UNIVERSIDADE FEDERAL DE GOIÁS
PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS DA SAÚDE

OMAR ZINA

Propriedades físico-químicas de cimentos Portland e à base de MTA associados ao *Aloe vera*

Goiânia
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Tese de doutorado apresentada ao Programa de Pós-Graduação em Ciências da Saúde da Universidade Federal de Goiás para a obtenção do título de Doutor em Ciências da Saúde.

Orientador: Prof. Dr. Carlos Estrela

Co-Orientadora: Prof. Drª. Ana Helena Gonçalves Alencar

Goiânia
2013

Dados Internacionais de Catalogação na Publicação (CIP)
GPT/BC/UFG

Zina, Omar.

Propriedades físico-químicas de cimentos Portland e à base de MTA associados ao *Aloe Vera* / Omar Zina.- 2013.

101 f.: 09 figs, 06 tabs, 01 quadro, 01 anexo

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Dedicatória

À Glacy, minha esposa e aos meus filhos Gustavo e Carolina, pela compreensão com tantas ausências e pelo estímulo carinhoso renovado constantemente.

Agradecimentos

Ao Dr. Dráuzio Antonio Medeiros, Reitor do Centro Universitário de Várzea Grande – Univag, pelo apoio institucional;

À Professora Elisabet Aguirre, Pró-Reitora Acadêmica do Centro Universitário de Várzea Grande – Univag, pela oportunidade de trabalhar ao seu lado e amizade sempre renovada;

À Professora Laura Maria Furtado Abreu, companheira de trabalho, amiga e grande incentivadora para que eu enfrentasse mais este desafio;

Ao amigo, companheiro e orientador Professor Carlos Estrela, pela oportunidade de continuar a formação acadêmica, interrompida há quase 30 anos e que, sem o seu envolvimento, não teria acontecido;

À Professora Dra. Ana Helena Gonçalves de Alencar, pela especial atenção, amizade e colaboração.

Ao Professor Dr. Álvaro Henrique Borges, nossos agradecimentos especiais, por sua colaboração espontânea, característica de pessoas especiais e generosas;

Ao ex-aluno e agora mentor, Professor Dr. Orlando Aguirre Guedes, por sua disponibilidade, apoio, orientação e amizade;

Aos amigos Daniel de Almeida Decurcio, Felipe Cavalcanti Sampaio, Marcel Garrot e Iussif Mamede Neto pelo companheirismo, oportunidade de convivência e amizade.

Ao Programa de Pós Graduação em Ciências da Saúde, da Universidade Federal de Goiás, pela multidisciplinaridade oferecida durante a realização do curso, que muito engrandeceu a minha formação acadêmica;

Aos Professores do Curso de Doutorado em Ciências da Saúde da Universidade Federal de Goiás, pela maneira carinhosa que nos receberam e pelos ensinamentos recebidos;

À Sra. Valdecina Quirino, Secretária do Programa de Pós-Graduação em Ciências da Saúde da Universidade Federal de Goiás, por sua atenção educada e gentil, presentes durante todo o transcorrer do curso.

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SÍMBOLOS, SIGLAS E ABREVIATURAS

α	nível de significância
MTA	*Mineral trioxide agregate*
ANSI/ADA	*American National Standards Institute/American Dental Association*
FDA	*Food and Drugs Administration*
ISO	*International Organization for Standardization*
®	marca registrada
pH	potencial hidrogeniônico
°C	graus centígrados
g	grama
n.	número
mm	milímetro
min	minutos
s	segundos
mm^2	milímetro quadrado
mL	mililitro
kVp	kilovolt

mA	miliamper
mmAl	milímetro de alumínio

RESUMO

Esse estudo teve como objetivo determinar propriedades físico-químicas dos cimentos Portland branco estrutural e não estrutural, ProRoot MTA® e MTA BIO® associados à solução glicólica de *Aloe vera* a 2%. Após o estabelecimento da proporção pó/líquido para cada cimento, foram analisadas as propriedaes físico-químicas: tempo de endurecimento, alteração dimensional, pH e radiopacidade. Cinco amostras de cada cimento foram usadas para cada teste, seguindo a especificação n. 57 da *American National Standards Institute/American Dental Association* (ANSI/ADA). A análise estatística foi realizada usando ANOVA e o teste de *Tukey* em nível de significância de 5%. Quando a distribuição das amostras foi não normal, a análise de variância não paramétrica foi realizada com o teste de *Kruskal-Wallis* (α=0,05). Os resultados não mostraram diferenças estatisticamente significantes ($p>0,05$) entre os cimentos em relação à proporção pó/líquido, que variou de 3,14 a 3,52 gramas. O ProRoot MTA® apresentou o tempo de endurecimento mais longo (116,50 min) e o MTA BIO® o menor (66,75 min) ($p<0,05$). Os cimentos Portland brancos estrutural e não estrutural apresentaram valores médios de solubilidade mais elevados (2,30 e 2,81%, respectivamente) ($p<0,05$). Todos os cimentos testados apresentaram valores de alteração dimensional semelhantes entre si (1,00%). O pH dos cimentos manteve-se elevado do início ao final do experimento, não havendo diferenças significantes entre os valores médios obtidos (cimento Portland branco estrutural, 11,96; cimento Portland branco não estrutural, 11,80; MTA BIO® 12,07; ProRoot MTA® 11,77) ($p>0,05$). Os cimentos Portland branco estrutural e não estrutural não apresentaram valores médios em conformidade com ANSI/ADA no teste de radiopacidade (109,76 e 118,75 mmAl, respectivamente), significantemente diferentes dos cimentos MTA BIO® (156,83 mmAl) e ProRoot MTA® (157,95 mmAl) ($p<0,05$), que apresentaram valores de densidade radiográfica acima do degrau 3 do penetrômetro de alumínio. O MTA BIO® evidenciou menor tempo de endurecimento e menor percentual de solubilidade. Nenhum cimento apresentou solubilidade maior que 3%. Todos os cimentos analisados mostraram valores aceitáveis de alteração dimensional e pH elevado durante todo o período de teste. O MTA BIO® e o ProRoot MTA® apresentaram valores de radiopacidade adequados..

