

## Original Research Article

# Interference of the assessment method in pH values of an epoxy-based cement

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## Abstract

**Introduction:** Alkalinization potential is a fundamental property of endodontic epoxy-based cements containing calcium hydroxide. Studies have shown discrepant pH results for same materials at different evaluation periods. A possible reason accounting for these differences may be the assessment procedures. **Objective:** To evaluate the pH value of an epoxy-based cement (Sealer 26) in different periods of analysis, using two assessment methods. **Material and methods:** Sealer 26 was manipulated and immediately placed into polyethylene tubes (n=10, each group) and immersed in distilled water. In G1, the tubes were kept in the same water during all experiment; and in G2, the tubes were removed and placed into another flask with an equal amount of water after the pH evaluation. The pH of these solutions was measured at 24 hours, 7, 14 and 28 days. Analysis were made within the same group according to the experimental periods and between groups in each experimental period. Data were submitted to ANOVA ( $\alpha = 5\%$ ) and *t* test, respectively. **Results:** For G1 and G2, all periods

showed different pH values ( $p < 0.05$ ), except between 14 and 28 days ( $p > 0.05$ ) and between 7 and 14 days ( $p > 0.05$ ), respectively. In each period, no significant differences were observed between the groups. **Conclusion:** The method to obtain the pH values in different experimental periods no interfered in the final results. However, difference was observed when the results were analyzed at same group.

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## Introduction

Endodontic cements should have both antimicrobial action [15] and the capacity to stimulate the healing of periapical tissue [9]. Antimicrobial action is related to the release of hydroxyl ions [4], increasing pH and creating an unsuitable environment for microbial growth [6].

The pH of a solution or substance is usually measured with a specific device (pHmeter) using the electrode directly into the substances [13] or indirectly in the solutions in which the specimens were immersed [8]. Sometimes, in the analysis with the same material, in similar conditions of manipulation and at the same period of analysis, different values are obtained, as observed in studies with MTA-based cements [5, 14]. Possible reasons to explain these results could be either different procedures to analyze the pH changing or the distilled water in which the specimens remained immersed, but no study has confirmed these hypotheses.

Sealer 26 cement is a root canal filling material commonly used in Brazil. According to the manufacturer, the main constituents of Sealer 26 are: a) powder (bismuth trioxide, calcium hydroxide, hexamethylene tetramine, titanium dioxide) and b) resin (Bisphenol-epoxy resin). Silva *et al.* (1997) [11] observed that the highest pH values were obtained for Sealapex in comparison with CRCS, Apexit and Sealer 26. Duarte *et al.* (2000) [3] obtained similar results, except that Sealer 26 showed highest pH during the initial periods. However, after its final setting, Sealer 26 pH decreased its pH [2].

Santos *et al.* (2005) [10] recommended the specimens change into another flask with an equal amount of distilled water to avoid saturation of the medium. On the other hand, Gonçalves *et al.* [5] assessed the pH without liquid substitution, because other reactions may occur, such as the formation of different substances involving hydroxyl ions ionized from  $\text{Ca}(\text{OH})_2$  and interfere with the real pH values of the cement [11].

The aim of this study was to evaluate the interference of the method for obtaining the pH values of the epoxy-based cement (Sealer 26), at 24 h, 7, 14, and 28 days, within the same group and between each experimental period. The null hypothesis is that the method influences pH value assessment, within the same group throughout the experiment and between the groups at each evaluation period.

## Material and methods

Throughout the experiment, Sealer 26 (Dentsply Ind Com Ltda, Rio de Janeiro, RJ, Brazil) was prepared according to the manufacturers' ratio recommendation: 2.0 g powder: 1.0 g resin [3]. Twenty polyethylene tubes measuring 0.5 cm length and 1.0 mm internal diameter were filled with Sealer 26 (Dentsply) using a Lentulo spiral (Maillefer, Ballaigues, Switzerland). The tubes filled with fresh cement were weighed in order to check the standardization of the amount of cement.

In sequence, the specimens were divided into two groups ( $n = 10$ ), according to pH assessment method: (G1) the tubes were kept into the same distilled water throughout the experiment; (G2) the tubes were removed and placed into another flask with an equal amount of distilled water after the pH evaluation.

