UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

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Development and analysis of two cements for employment in retrograde filling and sealing of perforations

Desenvolvimento e análise de dois cimentos para emprego em obturações retrógradas e selamento de perfurações

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Orientador: Prof. Dr. Marco Antonio Hungaro Duarte

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Com todo meu carinho e respeito, amo vocês.

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ABSTRACT

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The objective of this study was to develop and analyze the physical-chemical properties and dental color changes of two experimental and retrofiller cements to be used in perforations, containing different radiopacifiers and compare them to the properties of MTA HP and MTA Angelus. The cements were divided into 6 groups: I - MTA Angelus; II - HP MTA; III - Experimental 1 with powder (60% calcium silicate, 10% calcium phosphate, 30% zirconium oxide) and liquid (80% water and 20% arnica extract); IV - Experimental 2 with powder (60% calcium silicate, 10% calcium phosphate, 30% calcium tungstate) and liquid (80% water and 20% arnica extract); V - Experimental 3: silicone cement (Roeko Seal) + 20% calcium silicate + 10% zirconium oxide and VI - Experimental 4: silicone cement (Roeko Seal) + 20% calcium silicate + 10% calcium tungstate. For the accomplishment of the tests of radiopacity, flow and time of prey were followed the ISO 6876/2001 specifications and ASTM C266/2008. pH was determined by a pH meter previously calibrated. To determine the release of calcium ions, an atomic absorption spectrophotometer was used. The volumetric change was measured by Micro-CT. For the evaluation of tooth discoloration, 10 bovine teeth were used for each group. The teeth had the pulp chambers cleaned and filled with the cements. After the periods of 7, 30 and 60 days specimens were submitted to spectrophotometry. The data were statistically compared using significant level ($p<0,05$). All experimental materials tested showed radiopacity above the recommended minimum (3 mm / Al). Groups G5 and G6 showed a higher flow rate. All materials provided alkalization of the water in which they were immersed after 3 days, except groups G5 and G6. Regarding prey time, there were no significant differences in the comparisons between G2 and G3 and between G5 and G6. In the 3-day period, G1 stood out as the group that released more calcium ions, obtaining a statistically significant difference when compared to the others, except for G2. When we compare the color variation for the three periods, it was noticed statistically stable values for groups G2, G3 and G4. It is concluded that the experimental cements based on calcium silicate showed more favorable properties, such as lower flow, alkalization of the medium, higher working time and color stability.

Keywords: Retrograde Obturation, Dental Cements, MTA

RESUMO

RESUMO

O objetivo deste estudo foi desenvolver e analisar, quanto às propriedades físico-químicas e alteração de cor, os cimentos experimentais retro-obturadores e para serem empregados em perfurações, contendo diferentes radiopacificadores e por fim os comparar com as propriedades do MTA HP e MTA Angelus. Os cimentos foram divididos em 6 grupos: I - MTA Angelus; II - MTA HP; III - Experimental 1 com pó (60% silicato de cálcio, 10% fosfato de cálcio, 30% óxido de zircônio) e líquido (80% água e 20% extrato de arnica); IV - Experimental 2 com pó (60% silicato de cálcio, 10% fosfato de cálcio, 30% tungstato de cálcio) e líquido (80% água e 20% extrato de arnica); V - Experimental 3: cimento de silicone (Roeko Seal) + 20% de silicato tricálcio + 10% óxido de zircônio e VI - Experimental 4 cimento silicone (Roeko Seal) + 20% de silicato tricálcio + 10% de tungstato de cálcio. Para a realização dos testes de radiopacidade, escoamento e tempo de presa foram seguidas as especificações ISO 6876/2001 e ASTM C266/2008. A determinação do pH foi realizada por meio de um pHmetro previamente calibrado com soluções de pHs conhecidos. Para a determinação da liberação de íons cálcio, foi utilizado um espectrofotômetro de absorção atômica. A alteração volumétrica foi mensurada por Micro-CT. Para avaliação da descoloração dental foram utilizados 10 dentes bovinos para cada grupo de cimento. Os dentes tiveram as câmaras pulpares limpas e obturadas com os cimentos. Após os períodos de 7, 30 e 60 dias os dentes foram submetidos à espectrofotometria. Os dados foram comparados estatisticamente empregando nível de significância ($p<0,05$). Todos os cimentos experimentais apresentaram radiopacidade acima do mínimo recomendado (3 mm/Al). Os grupos G5 e G6 apresentaram uma taxa de escoamento maior. Todos os materiais proporcionaram alcalinização da água na qual foram imersos após 3 dias, exceto os grupos G5 e G6. Na análise do tempo de presa, não houve diferenças significantes nas comparações entre G2 e G3 e entre G5 e G6. No período de 3 dias, G1 se destacou sendo o grupo que mais liberou ions cálcio, obtendo diferença estatisticamente significante quando comparado aos outros, exceto pelo G2. Quando comparamos a variação de cor para os três tempos, podemos notar valores estatisticamente estáveis para os grupos G2, G3 e G4. Conclui-se que os cimentos experimentais à base de silicato cálcio demonstraram propriedades mais favoráveis, como menor escoamento, alcalinização do meio, maior tempo de trabalho e estabilidade de cor.

Palavras-chave: Obturação retrograda, Cimentos Dentários, MTA.

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1 INTRODUCTION

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When the treatment of the root canal system is compromised coronally and, in addition to other factors such as accidents, anatomical complexities and persistent infections, the failure rate of endodontic treatment increases, and parendodontic surgery with apical access is required (Molven et al., 1991).

An endodontic cement to be used as repairer in perforations and retrograde fillings must have certain biological and physicochemical properties so that its use becomes viable, such as prey time, radiopacity, low volumetric change, not a very high flow, pH, release of calcium ions, among other characteristics. Also, these cements should not be irritating to adjacent tissues and provide adequate three-dimensional sealing (Grossman et al., 1976).

The function of a retrofiller cement is to provide an apical root seal, sealing microorganisms and bacterial products away. This objective is achieved by preparing a cavity at the root and then filling it with a biocompatible material (Abedi et al., 1995).

A wide variety of materials have been used to seal the communication pathways between the root canal system and the oral cavity as well as the periradicular tissues. These include amalgam, zinc oxide and eugenol cements such as Super-EBA (Harry J. Bosworth Co., Skokie, IL) and IRM (LD Caulk Co., Milford, DE), Cavit (ESPE America, Norristown, PA) composite resins and glass ionomer cements. The main disadvantages of these materials include microleakage and sensitivity to moisture (Torabinejad et al., 1995).