Descritores: MTA, cimento Portland, materiais retrobturadores, propriedades físico químicas, *Aloe vera*

ABSTRACT

The aim of this study was to determine the solution-powder ration and to evaluate the setting time, solubility, dimensional change, pH and radiopacity of White structural and non-structural Portland cement, ProRoot MTA® and MTA BIO® associated to 2% glycolic solution of *Aloe vera*. Five samples of each material were used for each test according to American National Standards Institute/American Dental Association specification 57. Statistical analyses were carried out using ANOVA and Tukey's test at 5% level of significance. When sample distribution was non-normal, nonparametric analysis of variance were performed with Kruskal-Wallis test (α=0.05). The results revealed no statistical difference in the water-powder ratio among the tested materials. ProRoot MTA® had the longest setting time (116.50 ± 4.08) ($P<0.05$). White structural and non-structural Portland cements did not have means values on the radiopacity test (109.76 ± 10.40 and 118.75 ± 15.44, respectively) in accordance with ANSI/ADA, being significantly different from the MTA-based cements ($P<0.05$), and demonstrated higher mean solubility values (2.30 ± 0.36 and 2.81 ± 0.40, respectively) ($P<0.05$). The values for dimensional change of all tested materials were considered acceptable by ANSI/ADA. There were no significant differences between the mean pH values recorded for the materials ($p>0.05$). It was alkaline from the beginning to the end of the tests. MTA BIO presented lower setting time and lower solubility. The cements did not showed a higher solubility than 3%. All the assessed cements showed acceptable values of dimensional variation and high pH during the test time. MTA BIO and ProRoot MTA had adequate radiopacity values.

Descriptors: Mineral trioxide aggregate, portland cement, root-end filling materials, physicochemical properties, *Aloe vera.*

1. INTRODUÇÃO

O agregado de trióxido mineral (MTA) foi desenvolvido por Torabinejad nos anos 90, na Universidade de Loma Linda (USA), com indicações para casos de perfurações radiculares (Lee *et al.,* 1993) e como material para retrobturação (Torabinejad *et al.,* 1995a; Torabinejad *et al*, 1995b). Desde a sua introdução e aprovação pela U. S. FDA (*Food and Drugs Administration*), o MTA conquistou aceitação devido a biocompatibilidade e capacidade de selamento (Torabinejad *et al.,* 1995a). Esse material tem sido indicado para capeamento pulpar direto, perfurações em região de furca, pulpotomia, tratamento de dentes com ápices abertos e de reabsorções radiculares (Witherspoon *et al.*, 2008; Martins *et al.*, 2009; Darvell & Wu, 2011).

Os principais componentes do MTA são o silicato de tricálcio, aluminato de tricálcio, óxido de tricálcio e óxido de silicato (Lee *et al.,* 1993; Torabinejad *et al.,* 1995b). Basicamente é composto por cimento Portland convencional tipo I, usado na construção civil e óxido de bismuto, adicionado com a finalidade de melhorar a radiopacidade (Estrela *et al.,* 2000; Funteas *et al.,* 2003; Camilleri *et al.,* 2005; Danesh *et al.,* 2006). Atualmente, duas formas de MTA encontram-se disponíveis comercialmente: ProRoot MTA® (Dentsply Tulsa Dental, Oklahoma, Estados Unidos) e MTA BIO® (Angelus Indústria de Produtos Odontológicos Ltda, Londrina, PR, Brasil). O ProRoot MTA® é composto por 50-65% de óxido de cálcio, 15-25% de dióxido de silício e adição de 20% de óxido de bismuto (Dammaschke *et al.,* 2005; Camilleri *et al.,* 2005), enquanto que o MTA BIO® é composto por 80% de cimento Portland e 20% de óxido de bismuto (Duarte *et al.,* 2003; Song *et al.,* 2006; Oliveira *et al.,* 2007). Tanto os cimentos à base de MTA quanto os cimentos Portland estão disponíveis nas formulações branco e cinza, devido a presença do óxido de ferro (Asgary *et al.,* 2005; Camilleri *et al.,* 2005; Islam *et al.,* 2006a). Em função da presença de material carbonático, substância relacionada à resistência do material, o cimento Portland branco também é classificado como estrutural e não estrutural (ABCP, 2002).

A semelhança química entre o MTA e o cimento Portland tem sido alvo de investigações e, com exceção do óxido de bismuto, apresentam componentes comuns (Camilleri *et al.,* 2005; Dammaschke *et al.,* 2005; Islam *et al.,* 2006a; Oliveira *et al.,* 2007; Estrela *et al.,* 2012). Além deste aspecto, apresentam semelhanças quanto à atividade antimicrobiana (Estrela *et al.,* 2000), biocompatibilidade (Camilleri *et al.,* 2005), selamento, adaptação marginal (Pereira *et al.,* 2004), estabilidade dimensional (Duarte *et al.,* 2003) e solubilidade (Chong *et al*., 2003). Apresentam capacidade de estimular o reparo dos tecidos periapicais (Bernabé *et al.,* 2007) e de formar barreira de tecido duro (Briso *et al.,* 2006). Essas semelhanças tem despertado interesse na avaliação do cimento Portland como alternativa ao MTA (Funteas *et al*., 2003; Danesh *et al.,* 2006; Oliveira *et al*., 2007)

Embora o MTA apresente muitas propriedades favoráveis, há preocupação com o custo elevado, o que limita sua utilização em todos os níveis de atenção à saúde (Oliveira *et al.,* 2007). Também têm sido relatadas limitações relacionadas com a manipulação e tempo de endurecimento (Camilleri *et al.,* 2005; Islam *et al.,* 2006a; Kogan *et al.,* 2006; Oliveira *et al.,* 2007). O fabricante recomenda a manipulação do MTA com água destilada, o que produz uma mistura com consistência de areia granulosa, a qual toma presa completamente em 2 horas e 45 minutos (Torabinejad *et al.,* 1995b). Além disso, existe a dificuldade para ser levada ao local de inserção e por fim ser compactada adequadamente (Kogan *et al.,* 2006). Os cimentos Portland não apresentam em sua forma original o óxido de bismuto (Estrela *et al*., 2000), o que confere menor radiopacidade e torna díficil a distinção dos tecidos duros (Duarte *et al*., 2009). Holland *et al.* (2007) investigaram o propilenoglicol como veículo para o o MTA e relataram, além da compatibilidade biológica, melhora nas condições de manipulação da mistura. Duarte *et al.* (2012) verificaram que a manipulação do MTA com o propilenoglicol aumentou o tempo de endurecimento, melhorou a fluidez, aumentou o pH e a liberação de íons cálcio nos períodos iniciais.

