UNIVERSIDADE FEDERAL DO RIO GRANDE DO SUL
FACULDADE DE ODONTOLOGIA

TUANE REGINA GRECHI

PROPRIEDADES FÍSICO-QUÍMICAS DE CIMENTOS DE USO ENDODÔNTICO QUE CONTÊM SILICATO DE CÁLCIO

Porto Alegre
2016

TUANE REGINA GRECHI

PROPRIEDADES FÍSICO-QUÍMICAS DE CIMENTOS DE USO ENDODÔNTICO QUE CONTÊM SILICATO DE CÁLCIO

Trabalho de Conclusão de Curso apresentado ao Curso de Graduação em Odontologia da Faculdade de Odontologia da Universidade Federal do Rio Grande do Sul, como requisito parcial para obtenção do título de Cirurgião-Dentista.

Orientadora: Profª. Drª. Patrícia Maria Poli Kopper Móra

Porto Alegre
2016

CIP - Catalogação na Publicação

Grechi, Tuane Regina
Propriedades físico-químicas de cimentos de uso endodôntico que contêm silicato de cálcio / Tuane Regina Grechi. -- 2016.
34 f.

Orientador: Patrícia Maria Poli Kopper Móra.

Trabalho de conclusão de curso (Graduação) -- Universidade Federal do Rio Grande do Sul, Faculdade de Odontologia, Curso de Odontologia, Porto Alegre, BR-RS, 2016.

1. Endodontia. 2. Propriedades físico-químicas. 3. Cimento de silicato de cálcio. I. Poli Kopper Móra, Patrícia Maria, orient. II. Título.

Elaborada pelo Sistema de Geração Automática de Ficha Catalográfica da UFRGS com os dados fornecidos pelo(a) autor(a).

Para minha mãe, por não medir
esforços para me fazer feliz.
Para meu pai, por me ensinar que o
que a memória ama, fica eterno.

AGRADECIMENTOS

À minha mãe e mestre, Edite Faccio Grechi, por acreditar que a educação é sempre o melhor caminho e por me incentivar a percorrê-lo sempre. Obrigada pelos exemplos de garra, coragem e persistência transmitidos, e por ser a melhor amiga e companheira que eu poderia ter. Você é e sempre será uma das pessoas mais importantes da minha vida.

Ao meu pai, Sérgio Grechi, pelo amor e carinho incondicional durante os dez (insuficientes) anos que convivemos juntos. Obrigada pela proteção que posso sentir diariamente.

Aos meus irmãos, Thiago Grechi e Tobias Grechi, pelo amor e cuidado que têm comigo desde quando souberam que teriam uma irmãzinha. Vocês são meus grandes exemplos de vida, meus maiores referenciais de profissionalismo, responsabilidade e determinação.

Aos meus tios e segundos pais, Gerson Poletto e Odete Faccio Poletto, por estarem presentes em todos os momentos da minha vida, por me acolherem como filha desde sempre, pelas orações intercedidas, pelo amor, proteção e carinho. Obrigada pelos melhores presentes que eu poderia ter: Mateus e Ana Luísa.

Ao meu namorado Tiago Grando, pelo imensurável apoio, incentivo e compreensão durante todos os momentos da faculdade, pelos exemplos de caráter e persistência demonstrados diariamente e por todo carinho que tem comigo. Esse só foi o início de uma longa caminhada de evolução e companheirismo que percorreremos juntos.

A todos os amigos da vida e da faculdade que torcem e torceram por mim, em especial à querida amiga Angélica Vincenzi, por contribuir grandiosamente na construção dos meus valores e por me incentivar a ser sempre uma pessoa e profissional melhor.

Ao grupo de professores de Endodontia da UFRGS, pelo conhecimento, generosidade e amizade compartilhados. Em especial à minha orientadora Patrícia Maria Poli Kopper Móra, pelo acolhimento neste projeto, na iniciação científica e na monitoria. Obrigada por me acompanhar nessa importante etapa da minha vida

acadêmica, pelo convívio, pelo exemplo, pelas oportunidades recebidas e por sempre responder meus questionamentos prontamente.

Ao grupo de professores de Periodontia da UFRGS, pela assistência e ensinamentos recebidos durante três anos e meio de convivência na iniciação científica. Em especial à professora Patrícia Weidlich, pelos ensinamentos e conselhos, e por despertar em mim o amor pela pesquisa científica.

Aos alunos de Pós-Graduação em Endodontia e Periodontia, em especial ao Alexander Pompermayer Jardine, Bruna Greggianin, Fernanda Milanesi, Ivana Zaccara, Marta Musskopf e Ramiro Quintana, pelos conhecimentos, auxílio, paciência, confiança, carinho e diversão compartilhados.

Por fim, agradeço por esse PRESENTE precioso que é a VIDA.

“A felicidade não está na chegada, mas sim no percurso”.

Autor desconhecido

RESUMO

GRECHI, Tuane Regina. **Propriedades físico-químicas de cimentos de uso endodôntico que contêm silicato de cálcio.** 2016. 34 f. Trabalho de Conclusão de Curso (Graduação em Odontologia) – Faculdade de Odontologia, Universidade Federal do Rio Grande do Sul, Porto Alegre, 2016.

O objetivo deste estudo foi avaliar o tempo de presa, a solubilidade e o pH de três cimentos a base de silicato de cálcio (MTA Angelus, Neo MTA Plus e Biodentine). O tempo de presa (n=7) foi determinado com o auxílio de agulhas de Gilmore (I: 100g de massa e 2mm de ponta ativa; II: 456g de massa e 1mm de ponta ativa). O intervalo de tempo entre a manipulação do material e o momento em que a primeira e a segunda agulha, respectivamente, não causaram mais endentações na superfície da amostra, foi calculado para determinar o tempo de presa inicial e final. A avaliação da solubilidade iniciou com a definição do peso inicial de cada amostra (n=11). Após, os especimens foram imersos em 7.5mL de água deionizada por 24 horas, secos e o peso foi aferido novamente. Esse procedimento foi repetido até completar-se 7 pesagens. A análise do pH foi realizada utilizando-se dez amostras (n=10), cada uma imersa em 10mL de água deionizada. O pH foi aferido nos períodos experimentais de 1, 3, 12 e 24 horas, e 7, 14, 21 e 28 dias. Os dados obtidos foram analisados estatisticamente no software GraphPad Prism 7.0 e o nível de significância foi estabelecido em 5%. O Biodentine apresentou o menor tempo de presa inicial e final (12 e 31 minutos, respectivamente), seguido do MTA Angelus e Neo MTA Plus. Depois de 24 horas, o MTA Angelus aumentou sua massa enquanto o Biodentine e o Neo MTA Plus reduziram. Em 7 dias todos os materiais perderam mais que 10% de sua massa original. Todos os materiais testados apresentaram pH alcalino. O Biodentine manteve um pH maior que 10 durante todo o período experimental. Conclui-se que o Biodentine se destacou devido ao seu curto tempo de presa e alto pH mantido ao longo do período experimental. Todos os materiais testados mostraram alta solubilidade.

Palavras-chave: Endodontia. Propriedades físico-químicas. Cimento de silicato de cálcio.