The mineral trioxide aggregate (MTA) was developed in the 1990s to seal perforations between the root canal system and the external surface of the tooth (Lee et al., 1993). Due to it biological properties, low solubility and good marginal sealing ability (Torabinejad et al., 1997), MTA has been indicated as a gold standard material as a retro filler cement in endodontic surgery. Several authors have reported that MTA is similar to Portland cement (Holland et al., 1999, Wucherpfenning and Green 1999, Estrela et al., 2000, Holland et al 2001a, b) and was first available as ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK, USA). In 2001, a new cement was launched commercially labeled MTA-Angelus (Angelus Soluções Odontológicas, Londrina, Brazil, being composed of 80% of Portland cement and 20% of bismuth oxide. (Duarte et al., 2003). Nowadays, the radiopacifier bismuth oxide has been replaced to calcium tungstate due to the color change caused by bismuth. To improve its characteristics, in 2016 Angelus has developed a new formulation called MTA REPAIR HP. This new formula maintains all the chemical and biological properties of the original MTA, which guarantees the success of the treatment, but changes its radiopacifier to calcium tungstate and also added a plasticizer to its liquid, resulting in a better handling (Angelus Indústria de Produtos Odontológicos, 2014).

The main components of the MTA are tricalcium oxide, tricalcium silicate, bismuth oxide, tricalcium aluminate, tricalcium oxide, tetracalcium aluminoferrate and silicate oxide. Along with, there are some other mineral oxides responsible for the chemical and physical properties of the MTA. The powder consists of fine hydrophilic particles that form a colloidal gel in the presence of water and then solidifies, leading to the prey of the material. MTA presents some disadvantages as the difficulty in its manipulation, due to its dryness, very low flow and low antimicrobial action (Torabinejad et al., 1995).

Due the necessity of a cement with ideal properties for endodontics surgery or to be used in perforations, the objective of this study was to develop two types of cements, one based on calcium silicate and another one with the addition of this same component to a silicone cement available in the dental materials market, and to analyze the physicochemical properties of these materials comparing them to MTA White Angelus and MTA HP employed as retrograde obturator and perforation sealer.

2 ARTICLE

2 ARTICLE

The article presented in this Dissertation was formatted according to the Journal of Endodontics instructions and guidelines for article submission.

Development and analysis of two cements for employment in retrograde filling and sealing of perforations

ABSTRACT

The objective of this study was to develop and analyze the physical-chemical properties and dental color changes of two experimental and retro filler cements to be used in perforations, containing different radiopacifiers and compare them to the properties of MTA HP and MTA Angelus. The cements were divided into 6 groups: I - MTA Angelus; II - HP MTA; III - Experimental 1 with powder (60% calcium silicate, 10% calcium phosphate, 30% zirconium oxide) and liquid (80% water and 20% arnica extract); IV - Experimental 2 with powder (60% calcium silicate, 10% calcium phosphate, 30% calcium tungstate) and liquid (80% water and 20% arnica extract); V - Experimental 3: silicone cement (Roeko Seal) + 20% calcium silicate + 10% zirconium oxide and VI - Experimental 4: silicone cement (Roeko Seal) + 20% calcium silicate + 10% calcium tungstate. For the accomplishment of the tests of radiopacity, flow and time of prey were followed the ISO 6876/2001 specifications and ASTM C266/2008. pH was determined by a pH meter previously calibrated. To determine the release of calcium ions, an atomic absorption spectrophotometer was used. The volumetric change was measured by Micro-CT. For the evaluation of tooth discoloration, 10 bovine teeth were used for each group. The teeth had the pulp chambers cleaned and filled with the cements. After the periods of 7, 30 and 60 days specimens were submitted to spectrophotometry. The data were statistically compared using significant level ($p<0,05$). All experimental materials tested showed radiopacity above the recommended minimum (3 mm / Al). Groups G5 and G6 showed a higher flow rate. All materials provided alkalization of the water in which they were immersed after 3 days, except groups G5 and G6. Regarding prey time, there were no significant differences in the comparisons between G2 and G3 and between G5 and G6. In the 3-day period, G1 stood out as the group that released more calcium ions, obtaining a statistically significant difference when compared to the others, except for G2. When we compare the color variation for the three periods, it was noticed statistically stable values for groups G2, G3 and G4. It is concluded that the experimental cements based on calcium silicate showed more favorable properties, such as lower flow, alkalization of the medium, higher working time and color stability.

Keywords: Retrograde Obturation, Dental Cements, MTA.

INTRODUCTION

Root canals repair cements are frequently used to solve accidents in endodontic treatment such as perforations during the pulp chamber access or instrumentation (1, 2, 3).

An ideal repair material should contain favorable physical-chemical and biological properties which can lead to a successful procedure. These properties include biocompatibility, neutral or alkaline pH, lower solubility, calcium ion release, sealing, radiopacity and setting time, as long as do not interfere or corroborate with the tissue repair. Several materials were tested and used in the repair function such as zinc oxide and eugenol cements (ZOE), resin, calcium silicate, calcium hydroxide and silicone based. (4, 5)

The ZOE and amalgam have not shown adequate biological properties and resin cements containing calcium hydroxide presents long setting time (6, 7, 8) although they presents biocompatibility when used as retrograde filling material. (9)

Calcium silicate cements such as Mineral Trioxide Aggregate (MTA) and Portland Cement have similar characteristics to their composition (tricalcium silicate, dicalcium silicate, bismuth oxide, tricalcium aluminate, tretacalcium aluminoferrite and dihydrated calcium sulfate) and presentes favorable physico-chemical and biological properties (10-15), being its pH 10,2 after manipulation, and 12,5 after setting time (16). They are widely used in endodontics, especially in cases of root perforations and as a retro filler (17- 21). The antimicrobial action is not great (21, 22, 23), presenting ability to stimulate the periapical tissue repair (12, 18). The disadvantage, in the case of MTA, it is the long setting time (2h 45min), the handling of the material and the consistency for condensation in the cavity is not adequate (19).