Recentemente, os produtos de origem natural vêm ganhando espaço dentro da Odontologia, muito em função de propriedades indesejáveis das soluções classicamente empregadas**.** Dentre aqueles, o *Aloe vera* vem conseguindo destaque na Odontologia em razão das suas propriedades terapêuticas (Wynn, 2005; Bhat *et al.,* 2011; Athiban *et al.,* 2012; Babaee *et al.,* 2012; Fani & Kohanteb, 2012)**.** O *Aloe vera*, cacto pertencente à família *Liliaceae*, é amplamente utilizado pela indústria farmacêutica devido às suas atividades anti-inflamatória (Vijayalakshmi *et al.,* 2012), antibacteriana (Fani & Kohanteb, 2012), antioxidante (Asadi-Shahmirzadi *et al.,* 2012), antiviral (Lowther *et al.,* 2012), antifúngica (Bernardes *et al.,* 2012) e aos efeitos hipoglicêmicos (Huseini *et al.,* 2012). Tem sido utilizado em casos de estomatite (Wynn, 2005; Babaee *et al.,* 2012) áftas ulceradas, queilites angulares (Babaee *et al.,* 2012), redução do sangramento, controle do processo inflamatório e do edema presentes nas doenças periodontais (Bhat *et al.,* 2011; Fani & Kohanteb, 2012). Na terapia endodôntica foi indicado como medicação intracanal, lubrificante durante o preparo biomecânico e também para descontaminação de cones de guta percha (Athiban *et al.,* 2012).

Considerando a importância da busca de novos medicamentos para a terapia endodôntica e a ausência de pesquisas, o objetivo deste estudo foi determinar propriedades físico-químicas dos cimentos Portland brancos estrutural e não estrutural, ProRoot MTA® e MTA BIO® associados à solução glicólica de *Aloe vera* a 2%.

2. OBJETIVOS

Objetivo Geral

Determinar a proporção pó/líquido e propriedades físico-químicas dos cimentos Portland brancos estrutural e não estrutural, ProRoot MTA® e MTA BIO® associados à solução glicólica de *Aloe vera* a 2%.

Objetivos Específicos

Determinar o tempo de endurecimento, solubilidade, alteração dimensional, pH e radiopacidade dos cimentos Portland brancos estrutural e não estrutural, ProRoot MTA® e MTA BIO® associados à solução glicólica de *Aloe vera* a 2%.

3. MATERIAL E MÉTODO

A composição química dos cimentos Portland branco estrutural, Portland branco não estrutural, MTA BIO® e ProRoot MTA® fornecida pelo fabricante, está descrita no Quadro 1 e os materiais utilizados estão ilustrados na Figura 1.

Quadro 1 - Descrição da composição química dos cimentos de acordo com informações do fabricante

Cimento	Composição	Fabricante
Portland branco estrutural	Clinquer branco (100-75%) gesso (3%) material carbonático (0-25%)	Votorantin, SP, Brasil
Portland branco não estrutural	Clinquer branco (74-50%), gesso (3%) material carbonático (26-50%)	Votorantin, SP, Brasil
MTA BIO®	Cimento Portland (80%) óxido de bismuto (20%)	Angelus Ind. Prod., PR, Brasil
ProRoot MTA®	Cimento Portland (75%), óxido de bismuto (20%) gesso (5%)	Dentsply Tulsa Dental, OK, USA

Os testes realizados nesse experimento seguiram a especificação n. 57 da *American National Standard Institute/American Dental Association* (ANSI-ADA) (2000) para materiais obturadores, a qual determina a realização dos testes nas condições ambientais de 23 ± 2°C de temperatura e 50 ± 5% de umidade relativa do ar, 48 horas antes do início dos procedimentos.

Figura 1. A - Cimento Portland branco não estrutural; B - Cimento Portland branco estrutural; C - MTA BIO®; D - ProRoot MTA®

Os materiais foram armazenados no laboratório onde foram desenvolvidos os testes, que continha dois aparelhos de ar condicionado, que possibilitaram o controle da temperatura, em torno de 23 ± 2℃. A umidade e a temperatura foram aferidas por meio de um termohigrômetro (Minipa ind. e com. ltda, São Paulo, SP, Brasil).

Determinação da proporção pó/líquido

Para a realização dos experimentos foi necessário estabelecer a relação pó/líquido, específica para cada cimento avaliado. Esta relação foi rigorosamente seguida durante a execução de todos os testes. O objetivo foi determinar a quantidade exata de pó que manipulada com um volume préestabelecido e fixo do líquido, pudesse fornecer um cimento com consistência de massa firme, que não escoasse, e fosse capaz de simular a situação de uso clínico. A solução glicólica de *Aloe vera* a 2% foi adquirida em farmácia de manipulação (Phloracea, Cuiabá, MT).

A relação pó/líquido foi obtida pesando-se 3 gramas de pó do cimento estudado em balança de precisão de 0,0001 grama (Ohaus Corporation, New Jersey, NJ, EUA). A seguir, 0,20 mL da solução foi colocado, com auxílio de uma pipeta graduada, ao lado do pó sobre uma placa de vidro, lisa e limpa, de 20mm de espessura. O pó foi incorporado à solução aos poucos, com auxílio de uma espátula metálica (SS White/ Duflex, Rio de Janeiro, RJ, Brasil) número 24 flexível, e submetido a espatulação vigorosa. Uma vez obtida a consistência adequada, a quantidade de pó que não foi utilizada durante a manipulação foi pesada e determinado, por subtração, a quantidade de pó efetivamente utilizada. Por meio de uma regra de três, determinou-se a quantidade de pó que foi necessária para a manipulação com 1 mL de solução glicólica de *Aloe vera* a 2%. Os testes foram realizados cinco vezes para cada material testado. A partir de então, estabeleceu-se a média aritmética desses valores para cada cimento espatulado.