ABSTRACT

GRECHI, Tuane Regina. **Chemical and physical properties of calcium silicate-based cements.** 2016. 34 f. Final Paper (Graduation in Dentistry) – Faculdade de Odontologia, Universidade Federal do Rio Grande do Sul, Porto Alegre, 2016.

Aim: To evaluate setting time, solubility and pH of three calcium silicate-based materials (MTA Angelus, Biodentine and Neo MTA Plus). **Methodology:** The setting time (n=7) was measured using Gilmore needles (i: 100g/2mm tip; ii: 456g/1mm tip). The initial and final setting time was considered the interval between the handling of the cement and the moment when first or second needle, respectively, caused no more identations on the sample. Solubility analysis (n=11) started with definition of the initial mass of each sample. Then, the specimens were immersed in 7.5mL of deionized water for 24 hours, dryed and the mass determined again. This procedure was repeted until complete seven weighings. pH analysis was performed using ten samples (n=10) immersed each one in 10mL of deionized water. pH was measured at experimental times of 1, 3, 12 and 24 hours, and 7, 14, 21 and 28 days. Data were statistically analysed using GraphPad Prism 7.0 software and significance was set at 5%. **Results:** Biodentine had the shortest inital and final setting time (12 and 31 minutes, respectively), followed by MTA Angelus and Neo MTA Plus. After 24 hours, MTA Angelus had an increase in its mass, while Biodentine and Neo MTA Plus had reductions. In 7 days, all materials had losses of more than 10% of the original mass. All materials tested produced an alkaline pH. Biodentine remained with a pH greater than 10 during the whole experimental time. **Conclusions:** Biodentine stands out due to the short setting time and the high alkaline pH throughout the experimental period. All materials presented a high solubility.

Keywords: Endodontics. Chemical and physical properties. Calcium silicate cement.

SUMÁRIO

1 INTRODUÇÃO

Os cimentos endodônticos são utilizados nos procedimentos de obturação e retro-obturação do canal radicular, capeamento pulpar direto, pulpotomia, selamento de perfurações radiculares e apicificação radicular (GRECH et al., 2013). Nos últimos anos, vêm sendo lançados no mercado novos cimentos, contendo silicato de cálcio em sua composição, como o Neo MTA Plus e o Biodentine, indicados principalmente para casos de selamento de perfurações, retro-obturação e apicificação. Neste contexto, vêm sendo preconizado que tais materiais apresentem adequadas propriedades físico-químicas para que o sucesso do tratamento seja favorecido (DUARTE et al., 1997; WANG et al., 2013).

O fato de que os cimentos a base de silicato de cálcio se adaptam bem em ambientes úmidos onde há presença de fluidos como água, sangue e saliva faz com que tenham ganho destaque para o emprego em muitas aplicações clínicas (PRATI et al., 2015). Além das diferentes condições de umidade, também pode haver contaminação por microrganismos e inflamação tecidual associado aos procedimentos de rotina. Por esses e outros motivos, um material ideal deve fornecer propriedades físicas, químicas e biológicas adequadas (CAVENAGO et al., 2013). Dentre as propriedades físicas, o tempo de presa não deve ser longo (SHAH et al., 1996; AMERICAN NATIONAL STANDARDS INSTITUTE, 2001) e a solubilidade deve ser inferior a 3% (AMERICAN NATIONAL STANDARDS INSTITUTE, 2000), pois a dissolução do material poderia favorecer a infiltração bacteriana, comprometendo o tratamento (CAVENAGO et al., 2014). O pH alcalino é uma propriedade química importante porque esta condição poderia influenciar na capacidade antimicrobiana do material, podendo também auxiliar no reparo, estimulando o processo de mineralização (HOLLAND et al., 2002).

O MTA (mineral trioxideaggregate) foi inicialmente indicado como material retro-obturador e posteriormente utilizado também em outras situações clínicas. A expansão das indicações clínicas para o emprego do MTA tem como justificativa suas excelentes propriedades físicas e biológicas, dentre as quais vale destacar a sua adequada capacidade de selamento e sua expressiva bioatividade (TORABINEJAD et al., 1995; SCARPARO et al., 2010). Essas características tornaram o MTA o material mais próximo do ideal dentre os cimentos reparadores já desenvolvidos. Formado por um pó branco ou cinza de partículas hidrofílicas finas

que endurecem na presença de umidade, o MTA é composto, principalmente, por silicatos tricálcico ($3CaO.SiO_2$) e dicálcio ($2CaO.SiO_2$).

O MTA Angelus (Angelus, Londrina, PR, Brasil) é um cimento hidrofílico que contém silicato de cálcio, sendo semelhante ao ProRoot MTA. Apresenta características de bioatividade e biocompatibilidade e tem sido usado atualmente com sucesso em capeamento pulpar direto (TORABINEJAD; PARIROKH, 2010). Sua composição é de 80% de cimento Portland e 20% de óxido de bismuto, além de conter maiores quantidades de carbonato de cálcio e silicato de cálcio do que o MTA. De acordo com o fabricante, esse material apresenta tempo de presa inicial após 10 minutos e final após 15 minutos. Após a espatulação com água, o pH do material tem um valor 10 por até 3 horas, quando há a estabilização do pH em um valor 12 (TORABINEJAD et al., 1995). O MTA Angelus, portanto, produz um pH levemente maior e uma maior liberação de cálcio do que o MTA, sendo essa alcalinidade um dos fatores relacionados a sua satisfatória ação antimicrobiana (TORABINEJAD et al., 1995; TORABINEJAD; PARIROKH, 2010). Segundo Ceci et al. (2015), este material apresentou uma solubilidade menor de 3% de sua massa após os períodos de 24 horas e 60 dias.

Recentemente, foi lançado o Neo MTA Plus (Avalon Biomed Inc., Bradenton, Flórida, Estados Unidos), composto por silicato de tricálcio, silicato de dicálcio, sílica e óxido de tântalo. Este material apresenta as mesmas aplicações do MTA e composição semelhante ao MTA Plus (Avalon Biomed Inc., Bradenton, Flórida, Estados Unidos). Contudo, há a substituição do óxido de bismuto do MTA Plus pelo óxido de tântalo, o que evita que ocorra uma alteração de cor no dente após o contato do material com hipoclorito de sódio (CAMILLERI, 2015). Atualmente, poucos estudos envolveram o uso de Neo MTA Plus, porém o MTA Plus demonstrou a capacidade de liberação de íons cálcio, resultando no aumento do pH de maneira significativa após 7 dias, podendo a alteração do pH influenciar na ação antibacteriana do material (FORMOSA et al., 2013). Contudo, a solubilidade do MTA Plus alcançou índices de 18,5% após um período de 24 horas (GANDOLFI et al., 2014).