In the function of the difficulty of handling, inadequate consistency and low viscosity of the MTA and Portland Cements, the use of these does not become practical (22, 25). Thus, the addition of components that provide a higher flow becomes necessary, such as propylene glycol, which have been employed in order to provide a higher viscosity (24). Also, the association of antimicrobial agents have been proposed to increase the antimicrobial action (25). Lychnophora ericoides (Arnica) is a plant whose extract presents analgesic, anti-inflammatory and antimicrobial action (26). Portland cement presents a certain absence of radiopacity, so zirconium oxide and calcium tungstate have been associated and although they are less radiopaque when compared to bismuth oxide, they do not promote the darkening of the dentin (27-31). The MTA-based cements present in their composition bismuth oxide that is a radiopacifying agent (32, 33) and dispenses the addition of radiopacifying components.

Silicone based sealers such as Roeko seal have good sealing ability and are currently on the market, but these cements have not been tested as a repair material, as well as if the addition of calcium silicate to them improves biological behavior and becomes more consistent to be used in retrograde obturation and to seal perforations.

However, the development of new materials that have the major number of requirements for an ideal repair material is opportune.

The objective of this study was to evaluate the physical-chemical properties of two experimental cements containing calcium silicate and radiopacifier agent in the composition, one was associated with a liquid containing Lychnophora ericoides (Arnica) glycolic extract and the other consisted in the association of silicone based cement (Roeko Seal) and calcium silicate with radiopacifier agents.

MATERIAL AND METHODS

Material

The cements evaluated were:

- MTA Angelus White; (Angelus ind e com ltda, Londrina, Paraná, Brazil);
- MTA HP (Angelus Ind e com Ltda, Londrina, Paraná, Brazil);
- Experimental 1 - Powder (60% Calcium Silicate, 10% Calcium Phosphate, 30% Zirconium Oxide) Liquid (80% water and 20% *Lychnophora ericoides* (Arnica) glycolic extract);
- Experimental 2 - Powder (60% Calcium Silicate, 10% Calcium Phosphate, 30% Calcium Tungstate) Liquid (80% water and 20% *Lychnophora ericoides* (Arnica) glycolic extract);
- Experimental 3 - Silicone Cement + 20% (by weight) calcium silicate + 10% (by weight) Zirconium Oxide
- Experimental 4 - Silicone cement + 20% (by weight) of calcium silicate + 10% (by weight) calcium tungstate

For the silicone cements, the powder portion were placed in the base paste. In the handling, the same length of base paste and catalyst were employed.

For the preparation of the experimental cements, an electronic analytical balance (Gehaka AND-GR-202, Tokyo, Japan) were employed. Cement manipulation was performed following the ratio of 1 gram of powder to 0.3 ml of liquid for the MTAs groups and for the experimental cements 1 and 2.

Methods

Radiopacity

For the radiopacity analysis, metal rings of 10mm internal diameter and 1mm thickness were used according to ISO 6876/2001**,** consisting in three specimens filled with cement for each group.

The freshly spatulated cements were inserted into the rings and kept in an oven at 37 °C and 100% humidity until complete setting. Then the thickness of the samples was checked using a digital caliper (Mitutoyo Corp, Tokyo, Japan). The samples with problems were remade. The samples were then radiographed on a D-occlusal film (Kodak Comp, Rochester, New York, USA) with an aluminum scale ranging from 2 to 16mm (2mm increments) by following the specifications recommended by the standard ISO 6876/2001, 60kV, 10mA, 0.3 seconds and focus-film distance of 30cm (ISO, 2001). The radiographs were manually processed, later digitized and analyzed in the Adobe Photoshop CS6 (13.0). Radiopacity in millimeters of aluminum was performed according to Duarte et al. (28):

$$\frac{DRM - DRPAA \times 2}{DRPAS - DRPAA} + \text{mmAl below value} = \text{radiopacity mmAl}$$

DRM - Radiographic density of the material

DRPAA - Radiographic density of the aluminum step below

DRPAS - Radiographic density of the upper aluminum pitch

Setting time

The ISO 6876/2001 normative was used to obtain the samples and the ASTM C266/08 was followed for the determination of the setting time of the cements. The spatulated cements were immediately poured into metal rings of 10mm internal diameter and 2mm thick. Three specimens were used for each cement and were kept in an oven at 37 °C ± 1 °C temperature and 95% ± 5% humidity during the test. After 180 seconds of the start of the spatulation, the specimens were subjected to vertical pressure marks using Gilmore needles. For the determination of the initial prey time a needle of 113.4 g was used, later the needle of 453.6 g were used for the final prey analysis. The times, in minutes, elapsed from the beginning of the spatulation to the moment when it is not possible to visualize the marking of each needle on the surface of the samples represented the initial and final setting time of the cements. Five discs for cement were used.

Flowability

The flow test was performed according to ISO 6876/2001. A total volume of 0.5mL of cement was spatulated and placed in the center of a glass plate. After 3 (three) minutes from the beginning of the mixture, another glass plate of 20 ± 2g mass was fitted on the plate containing the cement and on both a weight corresponding to 100g. After 10 (ten) minutes of spatulation, the weight was removed and the largest and smallest diameter of the cement was measured using a digital caliper (Mitutoyo MTI Corporation, Tokyo, Japan). The average of the two diameters was considered the cement flow. Three measurements were made for each cement variable.

Volumetric change

The volumetric change was evaluated using computerized microtomography (micro-CT). Each sample was scanned in micro-CT (SkyScan 1174v2; SkyScan, Kontich, Belgium) with 50 kV and 800 µA, twice, consisting of sixty (60) acrylic teeth with backs filled with the experimental cements. The capture parameters of the images were 14.1µm of voxel, using scanning with rotation of 360º. Each scan provided 1024 x 1304 pixel images. The data were reconstructed using software (NReconv1.6.4.8, SkyScan) and CTan software (CTan v1.11.10.0, SkyScan) for volumetric analysis. In the CTan software, the images of the samples were analyzed individually, thus demarcating the areas of interest (ROI) for each sample. The values were noted to be used as the standard for the second scan. A quantitative analysis of the volume of the material was carried out by means of three-dimensional analysis and the total volume (mm3) was automatically calculated by means of the three-dimensional image. After the scanning, the samples were immersed in individual flasks containing 15 mL of deionized water and stored in an oven at 37ºC for 28 days. After the period, the samples were removed from the vials, dried on filter paper and re-scanned using the same parameters from the initial scan. The solubility of the cements was determined by calculating the volume lost during immersion and the values converted into percentages.

pH and release of calcium ions

For the determination of the pH and calcium release proportioned by the cements, 60 acrylic teeth (n = 10) were used with retrograde cavities made with diamond spherical drill 1012. The cavities were filled with the cements and the specimens were individually immersed in flaks containing 10mL of ultrapure with values of pH 6.61. These flasks were sealed and taken to the oven at 37 °C, where they remained during the experimental period. The evaluations were performed after periods of 3, 7 and 15 days of immersion. After each period, the specimens were removed from the flasks and immersed in new flasks containing the same volume (10mL) of ultrapure water. The pH was determined by a pHmeter previously calibrated with solutions of pHs (4, 7 and 14). For calibration, the specimen was initially removed from the flask and then the glass was taken to a shaker for 5 seconds. After stirring, the liquid was poured into a Becker and then placed in contact with the pH meter electrode. The calibration was performed in a room with a temperature of 25 ° C.