Tempo de endurecimento

Para realização deste experimento foram confeccionados cinco moldes de aço inoxidável, cilíndricos, com diâmetros internos de 10mm e espessuras uniformes de 2mm, para cada material. O molde foi fixado, em sua face externa com auxílio de cera utilidade, sobre placa de vidro de 1mm de espessura por 25mm de largura e 75mm de comprimento (Figura 2A).

A seguir, o cimento a ser testado foi manipulado e colocado no interior do molde metálico, até que este ficasse totalmente preenchido (Figura 2B). Passados 120 ± 10 segundos do início da manipulação colocou-se o conjunto placa de vidro/molde/cimento em recipiente plástico com vedação hermética que foi mantido a temperatura constante de 37 ± 2℃ e 95 ± 5% de umidade relativa do ar, no interior de uma estufa (Olidef, ind. com. Aparelhos Hospitalares, Ribeirão Preto, SP, Brasil), até o final do teste. Decorridos 150 ± 10 segundos do início da manipulação, uma agulha tipo Gillmore de 100 ± 0,5 gramas e ponta ativa de 2 ± 0,1mm foi abaixada verticalmente sobre a superfície horizontal do material (Figura 2C). A colocação da agulha sobre o material foi repetida a intervalos regulares de sessenta segundos até que ela não provocasse mais indentações no cimento que estava sendo testado. O tempo de endurecimento do cimento foi dado como sendo o tempo decorrido entre o início da manipulação e o momento no qual as indentações deixaram de ser visíveis na superfície do cimento, realizado por três examinadores devidamente calibrados. O teste foi considerado finalizado apenas quando houve concordância entre os examinadores. Esse procedimento foi realizado cinco vezes para cada cimento. O tempo de endurecimento foi considerado como sendo a média aritmética das repetições.

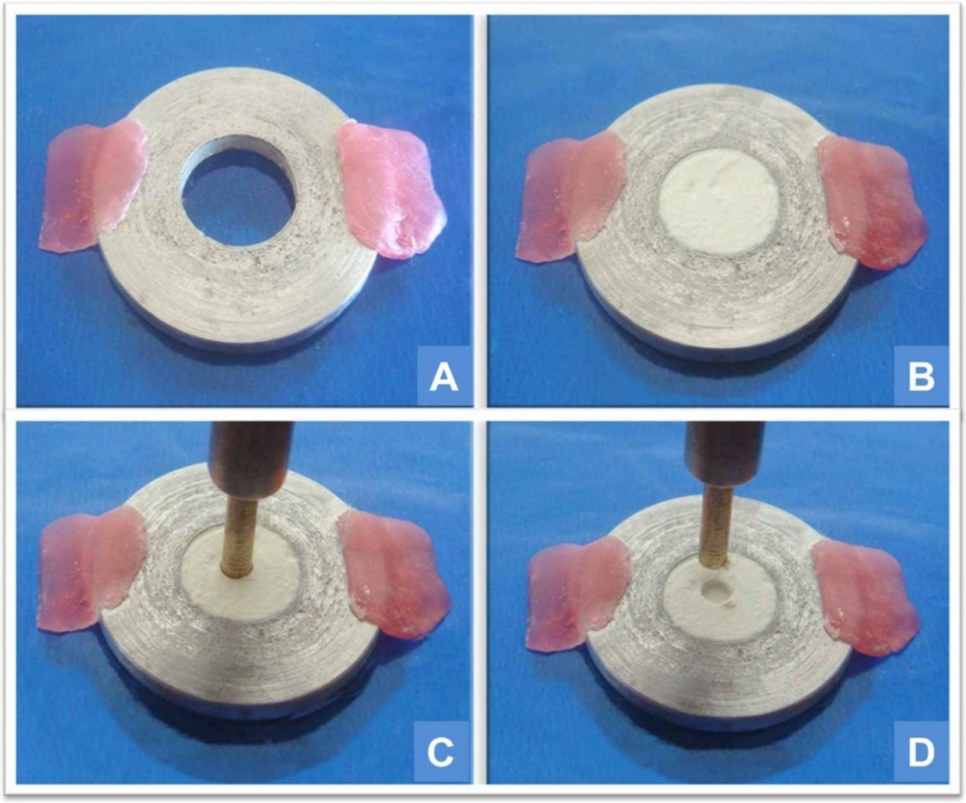

Figura 2. Sequência da técnica para execução do teste de tempo de endurecimento. A- Molde vazio; B- Molde preenchido com cimento; C- Agulha de Gillmore abaixada no cimento; D- Indentação no cimento, evidenciado que o cimento ainda não endureceu.

Solubilidade

Para a realização deste teste foram utilizados cinco moldes de Teflon circulares, com 1,5mm de espessura e 7,75mm de diâmetro interno, para cada cimento. Os moldes foram colocados sobre uma fina lâmina de celofane sustentada por uma placa de vidro de 40 X 80 X 5mm (Figura 3A). O cimento a ser testado foi manipulado e colocado dentro dos moldes. A seguir, foi inserido um fio de nylon impermeável de diâmetro de aproximadamente 0,5mm na massa do cimento amolecido (Figura 3B).

Posteriormente, foi colocada outra placa de vidro, de dimensões iguais às daquela colocada sob o cimento, envolta por lâmina de celofane, sobre os moldes preenchidos de material. Sobre esse conjunto, colocou-se um peso de

100 gramas. Todo esse conjunto foi transportado para uma câmara climatizada, com temperatura de 37 ± 2℃ e umidade relativa do ar de 95 ± 5%.

Decorrido intervalo de tempo três vezes maior ao de endurecimento de cada cimento, previamente determinado, as amostras foram removidas dos moldes (Figura 3C) e retirados quaisquer resíduos da superfície dos corpos de prova. Realizou-se a pesagem, com aproximação para 0,001 grama, em uma balança de precisão de 0,0001 grama (Ohaus Corporation, New Jersey, NJ, EUA).