O Biodentine (SeptodontLtd., Saint MaurdesFausse's, França) é um novo cimento reparador inorgânico que contém silicato tricálcio (Ca_3SiO_5), anunciado como “substituto bioativo da dentina". O material é indicado como alternativa aos outros cimentos que possuem silicato de cálcio, tais como MTA e BioaggregateTM

(Bioagregado), pois o Biodentine apresenta melhores propriedades físicas e biológicas e apresenta as mesmas indicações clínicas do MTA (RAJASEKHARAN et al., 2014). O Biodentine é composto por uma porção sólida, que consiste em um pó contendo silicato tricálcico ($3CaO.SiO_2$), carbonato de cálcio ($CaCO_3$) e óxido de zircônio (ZrO_2) e uma porção líquida contendo cloreto de cálcio ($CaCl_2.2H_2O$) (CAMILLERI et al., 2012). O material possui um tempo de manipulação de 6 minutos, com presa inicial em até 12 minutos e presa final em 45 minutos (MALKONDU et al., 2014). O Biodentine possui um pH alcalino de valor 10 após 3 horas, não havendo diminuição significativa do valor do pH no período de até 24 horas. Além disso, quando avaliada a solubilidade do material, houve uma perda menor de 3% da massa após os períodos de 24 horas e 60 dias, sendo semelhante ao MTA Angelus (CECI et al., 2015).

Embora sejam encontrados na literatura alguns estudos sobre as propriedades físico-químicas de alguns cimentos de uso endodôntico, incluindo os cimentos que contêm silicato de cálcio, a comparação entre o Neo MTA Plus e o Biodentine foi pouco explorada. Sendo assim, o presente estudo tem como objetivo elucidar questões a respeito do tempo de presa, solubilidade e pH deste dois materiais em comparação com o MTA Angelus que já foi mais estudado.

2 ARTIGO CIENTÍFICO

O desenvolvimento do trabalho está apresentado na forma de artigo científico de periódico em inglês, que será enviado para publicação na revista International Endodontic Journal.

Title

Evaluation of setting time, solubility and pH of three calcium silicate-based cements

Abstract

Aim: To evaluate setting time, solubility and pH of three calcium silicate-based materials (MTA Angelus, Biodentine and Neo MTA Plus). **Methodology:** The setting time (n=7) was measured using Gilmore needles (i: 100g/2mm tip; ii: 456g/1mm tip). The initial and final setting time was considered the interval between the handling of the cement and the moment when first or second needle, respectively, caused no more identations on the sample. Solubility analysis (n=11) started with definition of the initial mass of each sample. Then, the specimens were immersed in 7.5mL of deionized water for 24 hours, dryed and the mass determined again. This procedure was repeted until complete seven weighings. pH analysis was performed using ten samples (n=10) immersed each one in 10mL of deionized water. pH was measured at experimental times of 1, 3, 12 and 24 hours, and 7, 14, 21 and 28 days. Data were statistically analysed using GraphPad Prism 7.0 software and significance was set at 5%. **Results:** Biodentine had the shortest inital and final setting time (12 and 31 minutes, respectively), followed by MTA Angelus and Neo MTA Plus. After 24 hours, MTA Angelus had an increase in its mass, while Biodentine and Neo MTA Plus had reductions. In 7 days, all materials had losses of more than 10% of the original mass. All materials tested produced an alkaline pH. Biodentine remained with a pH greater than 10 during the whole experimental time. **Conclusions:** Biodentine stands out due to the short setting time and the high alkaline pH throughout the experimental period. All materials presented a high solubility.

Keywords: Endodontics. Chemical and physical properties. Calcium silicate cement.

Introduction

The endodontic cements have been indicated in cases of pulp capping, sealing root perforations, apexification and retrofilling. The mineral trioxide aggregate (MTA) stands out among the endodontic cements because of the great biological properties (Torabinejad et al. 1995, Scarparo et al. 2010). The MTA is a calcium silicate-based material, so during the seting process occurs the hydration of calcium silicate, resulting in resistance to the material (Camilleri 2008).

The MTA Angelus (Angelus, Londrina, PR, Brazil) is a hydrophilic cement similar to the original MTA. This material exhibits characteristics of bioactivity and biocompatibility and, therefore, has been used with success in cases of pulp capping (Torabinejad & Parirokh 2010). According to the manufacturer, the material has an initial setting time of 10 minutes and final of 15 minutes. In addition, the MTA Angelus produces an alkaline pH and releases calcium ions (Torabinejad et al. 1995, Torabinejad & Parirokh 2010). The material has a solubility of less than 3% of the initial mass after periods of 24 hours and 60 days (Ceci et al. 2015).

Biodentine (Septodont LTD., Saint Maur des Fossés, France) is an inorganic restorative cement containing calcium silicate. This material showed better physical and biological properties than MTA (Rajasekharan et al. 2014). The Biodentine has a handling time of 6 minutes, an initial setting time up to 12 minutes and a final setting time of 45 minutes (Malkondu et al. 2014). This cement was able to reach a pH=10 in 3 hours, maintaning this value up to 24 hours (Ceci et al. 2015). Also, Biodentine has a lower solubility, losing less than 3% of its mass in periods of 24 hours and 60 days (Ceci et al. 2015).

Recently launched, the Neo MTA Plus (Avalon Biomed Inc., Bradenton, FL, USA) is bioceramic and a calcium silicate-based material with similar composition to MTA Plus (Avalon Biomed Inc., Bradenton, FL, USA). However, the bismuth oxide of the MTA Plus was replaced by the tantalun oxide in the Neo MTA Plus. Few studies involved Neo MTA Plus, but the MTA Plus showed ability to release calcium ions and to increase the pH after 7 days (Formosa et al. 2013).

The lack of knowledge on the physicochemical properties of calcium silicate-based materials – especially Neo MTA Plus – and therefore the inability to connect these properties with biological and antimicrobials factors may be determinant in the clinical choise of the material. Thus, the present study aimed to evaluate the

properties of three different calcium silicate-based materials considering: initial and final setting time, solubility and pH.

Materials and methods

The present in vitro study was approved by the Research Committee of the Faculty of Dentistry of the Federal University of Rio Grande do Sul, Porto Alegre, Brazil (02/2016).

Setting time analysis

The setting time was measured according to the Specification No. 57 of the American Dental Association (1984) and C266-03 of the American Society for Testing and Materials (2000) and ISO 6876 (2001) and Vivan et al. (2010). Seven samples of each tested material (Neo MTA Plus, Biodentine and MTA Angelus), with dimensions of 2mm high and 10mm in diameter were prepared (n=7). The materials were handled in accordance with the standarts of the manufacturers and, after 120 seconds, the samples were placed at an incubator at 37ºC and 95% humidity. One hundred and fifty seconds after the end of sample preparation, a Gilmore needle (100g mass and active tip of 2mm) was positioned vertically on each sample. This procedure was repeated every 60 seconds. The initial setting time was considered the time interval between the manipulation of the material and the moment when the Gilmore needle caused no more indentations on the sample.

After determining the initial setting time, started the evaluation of the final set time. In this test, another Gilmore needle was used (456g mass and active tip 1mm). This needle was also positioned vertically on each sample every 60 seconds interval. The final setting time was considered the time interval between the manipulation of the material and the moment when the heavier Gilmore needle caused no more indentations on the sample.