The ion release was carried out in the same periods used for the pH reading. For the determination of the calcium ion release, an atomic absorption spectrophotometer equipped with a calcium-specific hollow cathode lamp was used. To prevent possible alkali metal interferences, lanthanum solution was used.

A standard solution of calcium was prepared, being: 20mg/L, 10mg/L, 5mg/L, 2,5mg/L, 1,25mg/L. 8mL of the samples standards or water were associated with 2mL of lanthanum chloride solution. For blank sample, 6mL of MiliQ water were associated with the same amount (2mL) of lanthanum chloride solution. With the standards, the blank and the samples prepared, the reading was carried out in the atomic absorption spectrophotometer. The solution of nitric acid was used to bring the apparatus to zero absorbance. Calcium ion release calculations were performed using the standard curve straight equation.

Color change

For the color change analysis, 10 (ten) bovine teeth were used per group. Color evaluation with the Vita Easyshade spectrophotometer (Vita-Zanhnfabrik, Germany) was based on the criteria defined by International Commission on Illumination (1978). The teeth were scanned for initial color determination and then coronary opening, complete removal of pulp tissue present in the pulp chamber and irrigation with 2.5% sodium hypochlorite for 1 minute using passive ultrasonic method. After irrigation, the pulp chamber was dried with a cotton ball and filled with the spatulated cements until complete sealing. Analyzes were performed after 7, 30 and 60 days, and in each period consisted of 3 (three) reading for each tooth.

The difference between the initial and final color, or the distance between the two colors on the axes, known as (ΔE) was calculated using the formula:

$$\Delta E = [(\Delta L^*) 2 + (\Delta a^*) 2 + (\Delta b^*) 2] 1/2 \quad (10)$$

Where L * represents the brightness values of the color, a * corresponds to the measurement along the red-green axis and b * is the measurement along the yellow-blue axis. A positive value of a * indicates that the color is directed to red while a negative value indicates the direction to the green. A Positive value of b * indicates the direction of color to the yellow axis, while its negative value indicates direction to blue.

Statistical analysis

The results were submitted to the Kolmogorov-Smirnov tests for verification of normal distribution. In case of absence of normality, the non-parametric Kruskal-Wallis and Dunn tests were employed. In case of normal distribution, the parametric ANOVA and Tukey tests were employed. For all tests, the significance level of 5% was considered.

RESULTS

Radiopacity

Table 1 presents the means, standard deviations and significant differences of the radiopacity values expressed in millimeter of Aluminum (mm / Al) of the materials. WMTA and MTA HP reported radiopacity below the minimum recommended by ADA standards 57 and ISO 6876/2001, which is 3 mm / Al. All the experimental materials presented higher values when compared to these. The highest value occurred for Experimental 3.

Setting Time

Table 1 presents the mean and standard deviation of the initial and final setting times (in minutes) of the materials evaluated in this study.

The analysis of the initial setting time of the materials showed that there were statistically significant differences for all groups ($p < 0.05$), except between WMTA and experimental 2, and when comparing experimental 3 and 4.

Analyzing the final setting time, there were no statistically significant differences ($p > 0.05$) in the comparisons between MTA HP and experimental 1, and between experimental 3 and 4. In the other comparisons there were statistically significant differences ($p < 0.05$) among the groups studied.

Flowability

Table 1 shows the means and standard deviations of the flow obtained through the mean of the two diameters.

The silicone experimental groups presented a similar and significantly higher flow rate ($P < 0.05$) when compared to the other cements analyzed. Among the other groups, there were no significant differences in the comparison between them ($P > 0.05$).

Volumetric change

Table 1 contains the median, minimum and maximum value of the volumetric loss of material in percentage, obtained through the difference between the final and initial volume of each specimen in the period of 28 days. The experimental 3 and 4 whose cements are silicone based had a lower volumetric loss rate (9.62% and 5.49%, respectively) when compared to the other groups, but only the group in which the radiopacifier was calcium tungstate obtained a statistically significant difference in relation to the others ($P<0.05$). The experimentals 1 and 2 groups presented higher solubility (15.44% and 16.59%), but did not present a significant difference between the remaining groups.

pH and Calcium ion release

Table 2 presents the averages and standard deviations of the pH of the analyzed materials in the periods of 3, 7 and 15 days. The pH of the water used in this experiment was 6.61 and, with the exception of experimental 3 and 4 groups, all the materials provided alkalinization of the water in which they were immersed for the period of 3 days.

For the 3-day period, WMTA, MTAHP and experimental 1 and 2 groups did not present significant statistical differences among their ($P>0.05$), maintaining their pH around 7. Experimental 3 and 4 presented a slight decrease of pH when compared to the initial value.

In the 7-day period, WMTA and MTAHP differed statistically from all experimental groups 3, 4, 5 and 6, suffering a slight alkalinization. The other groups obtained a small decrease in pH for this period.

In the analysis of the period of 15 days, only MTA HP was statistically different from the other groups, keeping its pH stable around the considered neutral.

When comparing intra-group, the only ones presenting a stable pH were the MTA HP and experimental 1, whose did not obtain statistically significant differences between the periods ($P> 0.05$).

In relation to the calcium ion release, table 2 presents values for the experimental cements at different periods (3, 7 and 15 days). In the 3-day period, WMTA stood out as the group that released more calcium ion, obtaining a statistically significant difference when compared to the others, except for MTA HP. Experimental 4 was the group that presented the least calcium ion release, and also obtained a significant difference of WMTA and MTAHP in the time of 3 days and 7 days. At 15 days, WMTA continued to stand out with greater release when compared to the others, followed by MTAHP without significant differences between them ($P > 0.05$).

For intra-group comparison, only WMTA and Experimental 4 (Roeko Seal cement and calcium silicate) showed a slight decrease in the calcium ion release with statistically significant differences between the times of 3 and 7 days ($P<0.05$).