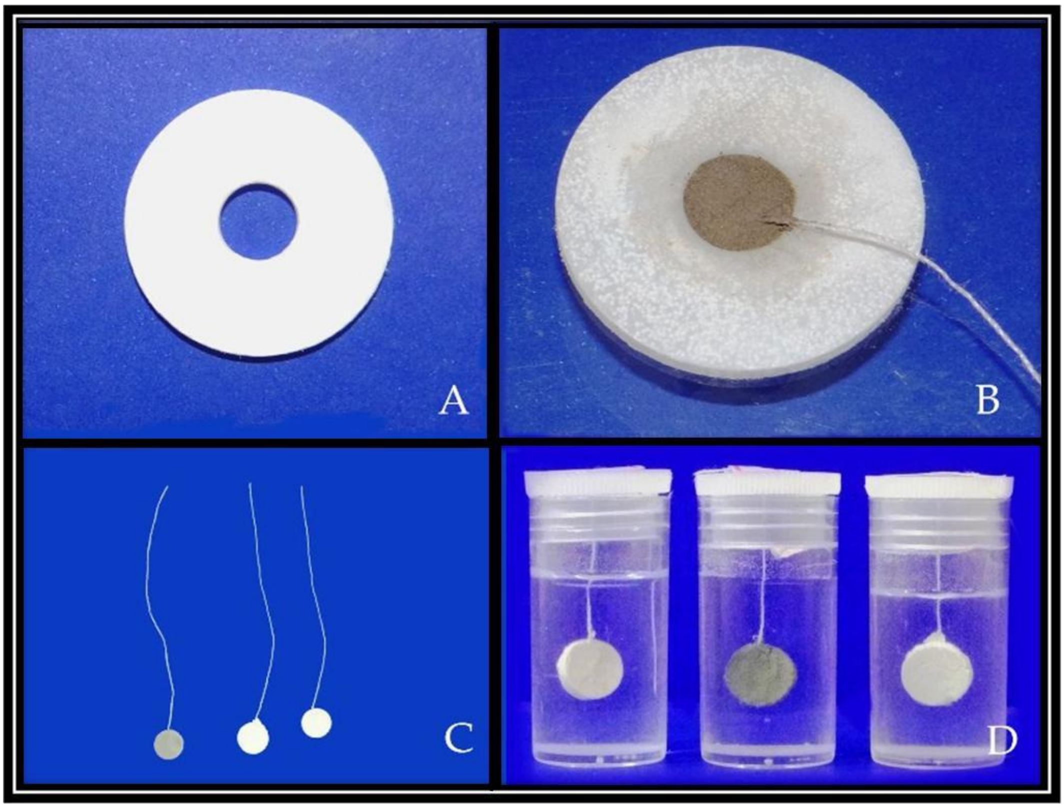

Figura 3. A - Molde circular de teflon confeccionado com 7,75mm de diâmetro interno e 1,5mm de espessura; B - Molde preenchido com o cimento e com fio de nylon inserido na massa do material ainda amolecido; C – Corpos de prova desinformados e pesados; D – Corpos de prova imersos em 7,5mL de água destilada.

As amostras suspensas pelos fios de nylon foram colocadas no interior de recipientes de plástico com boca larga, contendo 7,5 mL de água destilada, tomando-se o cuidado de não permitir nenhum contato entre as amostras e a superfície interna dos recipientes (Figura 3D). Os recipientes foram fechados e levados para estufa a 37 ± 2℃, onde permaneceram por 24 horas.

Decorrido esse prazo, as amostras foram removidas, enxaguadas, com água destilada, e retirados os excessos com a ajuda de lenço de papel absorvente. As amostras foram mantidas no desumidificador por 24 horas e depois retiradas para a segunda pesagem, aproximando-se novamente para 0,001 grama.

A perda de massa de cada amostra foi anotada e expressa como a porcentagem da massa original do material. Essa perda consiste na solubilidade do material testado.

A média de cinco realizações do teste foi considerada, aproximada para os 0,01 percentual, como sendo a solubilidade do cimento estudado.

Alteração dimensional

Foram preparados cinco moldes de Teflon® (polytetrafluroethylene, DuPont, HABIA, Knivsta, Sweden) com 3,58mm de altura e 3,0mm de diâmetro (Figura 4). Os moldes foram preenchidos com os cimentos e os conjuntos foram transportados para uma câmara climatizada, com temperatura de 37 ± 2℃ e umidade relativa do ar de 95 ± 5%, por período de tempo igual a três vezes o tempo de endurecimento do cimento. Após, as extremidades foram regularizadas com lixa úmida de granulação 600. Os moldes foram abertos, as amostras removidas e cada uma delas teve o comprimento medido com paquímetro digital. A seguir foram armazenadas em recipiente contendo 2,24 mL de água destilada a 37ºC por trinta dias; após esse período foram removidas dos recipientes, secas e os comprimentos medidos novamente. A porcentagem de alteração dimensional foi calculada usando a fórmula: [(L30-L)/L] X 100, onde L30 = comprimento da amostra após trinta dias e L= comprimento inicial da amostra. A média aritmética de cinco medidas para cada cimento foi registrada como alteração dimensional do cimento testado.

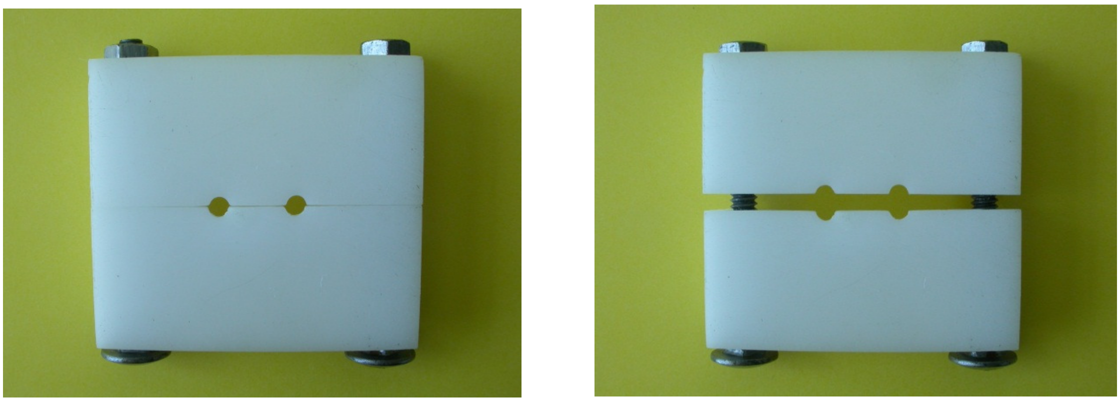

Figura 4. Molde para o teste de alteração dimensional

Avaliação do potencial hidrogeniônico (pH)

Para confecção dos corpos de prova, seguiu-se a mesma metodologia e padronização dos materiais empregados para o teste de solubilidade. Foram confeccionados cinco corpos de prova para cada cimento.