They were placed into polypropylene closed flasks (Injeplast, São Paulo, SP, Brazil) containing 10 mL of distilled water and kept in oven at 37°C (Farmen, São Paulo, SP, Brazil). Prior to the specimens' immersion, the distilled water showed a pH of 6.8. At 3 hours, all specimens of G2 were placed into another flask with the same volume of new distilled water. The specimens of G1 were kept into the same distilled water. Measurement of pH was performed with a pHmeter (model DM22, Digimed, São Paulo, SP, Brazil), previously calibrated with solutions of known pH (4, 7 and

14), at constant room temperature (25°C), after 24 hours, 7, 14 and 28 days. After removal of the specimens, the flasks were placed in a shaker (251, Farmen) for 5 seconds before pH measurement. Control sample comprised pH measuring of the water in which no specimens had been immersed. Each group data at the several periods were analyzed by one-way ANOVA ( $\alpha = 5\%$ ). *t* test ( $\alpha = 5\%$ ) was used to compare between groups at each period

## Results

Table I shows the pH values provided by the tested groups (G1 and G2) at 24 hours, 7, 14 and 28 days of immersion. In both groups, there was a decreased of pH value during the experiment. In G1, pH value was different at all periods, except between 14 and 28 days. In G2, pH value was different at all the periods, except between 7 and 14 days. There was no difference at each experimental period between the groups (table I).

**Table I** - Mean and standard deviation of pH values, in function of the period of analysis and experimental groups

	24 h	7 days	14 days	28 days
G1	9.98 (0.12) <sup>Aa</sup>	8.74 (0.52) <sup>Ab</sup>	8.14 (0.43) <sup>Ac</sup>	8.10 (0.46) <sup>Ac</sup>
G2	10.10 (0.13) <sup>Aa</sup>	8.73 (0.49) <sup>Ab</sup>	8.46 (0.47) <sup>Ab</sup>	7.58 (0.41) <sup>Ac</sup>

<sup>A</sup> Similar letter in each column indicate similar results ( $p > 0.05$ ); <sup>a,b</sup> Different letters in each line indicate significant different results ( $p < 0.05$ )

## Discussion

The method to assess the pH values of Sealer 26 cement no interfered in the final results. When the specimens are kept into the same distilled water there was a tendency to stabilize the pH after 14 days. On the other hand, when the specimens are placed into new distilled water the differences tended to occur regardless of the period of immersion. However when the groups were compared at each period, no statistical differences were found. Thus, the null hypothesis is rejected. The maintenance of the specimens in the same water should be performed when one aims to establish the balance of pH values. When the aim is to assess the pH value at an each specific time period, it is recommended to place the specimens into new amount of distilled water.

Several methods to assess the pH of endodontic cement have been proposed [2, 3, 8, 14]. Notwithstanding, the results interpretation of the different methods are controversial and conflicting. Small modifications at the moment of obtaining the pH value may lead to variable results. When the specimen is kept into the same solution during all experiment, at a determinate moment, there is a tendency toward balance because of the saturation process of the solution [11]. This method enables to check whether other reactions may occur, such as the formation of different substances involving

hydroxyl ions ionized by the calcium hydroxide-containing cement (Sealer 26) [11], mainly during the setting time of the cement [2], which directly interferes in the pH values [3, 11].

The advantage of placing the specimens into other flasks during the experiment is the possibility of quantifying the hydrogen ion released at each period [10] and to prevent the distilled water saturation [10]. This method was used in several studies [3, 7, 8, 10, 12]. In our study, after 3 hours of the beginning of the experiment, the specimens were removed from the initial flasks and placed into new distilled water. This procedure was intended to differentiate G2 from G1 group, at 24 hours period.

This study used as reference the epoxy-based cement (Sealer 26). Sealer 26 (Dentsply) contains calcium hydroxide in composition (0.370 g/g cement), and demonstrates favorable biological behavior [1]. The presence of some acid-hydrogen components in the cement, as bisphenol-epoxy, may have reacted with hydroxyl ion causing a pH decrease [11], which was mainly detected in G1 group after 14 days.

The results obtained in this present study cannot be fully transferred to clinical application because clinical conditions may interfere in the results. Further clinical and laboratorial studies should be carried out regarding to pH values of the Sealer 26 cement.

## Conclusion

pH assessment procedures of the materials no influenced in the final results. The method of specimens' changing into new distilled water provides more realistically values of hydrogen ion release, at each analysis period; on the other hand, the specimens' maintenance into the same distilled water allows visualization of pH values behavior throughout the experiment.

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