Spectrophotometric

Table 3 shows values of the color records, obtained in the times of 7, 30 and 60 days, with the help of the VITA Easyshade digital spectrophotometer. The color was evaluated through the CIELAB system, the values of L * a * and b * recorded, and the color change, described as Delta E, calculated. At the 7-day time point, MTAHP group differed from all other groups with statistical significant differences ($P <0.05$), except for Experimental 3 and 4, which are composed of the silicone-based experimental (Table 3), the Experimental 3 and 4 groups remained statistically different from each other ($P<0.05$), behaving in a similar way. Experimental 1 and 2 also did not obtain differences between them, both based on calcium silicate.

When we compare the three periods, we can analyze statistically stable values for groups MTAHP, experimental 1 and 2. Experimental 3 and 4 groups (based silicon) presented a decrease in the values in time of 30 days, being statistically different from the times of 7 and 60 days ($P<0.05$). WMTA obtained similar statistical values for the times of 7 and 30 days ($P>0.05$), which differed only from those of 60 days ($P<0.05$).

DISCUSSION

Due to the necessity of an ideal material that favors good handling, antimicrobial action, good biological behavior and radiopacity, researches have been constant (34).

For the radiopacity test, the analysis of radiographic images scanned in software developed for this purpose allowed an advance in the studies of this property, being a methodology reproducible and with reliable results, without destroying the sample. In this study, the software used was Adobe Photoshop CS6 (13.0) in which it was possible to obtain a mean in numbers of each step of the gray scale and of the specimens with the cements, then compare the results through a formula developed by Duarte et. al (28).

The results showed that all groups of experimental cements were more radiopaque when compared to white MTA and HP MTA that had radiopacity below the ISO standard (35). The differences could be related to the percentage of radiopacifiers, the proportion of the radiopacifiers being 30% by weight for the groups whose cements were calcium silicate based, and 10% for silicon cements (Roeko Seal) that already presented a radiopacifier**.** Possibly the WMTA and MTAHP present lower percentage of radiopacifier. This factor may interfere the radiographic visualization of the material and if they filled well the retrograde or e perforation cavity. Roeko Seal is a polydimethylsiloxane-based material, which contains zirconium dioxide, which already has good radiopacity (36), and with the addition of a further 10% of radiopacifier favor an increase in the radipacity.

Regarding the WMTA, another study demonstrated the values for radiopacity varying between 3 and 3.3 mmAl, however it should be emphasized that the radiopacifier was recently modified to calcium tungstate which is less radiopaque than bismuth oxide that was the WMTA used in the previous study (37). When compared to the Torabinejad study (19), the values differ since this author obtained a value of 7.17 mm Al for the MTA, however in this study the gray ProRoot was used, which presents bismuth oxide in the percentage of 20% radiopacifier. The experimental cements presented radiopacity values between 5.33 mmAl to 6.14, which are values within the standards established by ISO and ADA**.**

In endodontic surgery and in perforations, cements with long setting times may be more susceptible to dissolution. For this reason, laboratory studies that evaluate the setting times and the volumetric change of endodontic cements are of clinical relevance. This study followed ISO standards in the preparation of test specimens and ASSTM 266/08 for needle weights in determining setting time. The results showed that the silicon-based experimental cements (Experimental 3 and 4) demonstrated the shortest setting time (44 and 47 minutes) when compared to the calcium silicate base group (Experimental 1 and 2), but without statistically significant differences between them. This result is in agreement with previous studies (38, 39) in which they obtained values between 45 minutes and 50 minutes, demonstrating that the association of calcium silicate and radiopacifier did not interfere in the setting of the material. The initial settling time of 14 minutes for the MTAHP in this study was very similar to that mentioned by the manufacturer which is 15 minutes. For the final setting time, the value was 25 minutes higher than that observed in another study (40). This difference may be related to the powder / liquid ratio in the manipulation. The WMTA group demonstrated an initial setting time of 41 minutes and final setting time of 129 minutes, these times is in agreement with other studies (41, 42). Concerning the experimental cements that employed the calcium silicate and Arnica's propylene glycol solution, initial setting times were higher than MTAHP and the WMTA, possibly due to the presence of 20% Arnica's propylene glycol extract, corroborating with other studies that employed the association of pure propylene glycol or other extracts (24, 25, 43).

Another property studied was the flowability, which is generally used to analyze root canal filling cements due to the need to fill the entire prepared conduit through the hermetic distribution of the cement. However, in restorative materials it may favor extravasation when inserted into perforations. In relation to the obtained data, the Experimental 3 and 4 groups of silicone-based experimental cements had the higher flow than other groups. These results may be justified by the sandy composition of the silicate-based cements, even those with propylene glycol extracts, and due the plasticity of Roeko Seal cement. However, the calcium silicate cements with the propylene glycol extract had a higher flow rate than the WMTA and the MTA HP. Propoylene glycol favors the manipulation and flow of calcium silicate cements. (24, 25, 43)

The volumetric change is a relevant physical property to be analyzed in cements, considering that in retrograde cavity and in the perforation, the material will remain directly in contact with adjacent tissues for a long period. In addition, materials with high solubility tend to lead to inadequate sealing and presence of voids in the filling, increasing the possibility of bacterial infiltration and consequently lead to treatment failure. (44)

To evaluate the materials, there are different methodologies available. The most commonly used standards are ISO 4049/2009, ISO 6876/2001 and ANSI / ADA specifications no. 57/2000 for the study of the solubility of sealants. ISO 4049 describes procedures for evaluating both water absorption and solubility. This is characterized by the preparation of specimens that are immersed in water, and the initial and final mass difference after immersion is considered the solubility of the cements. They also state that the materials must not have a total weight loss higher than 3% over the initial weight. The methodology of verifying the change in volume using micro-CT has already been proposed in a previous study (44). In the analysis of the obtained results, it was possible to affirm that the cements based on calcium silicate (loss values 15.44% - 16.59%) were more soluble than those based on silicone (9.62% and 5.49 %), demonstrating that silicon favors low volumetric loss, even if calcium silicate powder and radiopacifier are added. The experimental based silicon with calcium tungstate added as a radiopacifier was the only one that showed differences with all other cements studied, obtaining the lowest volumetric loss value (5.49%). The WMTA and MTAHP did not present any differences, being the volumetric loss value for both 14.36% and 15.94%, respectively. Therefore, the group of silicone-based experimental cements was closer to the value established by the ISO standard.