Solubility analysis

The analysis of the solubility of Neo MTA Plus, Biodentine and MTA Angelus was performed according to the methodology described by Carvalho-Jr. (2007) and

American National Standart Institute/American Dental Association (ANSI/ADA) Specification No. 57 for Endodontic Sealing Materials (2000). Eleven samples were prepared for each material tested using plastic molds with dimensions of 1.5mm height and diameter 7.75mm. Each mold was positioned on a glass plate and filled with one of the tested material (n=11). A second glass plate was placed over the filled molds and the set were placed in an incubator at 37ºC and 95% humidity for a time equal to three times of the initial setting time of MTA Angelus (30min) and Biodentine (36min) and of the washout resistence time of Neo MTA Plus (15min), according to the manufactor. Next, the sample materials were removed from the plastic molds and the initial weight of each one was measured in a precision scale (Sartorius SA, Göttingen, Germany).

Then, the samples were kept in Falcon tubes containing 7.5mL of deionized water in an incubator at 37ºC during the experimental period. After 24 hours the samples were removed from the water and placed on absorbent papers in a drying chamber at 37ºC for a period of 48 hours. Then, a new measurement of the mass was performed and the sample was replaced in the water and the set was kept in the incubator for more 24 hours. This procedure was repeated for 7 times, completing 7 days of imertion of each sample in water. The masses of the samples was expressed in percentage (%) and compared within materials over time and between groups in each time.

pH analysis

The pH analysis of the calcium silicate based materials started with the filling of 10 polyethylene tubes (with dimensions of 10mm lenght and diameter 1.6mm) with each one of the tested materials (n=10). After the initial setting time – indicated by the manufacturer – each sample was placed in Falcon tube containing 10mL of deionized water (pH=7.4). A calibrated pHmeter (Digimed, Digicrom analítica Ltda, Campo Grande, São Paulo, Brazil) messured the pH of deionized water containing the sample after 1, 3, 12 and 24 hours and 7, 14, 21 and 28 days using. Throughout the experimental period, the samples were stored at 37ºC.

Statistical analysis

The setting time, solubility and pH data were subjected to Kolgomorov-Smirnov normality test. Assuming non-normality of the data, the Kruskal-Wallis test, followed by Dunn, compared the initial and final setting time between groups. To compare solubility and pH data intergroups two-way ANOVA, followed by Tukey's test, was performed and for the intragroup analysis Friedman test, followed by Dunn, was employed. All statistical analysis was performed with GraphPad Prism 7.0 (GraphPad Software Inc., La Jolla, CA, USA) and significance was set at 5%.

Results

Setting Time analysis

The analysis of the initial setting time showed significant difference of Biodentine compared to MTA Angelus (P=.0496) and Neo MTA Plus (P=.0002). Biodentine also presented the shorter final setting time, but it was only statistically different compared to Neo MTA Plus (P<.0001) (Table 1).

Solubility analysis

After 24 hours, MTA Angelus had an average increase of 3.5% in its mass, with a significant difference compared to Biodentine (P<.0001) and Neo MTA Plus (P=.0477). In the same period, Biodentine and Neo Plus MTA had a decrease in the mass of 8.46% and 3%, respectively. After 48 hours, MTA Angelus showed a mass 1.23% smaller than the original, being different from Biodentine (P=.0008) that reduced its mass 11.42%. In 7 days, Biodentine and Neo MTA Plus showed loss of 17.42% and 16.8% of the original mass, respectively. However, only the Biodentine presented significant difference compared to the MTA Angelus (P=.0492) (Figure 1).

Table 2 summarizes the ingroup analysis. There is no statistically difference in the loss of mass comparing the initial mass until the 3-days period in MTA Angelus and Biodentine groups, and up to 4 days in Neo MTA Plus group. After these periods, there is a decrease in the materials' solubility.

pH analysis

Table 3 and Figure 2 summarize pH results. All materials tested produced an alkaline pH. Biodentine had the highest pH values, remaining at a pH greater than 10 during the entire experimental period. MTA Angelus and Neo MTA Plus were able to achieve a pH greater than 10 in 12 and 3 hours, respectively, and it was maintained up to 7 days. After this period, the pH decreased.

Discussion

This study aimed to evaluate the physicochemical properties of three calcium silicate-based materials: MTA Angelus, Biodentine and Neo MTA Plus. The results showed that Biodentine stood out presenting the shortest initial and final setting time, and also being able to maintain an alkaline pH with values greater than 10 during the whole experimental period of 28 days. On the other hand, Biodentine had the greatest loss values of the original mass and, thus, may be considered a material more susceptible to solubility than MTA Angelus and Neo MTA Plus.

The assessment of physicochemical properties is a constant subject in the study of dental and endodontic materials. Therefore, it is important that researchers base their methods in previously published articles and in specifications of reference associations focusing in produncing comparable data. This study, therefore, sought to base the methodology on the specifications of the American Dental Association, American National Standard Institute, American Society for Testing and Materials, ISO and published articles evaluating materials based on calcium silicate (Camilleri 2010, Vivan et al. 2010, Formosa et al. 2012, Gandolfi et al. 2014, Guerreiro-Tanomaru et al. 2014, Bosso-Martelo et al. 2015, Ceci et al. 2015). Based on these studies, the experimental periods were set up.

Between the calcium silicate-based materials evaluated in this study, Neo MTA Plus is the newest cement launched in the market and thus few studies have been conducted about this material. Neo MTA Plus is similar to MTA Plus due to their compositions by calcium, silicon, and oxygen with sodium, magnesium and sulfur traces and both materials are based on Portland cement (Camilleri 2015). The difference between them is the radiopacifier: in the Neo MTA Plus, the bismuth oxide was replaced by the tantalun oxide. Such substitution aimed to inhibit the discoloration of the material in contact with sodium hypochlorite (Camilleri 2015). Moreover, Neo MTA Plus and the newest version of the MTA Plus have as a

substitute for the mixing liquid: a anti-washout gel. It increases the viscosity of the material and therefore increases the cement washing resistance by external fluids, and reduces the tendency of the material dilution (Formosa et al. 2013).

The composition of the Neo MTA Plus can also be crucial to the lowest setting time compared to previous studies with MTA Plus. While in the present study Neo MTA Plus presented a final setting time of 68 minutes, MTA Plus had setting times of 115 and 255 minutes in a dry environment and in Hank's balanced salt solution, respectively (Formosa et al. 2013). This result can be explained due to MTA Plus presents in its composition bismuth oxide that may be responsible for a higher setting time of the material (Grazziotin-Soares et al. 2014).

Biodentine had the shortest setting time between the tested materials: 12 minutes (initial) and 31 minutes (final). The initial setting time achived in this study was equal to the informed by the manufacturer, while the final setting time was shorter. These results may be associated with the addition of calcium chloride to the mixing liquid of the Biodentine, since this substance is also used in the Portland cement to reduce its setting time (Grech et al. 2013). MTA Angelus is a well known calcium silicate-based material. It is a cement submitted by the manufacturer as an alternative to ProRoot MTA, with a low setting time. The results of this study corroborate this, reaching the final set in 33 minutes. However, the data available in the literature are controversial in regard to this material, with final setting times of 23.33 minutes and 175 minutes (Vivan et al. 2010, Bosso-Martelo et al. 2015). How the manufacturer does not recommend an exact powder/liquid ratio, differing amounts of these compounds may affect the results, explaining the controversial findings.

