared with NaOCl (9). Its incorporation in water can be more accurate than preparations by dilution of a more concentrated solution, which can be an advantage for clinical use. According to Dutta and Saunders (9), there is no difference in bovine muscle tissue dissolution among 5.25% NaOCl and 5% or 10% $Ca(OCl)_2$ solutions after 35 to 60 minutes of testing. More recently, de Almeida et al (10) showed that $Ca(OCl)_2$ associated with ultrasonic irrigation is efficient to reduce root canal contamination and can aid in chemomechanical preparation.

However, there is a lack of consistent information on the chemical properties of $Ca(OCl)_2$ solutions and their different concentrations. The aim of this study was to evaluate pH and the available chlorine content from NaOCl and $Ca(OCl)_2$ solutions stored in different conditions and time periods and $Ca(OCl)_2$ surface tension. The null hypothesis tested in this study was that there is no difference in surface tension between hypochlorite solutions and in pH and the available chlorine content between NaOCl and $Ca(OCl)_2$ tested at the same concentration, considering the same storage condition and period.

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Materials and Methods

This study was approved by the research board from the Dental School, Federal University of Rio Grande do Sul, Porto Alegre, Rio Grande do Sul, Brazil (protocol number 25157/2012 and protocol number 27789/2014).

Solution Preparation and Storage Condition

The NaOCl and $Ca(OCl)_2$ solutions were prepared immediately before the experiments. A 12% NaOCl solution (Farmaquímica SA Produtos Químicos, Porto Alegre, Brazil) was titrated, and then it was diluted in sterilized and distilled water to reach the concentrations of 0.5%, 1%, 2.5%, and 5.25%.

$Ca(OCl)_2$ powder (Farmaquímica SA Produtos Químicos) with 65% purity was weighted in a precision balance (Sartorius AG, Gottingen, Germany). The powder was mixed with distilled and sterilized water as follows:

1. $Ca(OCl)_2$ 0.5%: 0.1153 g $Ca(OCl)_2$ powder + 15 mL sterile distilled water
2. $Ca(OCl)_2$ 1%: 0.2307 g $Ca(OCl)_2$ powder + 15 mL sterile distilled water
3. $Ca(OCl)_2$ 2.5%: 0.5769 g $Ca(OC_l)_2$ powder + 15 mL sterile distilled water
4. $Ca(OCl_{)2}$ 5.25%: 1.2115 $Ca(OCl)_2$ powder + 15 mL sterile distilled water

All the solutions were prepared under constant agitation. After total powder dissolution, the solutions were filtered twice to remove debris. For storage, 15-mL plastic tubes were wrapped with aluminum foil. The solutions were maintained closed in the tubes. Individual plastic tubes were used for each test solution. All the solutions were stirred before analysis.

The solutions were stored at 4°C in a refrigerator, at 37°C in a microbiological incubator, and at room temperature (average equal to 25°C) inside a cupboard for different periods (30, 60, and 90 days).

pH Measurement

The pH of the solutions immediately after preparation (control solution) and in each storage condition and period was analyzed using a digital pH meter (Digimed, São Paulo, SP, Brazil). All experiments were performed in triplicate.

Determination of the Available Chlorine Content

The available chlorine content was evaluated by titration according to Pécora et al (11). Three analyses were performed for each sample. The result was expressed in grams of active chlorine per 100 mL solution.

Surface Tension Test

Surface tension was evaluated by the "ring method." The Du Nouy tensiometer (Sigma 702, Force Tensiometer; Attension, Espoo, Finland) was used in a room with a constant temperature (25°C).

Distilled water was used as the control and to perform an initial calibration of the instrument. The surface tension was expressed in mN/m with Huh-Mason correction. Nine measurements were performed for each solution.

Data Analysis

Statistical analysis was performed in BioEstat 5.0 (Fundação Mamirauá, Belém, PA, Brazil). The level of significance was 5%. The *t* test was used to compare data of pH and the available chlorine content between solutions with the same concentration, considering the same storage temperature and period, and to compare pH and the available chlorine content in solutions stored for 30, 60, and 90 days with immediately prepared solutions. For the surface tension test, results were expressed as means and standard deviations, and analysis of variance with the Tukey post hoc test was used.

Results

The 2.5% and 5.25% $Ca(OCl)_2$ solutions had a white precipitate that was observed over time in the bottom of the plastic tube, before evaluation. Morphology of the particles was observed with scanning electron microscopy (JSM-6010LA; JEOL, Peabody, MA). Their composition was measured with energy-dispersive X-ray spectroscopy (JSM-6010LA), which enabled detection of the individual elements of the particles that were formed predominantly by calcium (Fig. 1*A* and *B*).

Figure 2 shows the results for the available chlorine content in different storage conditions. $Ca(OCl)_2$ solutions showed a higher concentration of active chlorine compared with NaOCl with the same concentration, in the same storage condition, and in the same time period ($P < .05$), except for the 0.5% solution when stored at room temperature for 30 days and at 37°C for 60 and 90 days ($P > .05$). The available chlorine content of both 0.5% NaOCl and 0.5% $Ca(OCl)_2$ solutions stored at 4°C for 90 days was not different from the immediately prepared solutions. However, there was a decrease in the chlorine content for 2.5% and 5.25% solutions after 60 and 90 days of storage, especially at 25°C and 37°C. Both NaOCl and $Ca(OCl)_2$ freshly prepared solutions were alkaline. Freshly prepared 0.5% and 1% NaOCl solutions

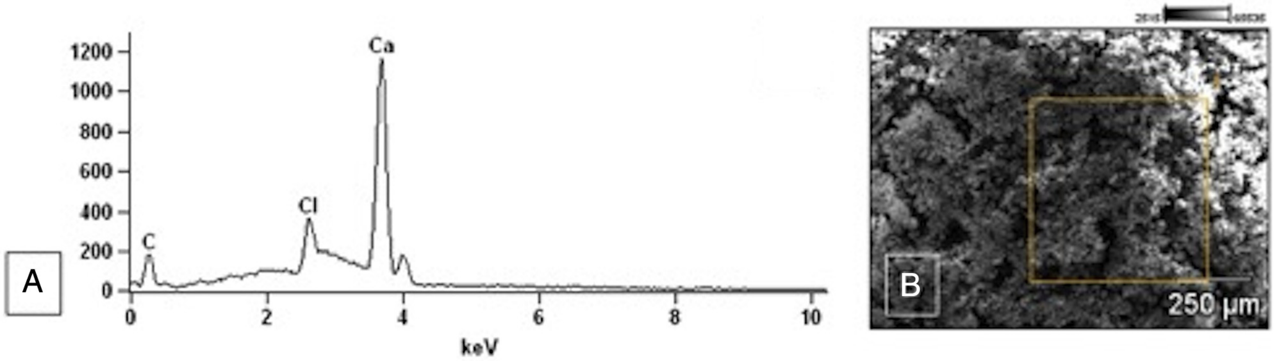

Figure 1. Energy-dispersive X-ray spectroscopy analysis. (*A*) Particle composition obtained with energy-dispersive X-ray spectroscopy and (*B*) morphologic analysis obtained with scanning electron microscopic images of $Ca(OCl)_2$ (×100, 20 kV).