The pH and the calcium ion release from calcium silicate cements have been extensively studied. The MTA cement can present a variation in the pH and the calcium ion release, being reported in the literature pH values between 8-12 (16, 42). In the present study, the WMTA and HP MTA groups (pH 7.4 and 7.7 respectively) along with the calcium silicate-based experimental cements presented slight alkalization in the initial period of 3 days, with differences when compared to the silicone based. The low pH of the silicone cements is related to the lower solubility. At the time of 7 days the white MTA showed an increase in its pH and then showed a decrease in the period of 15 days. On the other hand, the MTAHP presented a constant pH in all the periods.

The experimental cements containing arnica extract had the highest alkalinity only in the period of 3 days and did not present alkalinity in the periods of 7 and 15 days. Possibly because these materials use only calcium silicate and not Portland cement, the hydration reaction and the formation of Portlandite occurs totally in the period of 3 days, with no formations in the later periods, or another hypothesis is that the the arnica extract reduces the capacity of alkalization. Another factor is the lower amount of calcium silicate compared to the WMTA and MTAHP. A Recent study (45) showed an alkaline pH of 8.5 in the 2-day period, followed by 8.1 for 7 days and falling to 7.8 at the end of 28 days for WMTA. The highest values may be related to differences in the methodology or pH of the immersed water. These results are characterized by being more alkaline and stable than in the present study, despite having the same value for period of 7 days. Another study (43) obtained results similar to this, in the period of 3 and 7 days. An overly alkaline pH, above 9, can confer greater causticity to the material and lead to a more extensive area of necrosis, making the tissue organization to the repair.

Regarding the calcium ion release, WMTA was the only one that stood out for being the most statistically different from all groups, except for MTAHP group. The high value of MTA may be related to a higher formation of Portlandite and also to calcium tungstate which may also have been released. Only white MTA and Roeko Seal cement with calcium silicate and zirconium oxide showed a slight decrease in the release of calcium ions times of 3 and 7 days. At 7 and 15 days, the materials presented similar calcium release. The silicate cements presented the highest values of release in the period of 3 days, demonstrating that during the setting reaction they allow the release and it becomes lower over time. The cements containing arnica extract had the lowest calcium ion release value in all periods. This fact can be attributed to the smaller amount of calcium silicate that the experimental ones presented. While the WMTA and MTAHP presents around 85% of Portland cement in the composition, the experimental ones present 60% of calcium silicate.

In treatments involving areas with aesthetic compromise, color stability of the cements is critical. Dental discoloration is an undesirable consequence of some chemical reactions observed for certain materials when in contact with dental structures. The discoloration after application of the MTA in contact with the dentin surface has also been reported (46, 47). Factors associated with its darkening have been evaluated and some hypotheses suggested. Most of the theories suggest that the presence of bismuth oxide would be associated with the color change of the MTA (48). In the study, bovine teeth were used to evaluate the tooth color change after contact with the cements. Analyzing the results, it was possible to observe that the calcium silicate cements group and the MTAHP were the most stable in relation to the color change in the three analysis times. On the other hand, the group of silicon-based cements presented a decrease in the values of delta E in the time of 30 days, being statistically different from the times of 7 and 60 days. No changes in color were observed in all tested materials. The WMTA in previous studies showed darkening (48, 49), however the radiopacifier was bismuth oxide. In this study the WMTA radiopacifier was modified for calcium tungstate, which did not cause color change (49).

It is concluded that the experimental cements based on calcium silicate showed more favorable properties, such as lower flow, alkalization of the medium, higher working time and color stability.

Acknowledgments

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Table 1. Radiopacity, setting time, flowability and solubility of the tested materials. Different capital letters represent statistically significant differences ($p<0.05$) in the same column.

	Radiopacity (mm Al)	Setting Time (min)		Flowability (mm)	Volumetric change (%)
		Inicial	Final		
MTA white Angelus	2,39 ± 0,23 A,C	41,00 ± 5,5 A	129,7 ± 4,5 A	7,39 ± 0,48 A	14,36 (7,6 - 17,62) A,C
MTA HP Angelus	2,12 ± 0,44 A	14,67 ± 2,5 B	110,0 ± 8,0 B	7,92 ± 0,15 A	15,94 (7,99 - 21,84) A,C
Experimental cement 1	5,98 ± 0,41 B,C	59,33 ± 3,0 C	99,33 ± 3,05 B	8,66 ± 0,33 A	15,44 (6,2 - 30,44) A,C
Experimental cement 2	5,81 ± 0,54 B,C	45,00 ± 2,0 D,A	84,33 ± 6,02 C	8,51 ± 0,41 A	16,59 (10,19 - 37,49) A
Experimental cement 3	6,14 ± 2,12 B,C	25,00 ± 3,6 E	44,33 ± 3,51 D	14,27 ± 1,52 B	9,62 (4,81 - 13,61) B,C
Experimental cement 4	5,33 ± 2,53 C	29,00 ± 3,6 E	47,67 ± 2,51 D	14,84 ± 0,12 B	5,49 (3,0 - 10,75) B

Table 2. (A) pH of soaking water; (B) Calcium ions released in soaking water. Different capital letters represent statistically significance differences ($p<0.05$) between materials in the same time, whilst different small letters represent differences between the different period in the same material.