The solubility of a solid material is measured by the amount of material that can be dissolved in a certain volume of solvent (Parirokh & Torabinejad 2010). Considering this, the weight loss of the cement to the aqueous environment was measured over the period of 7 days. According to the established by ANSI/ADA and the International Standard 6876, it is appropriate that the materials exhibit a solubility lower than 3%. In this study, MTA Angelus had the lowest solubility values compared to Biodentine and Neo MTAPlus. However, all cements had solubility higher than 3% at the end of the experimental period (MTA Angelus: 10.99%, Biodentine: 17.42% and Neo MTA Plus: 16.8%).

The 1-day period showed an increase of MTA Angelus mass that can be related to the absorption of water by the material, causing an expansion. In turn, this setting expansion can be important for a better marginal adaptation of the material, contributing to the sealing ability (Parirokh & Torabinejad 2010). However, at the end of 7 days this material had a mass loss of 10,99%. This value is greater than the ones reported by the literature (Vivan et al., 2010, Bosso-Martelo et al. 2015).

Neo MTA Plus presented in this study a solubility of 3% at 24 hours. In the same period, MTA Plus had a loss of 14.62% in the original mass (Gandolfi et al. 2014), so may be assumed that Neo MTA Plus has a lower solubility than its predecessor. This difference may be explained due to the lowest setting time presented by Neo MTA Plus.

Biodentine had a weight lost of almost 10% in only 24 hours. This result is contradictory with the ones observed by Ceci et al. (2015) that found a solubility of 0.68% in 24 hours. The different findings may be explained by variation in methodological design. Herein the samples were in contact with the water 36 minutes after the material setting time, as reccomended by ANSI/ADA, and in the other investigation the material set for 24 hours before being immersed in the solution.

The solubility of the material may be related to pH changes in the environment in which it is located (Gandolfi et al. 2014). Thus, the high solubility of the cements tested can be associated with alkaline pH reached by all materials. In this context, stands out the Biodentine due to reached the highest pH compared to MTA Angelus and Neo MTA Plus, keeping it above 10 during the whole period. A similar result was found previously, in which the Biodentine presented a pH of 9.83 and 10.66 at 3 and 24 hours, respectively (Ceci et al. 2015).

Another important factor observed during the pH analysis was the behavior of Neo MTA Plus and MTA Angelus. Both materials were able to increase the pH until the periods of 24 hours and 7 days, respectively. In the 7-days period there were no significant differences between the pH of the three evaluated materials but Neo MTA Plus and MTA Angelus decreased the pH values, and Neo MTA Plus also presented a stability trend. In acordance with these findings, previous studies also observed pH values greater than 10 at 24 hours and a decreasing in 28 days to MTA Angelus (Vivan et al. 2010, Guerreiro-Tanomaru et al. 2014, Ceci et al. 2015). The literature up today has no data about Neo MTA Plus pH. It can be compared to data obteined

for MTA Plus in previous investigations that also showed a stability trend for pH values as observed here for Neo MTA Plus (Gandolfi et al. 2014).

The knowledge of the behavior and properties of endodontic materials is important to guide the selection of the most appropriate one, especially in critical situations of clinical practice, when the calcium silicate-based cements are used. Thus, the setting time must allow an adequade handling and inlfuence the solubility and washout resistence of the material (Qi et al. 2012). Furthermore, the high solubility might result in estructural and dimensional changes of the material, impairing its integrity in contact with fluids or blood over time (Carvalho-Junior et al. 2003, Williamson et al. 2005). The pH, in turn, is directly related to the biological activity of the material, influencing the inflammatory reaction caused by the contact of the tissue with the cement and consequently the repair and resolution of bone defects (Torabinejad et al. 1995, Parirokh & Torabinejad 2010, Scarparo et al. 2010, Oliveira et al. 2013, Poggio et al. 2014). Moreover, an alkaline pH may be associated with an unfavorable environment for the viability of microorganisms (Guerreiro-Tanomaru et al. 2014, Poggio et al. 2014, Hiremath et al. 2015, Ceci et al. 2015). Considering the findings of the present study and that the materials' physicochemical properties may influence the outcome of the endodontic treatment, all tested cements demostrated to be apropriated for clinical aplication. It is importante to emphasize that Biodentine stands out due to the short setting time and the high alkaline pH, that presented a stability trend over time.

Conclusions

Biodentine had the shortest inital and final setting time compared to Neo MTA Plus and MTA Angelus. All materials presented a high solubility, although MTA Angelus had a smaller solubility than Biodentine and Neo MTA Plus and was the only one that presented an increase in its mass at 24 hours. All materials tested were capable of producing an alkaline pH. Biodentine maintained the highest pH values throughout the whole experimental period and Neo MTA Plus and MTA Angelus reached their highest pH values at 12 hours and 7 days, respectively, and then the pH values decreased.

Acknolegements

The authors deny any conflict of interest. The authors would like to thank the Laboratory of Oral Biochemistry and Microbiology (LABIM) of the Faculty of Dentistry-UFRGS, for allowing the realization of solubility and pH tests.

References

American National Standard (1984) *American Dental Association Specification #57 for Endodontic Filling Materials*. Chicago: American Dental Association (ADA).

American Society for Testing and Materials (2008). *Standard Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles*, ASTM C266–08.

ANSI/ADA Specification #57, section 5.8. American Dental Association (2000) *Laboratory Testing Methods: Endodontic Filling and Sealing Materials*.

Bosso-Martelo R, Guerreiro-Tanomatu JM, Viapiana R, Berbert FL, Duarte MA, Tanomaru-Filho M (2016) Physicochemical properties of calcium silicate cements associated with microparticulate and nanoparticulate radiopacifiers. *Clinical Oral Investigation* **20**, 83-90.

Camilleri J (2008) Modification of mineral trioxide aggregate. Physical and mechanical properties. *International Endodontic Journal* **41**, 843–9.

Camilleri J (2010) Evaluation of the physical properties of an endodontic Portland cement incorporating alternative radiopacifiers used as root-end filling material. *International Endodontic Journal* **43**, 231–240.

Camilleri J (2015) Staning potencial of Neo MTA Plus, MTA Plus and Biodentine used for pulpotomy procedures. *JOE* **41**, 1139–45.

Carvalho-Junior JR, Guimarães LF, Correr-Sobrinho L, Pécora JD, Sousa-Neto MD (2003) Evaluation of solubility, disintegration, and dimensional alterations of a glass ionomer root canal sealer. *Brazilian Dental Journal* **14**, 114–8.

Carvalho-Junior JR, Correr-Sobrinho L, Correr AB, Sinhoreti MAC, Consani S, Sousa-Neto MD (2007) Solubility and Dimensional Change after Setting of Root Canal Sealers: A Proposal for Smaller Dimensions of Test Samples. *JOE* **33**, 1110–6.

Ceci M, Beltrami R, Chiesa M, Colombo M, Poggio C (2015) Biological and chemical-physical properties of root-end filling materials: A comparative study. *Journal of Conservative Dentistry* **18**, 94–9.

Formosa LM, Mallia B, Bull T, Camilleri J (2012) The microstructure and surface morphology of radiopaque tricalcium silicate cement exposed to different curing conditions. *Dental Materials* **28**, 584–95.