Inicialmente, o pH da água destilada foi medido, por meio de um pHmetro digital (PH30 SensorCorning; Corning Inc, New York, NY, USA) (Figura 5) antes da submersão do corpo de prova no recipiente. A medida do pH foi realizada em cinco recipientes e obtida a média aritmética.

Após a imersão do corpo de prova em 7,5mL de água destilada com pH de 5,68, o pH foi aferido nos intervalos de 1/4, 1/2, 1, 2, 3, 4, 6, 8, 12, 24, 48, 72, 96, 120, 144, 168 e 720 horas.

Para cada período determinado, obteve-se a leitura do pH do meio no qual foi imerso o corpo de prova. Dessa forma, foram obtidas cinco aferições para cada material, estabelecendo-se ao final, a média aritmética. Durante todo o experimento, a leitura do pH, para cada corpo de prova, era realizada sempre no mesmo recipiente plástico, sem que houvesse a substituição da água destilada.

Figura 5. Foto do pHmetro digital.

Radiopacidade

Cinco placas de acrílico (2,2cm x 4,5cm x1mm) foram confeccionadas, contendo orifícios com dimensões de 1mm de profundidade e 5mm de diâmetro interno (Figura 6A). Essas placas de acrílico foram colocadas sobre uma placa de vidro recoberta por uma lâmina de papel celofane. Os cimentos foram espatulados seguindo-se a proporção pó/líquido pré-estabelecida e os materiais introduzidos nos orifícios da placa de acrílico. Cada um dos orifícios foi preenchido com um dos cimentos estudados, seguindo a ordem do material com o maior para o de menor tempo de endurecimento. A disposição dos cimentos, devidamente identificados em cada uma das placas, sempre ocorreu na mesma posição, no sentido de padronizar e facilitar a localização dos materiais durante a análise da radiopacidade. As placas, após o preenchimento, foram armazenadas em estufa a 37 ± 2℃ e 95 ± 5% de umidade relativa do ar, durante período igual a três vezes o tempo de endurecimento dos cimentos.

Cada uma das placas de acrílico contendo os cimentos foi posicionada, no momento da exposição radiográfica, ao lado de outra placa de acrílico (1,3cm X 4,5cm X 1mm) (Figura 6B) contendo um penetrômetro de alumínio, confeccionado de liga 1100, com espessura variando de 1 a 10mm, em degraus uniformes de 1mm cada (ANSI/ADA, 2000) (Figura 6C).

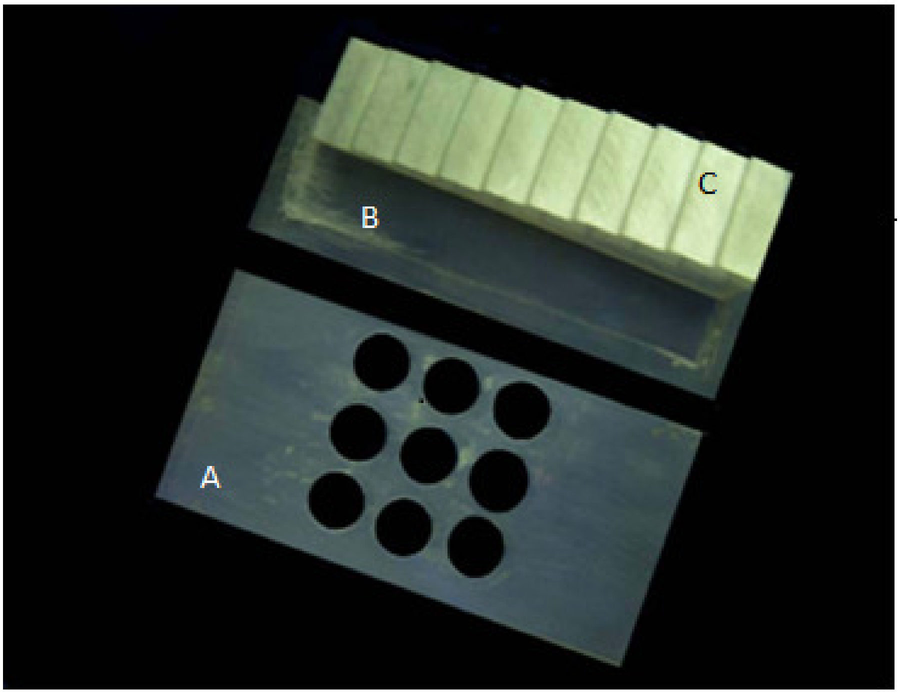

Figura 6. A- Placa de acrílico para inserção dos cimentos testados; B- Placa de acrílico; C – Penetrômetro de alumínio.

O conjunto das duas placas, uma contendo os cimentos e a outra o penetrômetro de alumínio, foi confeccionado com medidas padronizadas para que correspondesse ao tamanho exato do sensor (placa de fósforo) do sistema *Digora TM*® (Soredex Orion Corporation, Helsink, Finlândia), que foi usado para coletar as imagens radiográficas. Para tanto, utilizou-se um aparelho de raios-X Spectro 70X® (Dabi Atlante, Ribeirão Preto, SP, Brasil), de 70 kVp e 8 mA. A distância foco objeto utilizada foi de 30cm e o tempo de exposição de 0,2 segundos, como recomendado pelo fabricante para radiografia digital de placa de fósforo (Figura 7). Um estabilizador de voltagem foi utilizado para evitar a oscilação da energia, padronizando assim, a voltagem do aparelho de raios-X.

Para o perfeito posicionamento do sensor e assegurar que a distância foco-objeto fosse padronizada, um dispositivo posicionador de acrílico foi confeccionado com fixadores metálicos (Figura 7). O cabeçote do aparelho de raios-X foi fixado na mesma posição, direcionando o feixe central para incidir em ângulo de 90° com a superfície do conjunto placas de acrílico/sensor. Um colimador retangular (Dabi Atlante, Ribeirão Preto, SP, Brasil) com 3cm x 4cm de abertura foi acoplado na extremidade do cilindro para diminuir a possibilidade de radiação secundária. Antes das tomadas radiográficas, realizou-se a calibração prévia do aparelho de raios-X com o sensor. O mesmo sensor foi usado em todas as exposições para evitar possíveis diferenças entre as placas acrílicas. A dose de radiação foi padronizada de acordo com o *software Digora® for Windows 1.5*, empregado para realizar as leituras.