		3 days	7 days	15 days
(A) pH of soaking water	MTA white Angelus	7,45 ± 0,93 ^{A,a}	8,11 ± 1,03 ^{A,a,b}	6,70 ± 0,22 ^{A,a,c}
	MTA HP Angelus	7,73 ± 0,86 ^{A,a}	7,66 ± 0,84 ^{A,a}	7,64 ± 1,0 ^{B,a}
	Experimental cement 1	7,01 ± 0,70 ^{A,C,a}	6,53 ± 0,80 ^{B,a}	6,42 ± 0,48 ^{A,a}
	Experimental cement 2	7,03 ± 0,61 ^{A,a}	6,49 ± 1,20 ^{B,a,c}	5,93 ± 0,78 ^{A,b,c}
	Experimental cement 3	6,20 ± 0,36 ^{B,C,a,c}	5,47 ± 0,29 ^{B,b}	5,84 ± 0,68 ^{A,b,c}
	Experimental cement 4	6,20 ± 0,45 ^{B,C,a}	5,88 ± 0,61^{B,a}	6,28 ± 0,69 ^{A,a}
(B) Calcium release	MTA white Angelus	238,6 (215,8 - 270,6) ^{A,a}	213,8 (204,7 - 224,2) ^{A,b}	206,5 (199,1 - 215,4) ^{A,b}
	MTA HP Angelus	5,4 (1,54 - 14,18) $^{C.B,a}$	7,01 (3,16 - 14,7) ^{B,C,a}	6,82 (0,60 - 9,37) ^{B,C,a}
	Experimental cement 1	3,37 (0,28 - 37,82) ^{B,C,a}	1,43 (0,10 - 12,14) ^{B,C,a}	2,22 (0,67 - 30,2) ^{B,C,a}
	Experimental cement 2	2,9 (0,14 - 6,61) ^{B,C,a}	2,48 (1,49 - 6,4) ^{B,C,a}	4,28 (0,12 - 10,69) ^{B,C,a}
	Experimental cement 3	2,8 (2,13 - 4,76) ^{B,C,a}	1,97 (1,32 - 3,02) ^{B,C,a,b}	0,63 (0,06 - 5,24) ^{B,C,b}
	Experimental cement 4	0,35 (0,02 - 7,91) ^{B,a}	0,7 (0,22 - 5,71) ^{B,a}	0,7 (0,11 - 8,78) ^{B,C,a}

Table 3**.** Median, minimum and maximum of ΔE referring to color change in the times of 7, 30 and 60 days. Different capital letters represent statistically significance differences ($p<0.05$) between materials in the same time, whilst different small letters represent differences between the different period in the same material.

	7 days	30 days	60 days
		Median (min-máx)	
MTA White	6,268 (2,04 - 22,62) [A,a]	12,08 (0,87 - 37,75) [A,a]	26,32 (2,77 - 78,77) [A,C,b]
MTA HP	69,45 (27,64 - 113,5) [B,C,a]	83,35 (20,58 - 258,3) [B,a]	46,86 (21,01 - 132,2) [A,a]
Experimental Cement 1	11,37 (1,03 - 67,74) [A,a]	9,978 (1,79 - 205,7) [A,a]	10,42 (2,96 - 119,9) [B,a]
Experimental Cement 2	7,548 (0,37 - 13,03) [A,a]	5,988 (0,82 - 21,03) [A,a]	5,860 (3,08 - 18,90) [B,C,a]
Experimental Cement 3	26,01 (5,59 - 117,2) [A,C,a]	9,558 (0,75 - 25,03) [A,b,c]	18,96 (4,57 - 55,72) [A,C,a,c]
Experimental Cement 4	26,42 (9,96 - 160,3) [A,C,a]	5,220 (0,43 - 51,27) [A,b]	19,06 (1,03 - 77,94) [A,C,a]

3 DISCUSSION

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Due to the necessity of an ideal material that favors good handling, antimicrobial action, good biological behavior and radiopacity, researches have been constant (Orosco *et.* al 2010).

For the radiopacity test, the analysis of radiographic images scanned in software developed for this purpose allowed an advance in the studies of this property, being a methodology reproducible and with reliable results, without destroying the sample. In this study, the software used was Adobe Photoshop CS6 (13.0) in which it was possible to obtain a mean in numbers of each step of the gray scale and of the specimens with the cements, then compare the results through a formula developed by Duarte *et.* al 2009.

The results showed that all groups of experimental cements were more radiopaque when compared to white MTA and HP MTA that had radiopacity below the ISO standard. The differences could be related to the percentage of radiopacifiers, the proportion of the radiopacifiers being 30% by weight for the groups whose cements were calcium silicate based, and 10% for silicon cements (Roeko Seal) that already presented a radiopacifier. Possibly the White Angelus MTA and HP MTA have lower proportions. This factor may interfere the radiographic visualization of the material and if they filled well the retrograde cavity or the perforation cavity. Roeko Seal is a polydimethylsiloxane-based material, which contains zirconium dioxide, which already has good radiopacity (Tanomaru *et.* al 2004), and with the addition of a further 10% radiopacifier this value increased. Regarding the MTA White, another study demonstrated the values for radiopacity varying between 3 and 3.3 mmAl, however it should be emphasized that the radiopacifier was recently modified to calcium tungstate which is less radiopaque than bismuth oxide that was the MTA White radiopacifier used in the previous study (Tanomaru et. al 2008). When compared to other study (Torabinejad et. al 1995), the values differ, since this author obtained a value of 7.17 mm Al for the MTA, however in his study the gray ProRoot was used, which presents bismuth oxide in the percentage of 20% radiopacifier. The experimental cements obtained between 5,339 mmAl to 6,148, without statistical differences between them.

Therefore, the experimental cements tested would be within the standards established by ISO and ADA.

In endodontic surgery and in perforations, cements with long setting time may be more susceptible to dissolution. For this reason, laboratory studies that evaluate the setting time and the solubility of endodontic cements are of clinical relevance. This study followed ISO standards in the preparation of test specimens and ASSTM 266/08 for needle weights in determining setting time. The results showed that the silicon-based experimental 3 and 4 cements) obtained the shortest setting time (44 and 47 minutes) when compared to the calcium silicate base group (Experimental 1 and 2), but without statistically significant differences between them. This result is in agreement with previously developed studies (Camargo *et.* al 2014; Flores *et*. al 2011) in which they obtained values between 45 minutes and 50 minutes, demonstrating that the increase of calcium silicate and radiopacifier did not interfere in the prey of the material. The approximate initial settling time of 14 minutes for the G2 (MTA HP repair) in this study was very similar to that mentioned by the manufacturer which is 15 minutes. For the final setting time, the value was 25 minutes higher than that observed in another study (Guimarães et. al 2018). This difference may be related to the powder / liquid ratio in the manipulation. The MTA group (G1) obtained an initial prey time of 41 minutes and final prey time of 129 minutes, these times is in agreement with other studies (Bortoluzzi et. al 2009; Vivan et. al 2010). Concerning the experimental cements that employed the calcium silicate and Arnica's propylene glycol solution, initial setting time were higher than MTA HP and the white MTA, possibly due to the presence of 20% Arnica's propylene glycol extract, corroborating with other studies that employed the association of pure propylene glycol or other extracts (Holland et. al 2007; Cavenago et. al 2017; Duarte et. al 2012a).