Formosa LM, Mallia B, Camilleri J (2013) Mineral trioxide aggregate with anti-washout gel properties and microstructure. *Dental Materials* **29**, 294–306.

Gandolfi MG, Siboni F, Primus CM, Prati C (2014) Ion release, porosity, solubility, and bioactivity of MTA Plus tricalcium silicate. *Journal of Endodontics* **40**, 1632-7.

Grazziotin-Soares R, Nekoofar MH, Davies TE *et al*. (2014) Effect of bismuth oxide on white mineral trioxide aggregate: chemical characterization and physical properties. *International Endodontic Journal* **7**, 520–33.

Grech L, Mallia B, Camilleri J (2013) Characterization of set Intermediate Restorative Material, Biodentine, Bioaggregate and a prototype calcium silicate cement for use as root-end filling materials. *International Endodontic Journal* **46**, 632–641.

Guerreiro Tanomaru JM, Storto I, Da Silva GM *et al.* (2014) Radiopacity, pH and antimicrobial activity of Portland cement associated with micro- and nanoparticles of zirconium oxide and niobium oxide. *Dental Materials Journal* **33**, 466–470.

Hiremath GS, Kulkarni RD, Naik BD (2015) Evaluation of minimal inhibitory concentration of two new materials using tube dilution method: an *in vitro* study. *Jounal of Conservative Dentistry* **18** 159-162.

International Organization for Standardization (2001) Dental root canal sealing materials ISO 6786.

Malkondu O, Kazandag MK, Kazazoglu E (2014) A review on Biodentine, a contemporany dentine replacement and repair material. *BioMed Research International,* 1–10.

Qi Y, Li N, Niu L *et al*. (2012) Remineralization of artificial dentinal caries lesions by biometrically modified mineral trioxide aggregate. *Acta Biomaterialia* **8**, 836–42.

Poggio C *et al*. (2014) Cytocompatibility and antibacterial properties of capping materials.*The Scientific World Journal,* 1–10.

Rajasekharan S, Martens LC, Cauwels RG, Verbeeck RM (2014) Biodentine material characteristics and clinical applications: a review of the literature. *European Academy of Pediatric Dentistry* **15**, 147–58.

Scarparo RK, Haddad D, Acasigua GA, Fossati AC, Fachin EV, Grecca FS (2010) Mineral trioxide aggregate-based sealer: analysis of tissue reactions to a new endodontic material. *Journal of Endodontics* **36**, 1174-8.

Torabinejad M, Hong CU, Mcdonald F, Pittford, TR (1995) Physical and chemical properties of a new root-end filling material. *Journal of Endodontics* **21**, 349–53.

Torabinejad M, Parirokh M (2010) Mineral Trioxide Aggregate: a comprehensive litertura review – part II: leakage and biocompatibility investigations. *Journal of Endodontics* **36**, 190-202.

Vivan RR, Zapata RO, Zeferino MA *et al*. (2010) Evaluation of the physical and chemical properties of two commercial and three experimental root-end filling materials. *Oral Surgery Oral Medice Oral Pathology Oral Radiology* **110**, 250–6.

Williamson AE, Dawson DV, Drake DR, Walton RE, Rivera EM (2005) Effect of root canal filling/sealer systems on apical endotoxin penetration: a coronal leakage evaluation. *Journal of Endodontics* **31**, 599–604.

Figure legends

Figure 1. Comparsion of mean mass variation values (%) of MTA Angelus, Biodentine and Neo MTA Plus at each experimental period. Different letters indicate signicantly difference between the materials in the same experimental period (P<.05).

Figure 2. Comparsion of mean pH values of MTA Angelus, Biodentine and Neo MTA Plus at each experimental period. Different letters indicate signicantly difference between the materials in the same experimental period (P<.05).

Table 1

Table 1. Median, 75th and 25th percentiles of initial and final setting time (minutes) of MTA Angelus, Biodentine and Neo MTA Plus.

	MTA Angelus	Biodentine	Neo MTA Plus
Initial	28 (40/28)^{a}	12 (15/12)^{b}	47 (47/36)^{a}
Final	33 (48/33)^{ab}	31 (31/23)^{a}	68 (68/68)^{b}

* Different letters in each row indicate significant difference (P<.05).

Table 2

Table 2. Median, 75th and 25th percentiles of masses variation (%) of MTA Angelus, Biodentine and Neo MTA Plus in different experimental periods.

Period	MTA Angelus	Biodentine	Neo MTA Plus
0	100(100/100)^{ab}	100(100/100)^{a}	100(100/100)^{a}
1d	103(104.9/102)^{a}	90.67(94.04/89.24)^{ab}	97.07(98.94/95.47)^{a}
2d	99.47(101.6/97.06)^{ab}	88.84(91.68/85.79)^{abc}	94.57(97.88/91.56)^{ab}
3d	97.87(99.91/94.19)^{ab}	87.6(90.3/83.92)^{abcd}	92.45(96.44/87.92)^{abc}
4d	96.41(97/90.56)^{bc}	86.91(89.25/82.28)^{be}	89.72(93.43/83.19)^{abcd}
5d	94.46(95.32/88.39)^{bc}	86.55(88.4/80.91)^{ce}	88.46(92.11/80.36)^{bcd}
6d	94.04(95.02/87)^{bc}	85.47(87.82/79.42)^{de}	87.21(91.08/78.74)^{cd}
7d	93.28(93.74/83.32)^{c}	84.82(86.6/78.19)^{e}	85.9(90.52/74.96)^{d}

* Different letters in each column indicate significant difference (P<.05).

Table 3

Table 3. Median, 75th and 25th percentiles of pH values of MTA Angelus, Biodentine and Neo MTA Plus in different experimental periods.

	MTA Angelus	Biodentine	Neo MTA Plus
1h	8.76(9.2/8.64)^{acd}	10.72(11.25/10.48)^{a}	8.87(8.97/8.54)^{a}
3h	9.89(10.13/10.06)^{abcd}	11.12(12.02/10.85)^{ab}	10.44(10.54/10.24)^{ab}
12h	10.42(10.63/10.06)^{bc}	11.47(12.63/10.91)^{b}	11.4(11.55/11.01)^{b}
24h	10.54(10.78/9.96)^{bc}	11.36(12.64/10.45)^{ab}	11.2(11.57/11.07)^{b}
7d	10.83(11.09/10.39)^{b}	11.38(11.88/10.98)^{ab}	10.84(11.46/9.14)^{ab}
14d	9.9(10.46/9.09)^{abc}	10.84(11.23/10.22)^{a}	8.81(10.32/8.63)^{a}
21d	8.92(9.93/8.62)^{cd}	11.39(12.07/10.89)^{ab}	9.5(11.12/8.81)^{ab}
28d	8.63(8.76/8.53)^{d}	10.93(11.52/10.02)^{a}	8.6(10.21/8.49)^{a}

* Different letters in each column indicate significant difference (P<.05).