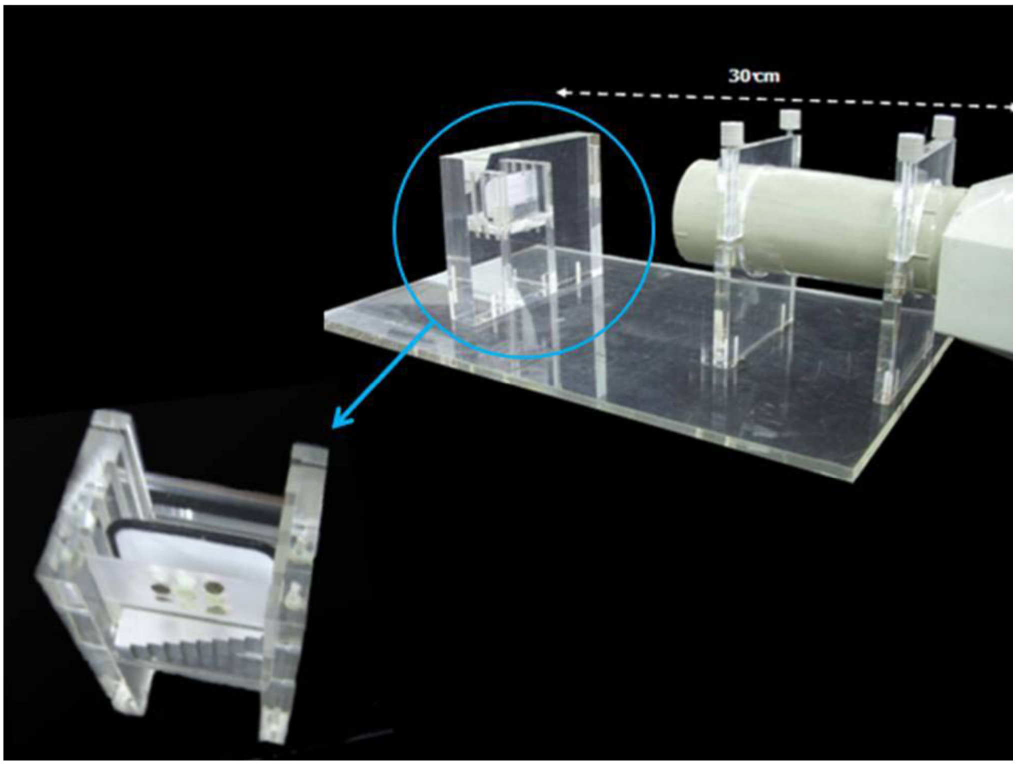

Figura 7. Dispositivo posicionador de acrílico, com fixadores metálicos, para manter o aparelho de raios-X em posição fixa. Em maior aumento, o aparato formado pela placa de acrílico, escada de alumínio e sensor de radiografia digital.

Após as tomadas radiográficas, o sensor foi introduzido no dispositivo de leitura óptica laser do sistema *Digora*®. O aparelho tem a capacidade de transformar o sinal analógico em digital e, por meio do software (*Digora*® *for Windows 1.5*), quantificar a densidade da imagem radiográfica (análise densitométrica), diretamente na tela do computador.

Assim que a primeira imagem foi evidenciada na tela do aparelho foram estabelecidos parâmetros oferecidos pelo próprio sistema *Digora*®, que permitiu a padronização da imagem. Foram utilizados os valores dos tons de cinza da área analisada, em torno de $2mm^2$, de cada cimento e de cada degrau do penetrômetro de alumínio, correspondentes a 30 x 30 pixels, identificados pelas coordenadas dos eixos X e Y, visualizados na tela do aparelho. A imagem padronizada oferecia densidade e contraste adequados, proporcionando qualidade e facilidade de visualização durante a leitura. Quando o sensor foi novamente introduzido no aparelho, a nova imagem projetada, enquadrava-se dentro dos parâmetros pré-estabelecidos, evitando assim, que esta nova imagem apresentasse tonalidades mais claras ou mais escuras que a imagem padrão.

Por meio da imagem revelada na tela, o aparelho realizava a leitura da densidade radiográfica de cada cimento da placa de acrílico, assim como de cada degrau do penetrômetro de alumínio, fornecendo um valor numérico para cada leitura (Figura 8), registrado por um especialista em Radiologia. Após a análise das cinco placas de acrílico, foram estabelecidas cinco medidas para cada tipo de cimento e cada degrau do penetrômetro de alumínio. A partir de então foram determinadas as médias das densidades radiográficas, respectiva para cada material e degrau da penetrômetro de alumínio.

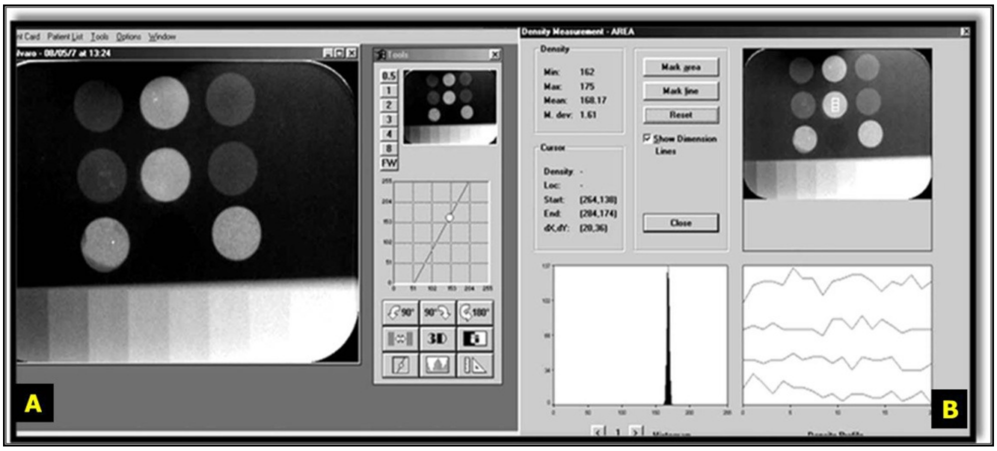

Figura 8. Imagens radiográficas dos corpos de prova para análise da radiopacidade dos cimentos (A e B), por meio do software do programa *Digora® for Windows 5.1*.