Another property studied was the flowability, which is generally used to analyze root canal filling cements due to the need to fill the entire prepared conduit through the hermetic distribution of the cement. However, in restorative materials it may favor extravasation when inserted into perforations. In relation to the obtained data, the Experimental 3 and 4 groups of silicone-based experimental cements had the higher flow than other groups. These results may be justified by the sandy composition of the silicate-based cements, even those with propylene glycol extracts, and due the plasticity of Roeko Seal cement. However, the calcium silicate cements with the propylene glycol extract had a higher flow rate than the WMTA and the MTAHP. Propoylene glycol favors the manipulation and flow of calcium silicate cements (Holland *et.* al 2007; Cavenago *et.* al 2017; Duarte *et.* al 2012a).

The volumetric change is a relevant physical property to be analyzed in cements, considering that these retro cavity fillers will remain directly in contact with adjacent tissues for a long period. In addition, materials with high solubility tend to lead to inadequate sealing and presence of voids in the filling, increasing the possibility of bacterial infiltration and consequently lead to treatment failure (Cavenago et. al 2013).

To evaluate the materials, there are different methodologies available. The most commonly used standards are ISO 4049/2009, ISO 6876/2001 and ANSI / ADA specifications no. 57/2000 for the study of the solubility of sealants. ISO 4049 describes procedures for evaluating both water absorption and solubility. This is characterized by the preparation of specimens that are immersed in water, and the initial and final mass difference after immersion is considered the solubility of the cements. They also state that the materials must not have a total weight loss higher than 3% over the initial weight. The methodology of verifying the change in volume using micro-CT has already been proposed in a previous study (Cavenago et. al 2013). In the analysis of the obtained results, it was possible to affirm that the cements based on calcium silicate (loss values 15.44% - 16.59%) were more soluble than those based on silicone (9.62% and 5.49 %), demonstrating that silicon favors low solubility, even if calcium silicate powder and radiopacifier are added. The experimental based silicon calcium tungstate added as a radiopacifier was the only one that showed differences with all other cements studied, obtaining the lowest solubility value of 5.49% loss of material. The white MTA and HP did not present any differences, being the loss value for both 14.36% and 15.94%, respectively. Therefore, the group of silicone-based experimental cements was closer to the value established by the ISO standard.

The pH and the release of calcium ions from calcium silicate cements have been extensively studied. The MTA cement can present a variation in the pH and the release of calcium ions, being reported in the literature pH values between 8-12 (Vasconcelos et. al 2009; Vivan et. al 2010). In the present study, the groups MTA Angelus white and HP MTA (pH 7.4 and 7.7 respectively) along with the calcium silicate-based experimental cements presented slight alkalization in the initial period of 3 days, with differences when compared to the silicone based. The low pH of the silicone cements is related to the low solubility. At the time of 7 days the white MTA showed an increase in its pH and then showed a decrease in the period of 15 days. On the other hand, the MTA HP presented a constant pH in all the periods. The experimental cements containing arnica extract had the highest alkalinity only in the period of 3 days and did not present alkalinity in the periods of 7 and 15 days. Possibly because these materials use only calcium silicate and not Portland cement, the hydration reaction and the formation of Portlandite occurs totally in the period of 3 days, with no formations in the later periods, or the arnica extract reduces the capacity of alkalization; another factor is the lower amount of calcium silicate compared to the Angelus MTA and MTA HP. Recent studies by Tanomaru-Filho et. al 2009, showed an alkaline pH of 8.5 in the 2-day period, followed by 8.1 for 7 days and falling to 7.8 at the end of 28 days for MTA white. The highest values may be related to differences in the methodology or pH of the immersed water. These results are characterized by being more alkaline and stable than in the present study, despite having the same value for period of 7 days. Another study by Duarte et. al 2012 obtained results similar to this, in the period of 3 and 7 days.

Regarding the ions calcium release, WMTA was the only one that stood out for being the most statistically different from all groups, except for MTA HP group. The high value of MTA may be related to a higher formation of Portlandite and also to calcium tungstate which may also have been released. Only WMTA and Roeko Seal cement with calcium silicate and zirconium oxide showed a slight decrease in the release of calcium ions times of 3 and 7 days. At 7 and 15 days, the materials presented similar calcium release. The silicate cements presented the highest values of release in the period of 3 days, demonstrating that during the prey reaction they allow the release and it becomes lower over time. The cements containing arnica extract had the lowest release value in all periods. This fact can be attributed to the smaller amount of calcium silicate that the experimental ones presented. While the WMTA and MTAHP presents around 85% of Portland cement in the composition, the experimental ones present 60% of calcium silicate.

In treatments involving areas with aesthetic compromise, color stability of the cements is critical. Dental discoloration is an undesirable consequence of some chemical reactions observed for certain materials when in contact with dental structures. The discoloration after application of the MTA in contact with the dentin surface has also been reported (Belobrov et. al 2011; Jacobovitz et. al 2009). Factors associated with its darkening have been evaluated and some hypotheses suggested. Most of the theories suggest that the presence of bismuth oxide would be associated with the color change of the MTA (Camilleri et. al 2014). In the study, bovine teeth were used to evaluate the tooth color change after contact with the cements. Analyzing the results, it was possible to observe that the calcium silicate cements group and the MTA HP were the most stable in relation to the color change in the three analysis times. On the other hand, the group of silicon-based cements presented a decrease in the values of delta E in the time of 30 days, being statistically different from the times of 7 and 60 days. No changes in color were observed in all tested materials. The white MTA in previous studies showed darkening (Camilleri et. al 2014; Marciano et. al 2014), however the radiopacifier was bismuth oxide. In this study the white MTA radiopacifier was modified for calcium tungstate, which did not cause color change (Marciano et. al 2014).

It is concluded that the experimental cements based on calcium silicate showed more favorable properties, such as lower flow, alkalization of the medium, higher working time and color stability.

4 CONCLUSIONS

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Calcium silicate and silicon-based experimental cements associated with zirconium oxide or calcium tungstate in the handling resulted in the development of materials that exhibited the following characteristics:

a) Both of experimental cements, based on calcium silicate or on silicon, had adequate radiopacity according to ISO 6876: 2001;

b) All materials provided alkalization of the water in which they were immersed after 3 days, except the silicone based experimental cements; The pH of the materials did not present great differences between them;

c) In the analysis of the setting time, the silicon-based experimental cements had a shorter setting time;

d) The MTA proportioned the higher calcium ion release.

e) The color variation values were stable for the experimental silicate-based cements in the three periods analyzed.

f) The volumetric loss was lower by the experimental materials containing silicon.

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