Figure 1

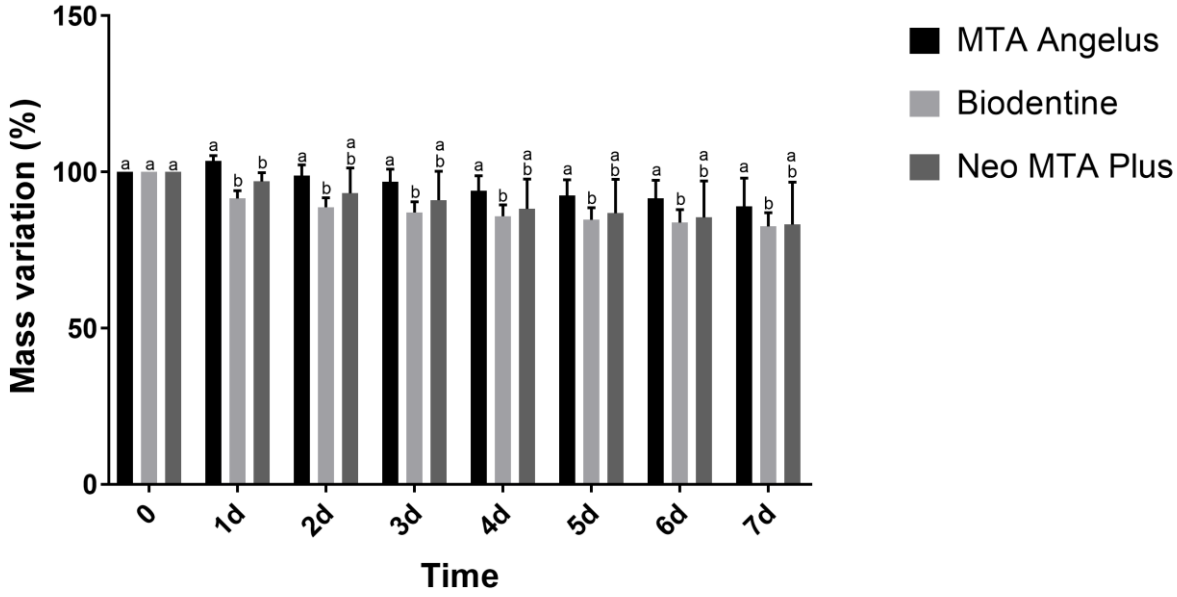
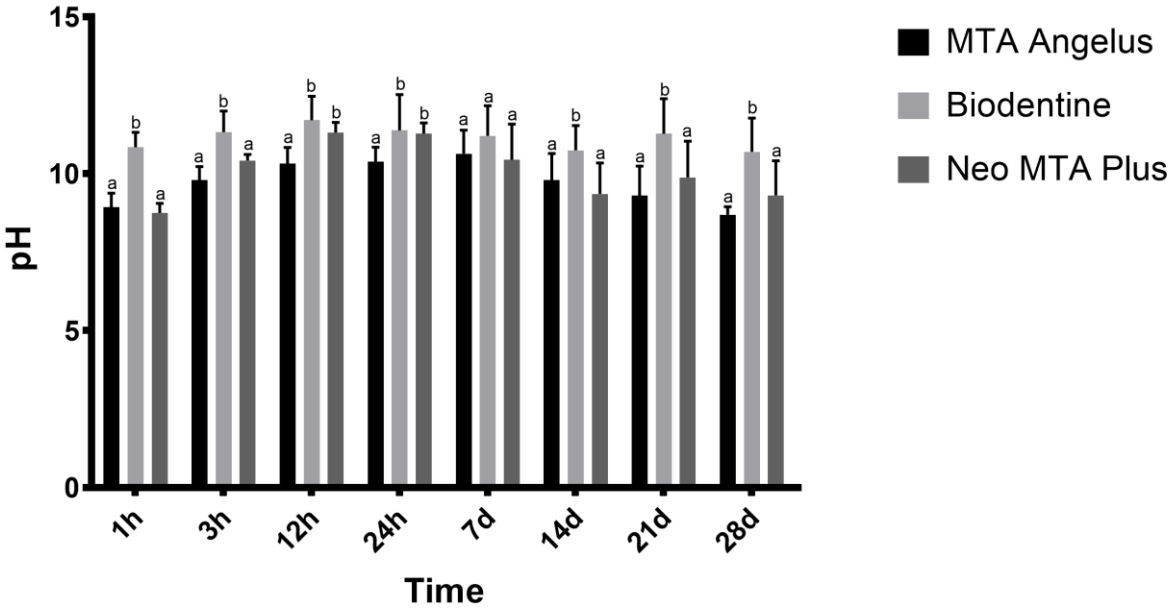

150
100
50
0
Mass variation (%)
a a a
a b a b
a b a b
a b a b
a b a b
a b a b
a b a b
0
1d
2d
3d
4d
5d
6d
7d
Time
MTA Angelus
Biodentine
Neo MTA Plus

Figure 2


15
10
5
0
pH
a
b
a
a
b
a
a
b
b
a
b
b
a
a
a
a
b
a
a
b
a
a
b
a
1h
3h
12h
24h
7d
14d
21d
28d
Time
MTA Angelus
Biodentine
Neo MTA Plus

3 CONSIDERAÇÕES FINAIS

De acordo com os resultados apresentados, observou-se que o Biodentine apresentou o menor tempo de presa inicial e final em comparação com o Neo MTA Plus e o MTA Angelus. Todos os materiais testados apresentaram alta solubilidade, apesar do MTA Angelus ter mostrado a menor solubilidade quando comparado com os outros cimentos e dele ter sido o único que aumentou a massa em 24 horas. Todos os materiais testados foram capazes de produzir um pH alcalino e o Biodentine manteve o elevado pH ao longo de todo o período experimental. O Neo MTA Plus and o MTA Angelus atingiram o maior valor de pH em 12 horas e 7 dias, respectivamente. Após esse período os valores de pH decresceram.

Considerando os resultados do presente estudo e que as propriedades físico-químicas dos materiais podem influenciar no sucesso dos tratamentos endodônticos, todos os materiais mostraram-se apropriados para utilização clínica. É importante ressaltar que o Biodentine se destacou em função de seu pequeno tempo de presa e do seu alto pH alcalino, que apresentou uma tendência de estabilização ao longo do tempo.

REFERÊNCIAS

AMERICAN NATIONAL STANDARDS INSTITUTE. American Dental Association. **Specification no. 57 – endodontic sealing materials**: 2000. Chicago, 2000.

CAMILLERI, J. et al. Characterization and analyses of acid-extractable and leached trace elements in dental cements. **Int. Endod. J.**, Oxford, v. 45, no. 8, p. 737-743, Mar. 2012.

CAMILLERI, J. Staining potencial of Neo MTA Plus, MTA Plus and Biodentine used for pulpotomy procedures. **J. Endod.**, Baltimore, v. 41, no. 7, p. 1139-45, Apr. 2015.

CAVENAGO, B.C. et al. Influence of powder-to-water ratio on radiopacity, setting time, pH, calcium ion release and a micro-CT volumetric solubility of white mineral trioxide aggregate. **Int. Endod. J.**, Oxford, v. 47, no. 2, p. 120-126, May. 2013.

CECI, M. et al. Biological and chemical-physical properties of root-end filling materials: a comparative study. **J. Conserv. Dent.**, Tamilnadu, v. 18, no. 2, p. 94-99, Mar.-Apr. 2015.

DUARTE, M.A.H.; WECKWETH, P.H.; MORAIS, I.G. Análise da ação antimicrobiana de cimentos e pastas empregados na prática endodôntica. **Ver. Odontol. Univ. São Paulo**, São Paulo, v.11, no. 4, p.1-7, Oct.-Dec. 1997.

FORMOSA, L.M.; MALLIA, B.; CAMILLERI, J. Mineral trioxide aggregate with anti-washout gel properties and microstructure. **Dent. Mater.**, Dallas, v. 29, no. 3, p. 294-306, Mar. 2013.

GANDOLFI, M.G. et al. Ion release, porosity, solubility, and bioactivity of MTA Plus tricalcium silicate. **J Endod.**, Baltimore, v. 40, no. 10, p. 1632-37, Oct. 2014.

GRECH L.; MALLIA B.; CAMILLERI J. Characterization of set Intermediate Restorative Material, Biodentine, Bioaggregate and a prototype calcium silicate cement for use as root-end filling materials. **Int. Endod. J.**, Oxford, v. 46, no. 7, p. 632-641, Jan. 2013.

HOLLAND, R.; SOUZA, V.; NERY, M.J. Calcium salts deposition in rat connective tissue after the implantation of calcium hydroxide-containing sealers. **Int. Endod. J.**, Oxford, v. 28, no. 3, p. 173-176, Mar. 2002.

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION DENTISTRY. **ISO 6876**: root canal sealing materials. London: British Standards Institution, 2001.

MALKONDU, O.; KAZANDAG, M.K.; KAZAZOGLU, E. A review on Biodentine, a contemporany dentine replacement and repair material. **BioMed. Res. Int.**, Cairo, p. 1-10, Jun. 2014.

PRATI, C.; GANDOLFI, M.G. Calcium silicate bioactive cements: Biological perspectives and clinical applications. **Dent. Mater.**, Dallas, v. 31, no. 4, p. 351-370, Feb. 2015.

RAJASEKHARAN, S. et al. Biodentine material characteristics and clinical applications: a review of the literature. **Eur. Arch. Paediatr. Dent.**, Athens, v. 15, no. 3, p. 147-158, Mar. 2014.

SCARPARO, R.K. et al. Mineral trioxide aggregate-based sealer: analysis of tissue reactions to a new endodontic material. **J. Endod.**, Baltimore, v.36, no.7, p. 1174-1178, Apr. 2010.

SHAH P.M. et al. Radiopacity of potential root-end filling materials. **Oral. Surg. Oral. Med. Oral. Pathol. Oral. Radiol. Endod.**, St. Louis, v. 81, no. 4, p. 476-479, Apr. 1996.

TORABINEJAD, M. et al. Physical and chemical properties of a new root-end filling material. **J. Endod.**, Baltimore, v.21, no.7, p. 349-353, Jul. 1995.

TORABINEJAD, M.; PARIROKH, M. Mineral Trioxide Aggregate: a comprehensive litertura review – part II: leakage and biocompatibility investigations. **J. Endod.**, Baltimore, v. 36, no. 2, p. 190-202, Feb. 2010.

WANG, Z. et al. Dentin extends the antibacterial effect of endodontic sealers against enterococcus faecalis biofilms. **J. Endod.**, Baltimore, v. 40, no. 4, p. 505-508, Dec. 2013.

ANEXO A – AUTORIZAÇÃO DO USO DE LABORATÓRIO – LABIM

Universidade Federal do Rio Grande do Sul
Faculdade de Odontologia

Porto Alegre, 15 de dezembro de 2015.

Declaro para os devidos fins que o projeto de pesquisa intitulado Propriedades Físico-químicas de Cimentos de Uso Endodôntico que Contêm Silicato de Cálcio será realizado no Laboratório de Bioquímica e Microbiologia Bucal (LABIM).

Atenciosamente,

Clarissa C. Fatturi Parolo

ANEXO B – PARECER DA COMPESQ

UFRGS

Universidade Federal do Rio Grande do Sul — Faculdade de Odontologia

PARECER CONSUBSTÂNCIADO DA COMISSÃO DE PESQUISA

Parecer aprovado em reunião do dia 18 de março de 2016

ATA nº 02/2016.

A Comissão de Pesquisa da Faculdade de Odontologia da Universidade Federal do Rio Grande do Sul após análise aprovou o projeto abaixo citado com o seguinte parecer:

rezado Pesquisador PATRICIA MARIA POLI KOPPER MORA
Informamos que o projeto de pesquisa 30803 - PROPRIEDADES FÍSICO-QUÍMICAS DE CIMENTOS DE USO ENDODÔNTICO QUE CONTÊM SILICATO DE CÁLCIO está aprovado com o seguinte parecer:

Os cimentos de uso endodôntico são utilizados nos procedimentos de obturação e retro-obturação do canal radicular, capeamento pulpar direto, pulpotomia, selamento de perfurações radiculares e apicificação radicular. Nos últimos anos, vêm sendo lançados no mercado novos cimentos contendo silicato de cálcio em sua composição, como o Neo MTA Plus e o Biodentine. Desse modo, embora sejam encontrados na literatura alguns estudos sobre características desses materiais, suas propriedades físico-químicas foram pouco estudadas até o momento e os resultados mostram-se controversos. Por esse motivo, o objetivo deste estudo é avaliar as propriedades físico-químicas de solubilidade, tempo de presa, pH e liberação de íons cálcio dos cimentos MTA Angelus, Neo MTA Plus e Biodentine. O presente estudo será realizado na Faculdade de Odontologia da UFRGS. Para a análise da solubilidade serão avaliadas 11 unidades amostrais de cada um dos cimentos testados, mantidas em estufa a 37ºC durante 7 dias, e terão suas massas medidas a cada 24 horas com o auxílio de uma balança de precisão. Em seguida, o tempo de presa inicial e final dos cimentos serão determinados através de 7 amostras com o auxílio de uma agulha de Gilmore que será verticalmente posicionada na superfície do cimento após 150 segundos da sua espatulação, repetindo o procedimento em intervalos de 60 segundos, até que não cause mais endentações na sua superfície. Para determinar o pH serão utilizadas 10 amostras nos períodos experimentais de 1, 3, 12 e 24 horas e 7, 14, 21 e 28 dias e a medição ocorrerá imediatamente após a remoção das amostras de cimento das soluções ao final de cada período experimental, utilizando um pHmetro. A liberação de íons cálcio será analisada a partir da submersão de 10 amostras em soluções de água destilada e mantidas em estufa a 37ºC durante todo o período experimental e avaliadas nos períodos de 1, 3, 12 e 24 horas e 7, 14, 21 e 28 dias, utilizando um espectrofotômetro de absorção atômica para mensurar a presença de íons cálcio. O projeto encontra-se bem delineado e apresenta mérito científico. Portanto, somos pela aprovação.

Atenciosamente,
Comissão de Pesquisa em OdontologiaAtenciosamente,

Prof. Dr. Fabrício Mezzomo Collares
Coordenador da Comissão de Pesquisa ODONTOLOGIA UFRGS