Análise estatística

Para análise estatística da proporção pó/líquido e dos testes de tempo de endurecimento, solubilidade e variação do pH, os dados originais foram submetidos a testes preliminares, visando verificar a normalidade da amostra, com auxílio do software GMC 8.1, desenvolvido pelo Prof. Dr Geraldo Maia Campos, da Faculdade de Odontologia de Ribeirão Preto, Universidade de São Paulo (Campos, 2001). Para o teste de radiopacidade, foi realizada análise estatística descritiva por meio de interpretação de tabelas e gráficos das médias obtidas.

Quando a distribuição amostral foi normal, aplicou-se a análise de variância. Na sequência, aplicou-se o teste complementar de *Tukey*, no sentido de verificar quais materiais seriam diferentes entre si, em nível de significância de 5%. Quando a distribuição amostral foi não normal, aplicou-se o teste de *Kruskal Wallis*, para evidenciar diferença entre os materiais, em nível de significância de 5%.

4. RESULTADOS

Proporção pó/líquido

Não foram observadas diferenças significantes na proporção pó/líquido entre os cimentos testados, que variou entre 3,14 e 3,52 gramas de pó para 1 mL da solução glicólica de *Aloe vera* a 2%, conforme descrito na Tabela 1.

Tabela 1. Proporção pó-líquido (gramas de pó/ 1 mL de água destilada) dos cimentos- Valores originais,médias e desvio padrão*.*

Cimento / Amostra	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®
1	3,21	3,52	3,41	3,28
2	3,35	3,10	3,66	3,63
3	3,15	3,63	3,68	3,50
4	3,13	3,47	3,52	3,16
5	3,51	3,69	3,34	3,49
Média ± dp	**3,14 ± 0,21 a**	**3,48 ± 0,23 a**	**3,52 ± 0,14 a**	**3,41 ± 0,18 a**

Letras iguais significam não haver diferenças significantes ($p>0,05$).

Tempo de endurecimento

Os resultados da análise de variância mostraram haver diferença estatisticamente significante entre os cimentos ($p<0,05$). O MTA BIO® apresentou o menor tempo de endurecimento (66,75 ± 1,71 minutos), enquanto que o ProRoot MTA ® apresentou o maior (116,50 ± 4,08 minutos). Não foram observadas diferenças entre os cimentos Portland brancos estrutural (85,75 ± 2,87 minutos) e não estrutural (84,00±3,16 minutos) ($p>0,05$), embora tenham sido diferentes de outros grupos ($p<0,05$), de acordo com os dados apresentados na Tabela 2.

Tabela 2. Tempo de endurecimento (em minutos) dos cimentos estudados- Valores originais, médias e desvio padrão

Cimento / Amostra	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®
1	84,50	80,50	66,50	118,50
2	87,50	83,50	67,50	119,50
3	81,50	84,50	68,50	115,50
4	87,50	87,50	**64,50**	112,50
5	85,15	85,20	66,70	116,00
Média ± dp	**85,23 ± 2,49 a**	**84,54 ± 2,56 a**	**66,74 ± 1,48 b**	**116,40 ± 2,75 c**

Letras diferentes significam diferencas estatisticamente significantes (p<0,05).

Solubilidade

Os resultados da análise estatística mostraram maior solubilidade para os cimentos Portland branco estrutural (2,30 ± 0,36%) e não estrutural (2,81 ± 0,40%) (p<0,05), porém semelhantes entre si (p>0,05). O ProRoot MTA® apresentou valores intermediários (1,74 ± 0,13%). O MTA BIO ® apresentou a menor solubilidade (1,45 ± 0,07%) (p<0,05), conforme enunciado na Tabela 3.

Tabela 3. . Solubilidade (em porcentagem) dos cimentos- Valores originais, médias e desvio padrão

Cimento / Amostra	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®
1	2,27	3,19	1,34	1,89
2	1,82	3,07	1,47	1,65
3	2,31	2,49	1,54	1,84
4	2,86	3,04	1,48	1,56
5	2,27	2,28	1,44	1,77
Média ± dp	**2,30 ± 0,36 a**	**2,81 ± 0,40 a**	**1,45 ± 0,07 b**	**1,74 ± 0,13 c**

Letras diferentes significam diferencas estatisticamente significantes (p<0,05).

Alteração Dimensional

Tabela 4. Alteração dimensional (em porcentagem) dos cimentos- Valores originais, médias e desvio padrão

Cimento / Amostra	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®
1	1,00	1,00	1,00	1,00
2	1,00	1,01	1,00	1,00
3	1,01	1,00	1,00	1,00
4	1,00	1,04	1,01	1,00
5	1,01	1,02	1,01	1,00
Média ± dp	**1,00 ± 0,00 a**	**1,01 ± 0,01 a**	**1,00 ± 0,00 a**	**1,00 ± 0,00 a**

Letras iguais significam não haver diferenças significantes (p>0,05).

Os cimentos testados apresentaram valores de alteração dimensional estatisticamente não significantes e semelhantes entre si (1,00±00%).

Potencial hidrogeniônico (pH)

A análise estatística mostrou não haver diferença significante em relação ao pH dos cimentos estudados (p>0,05). O pH das soluções estabeleceram-se na faixa de valores elevados do início ao final dos testes, variando entre 11,77 ± 0,39 (ProRoot MTA®) e 12,07 ± 0,33 (MTA BIO®), cujos resultados estão descritos na Tabela 5.

Tabela 5. Médias das leituras do pH da solução de imersão dos corpos de prova por intervalo de tempo-Valores médios

Cimento / Tempo (h)	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®
1/4	11,41	11,38	11,34	11,03
1/2	11,69	11,50	11,63	11,27
1	11,80	11,61	11,96	11,37
2	11,87	11,75	12,10	11,62
3	11,94	11,77	12,12	11,70
4	11,91	11,73	12,09	11,70
6	11,94	11,71	12,09	11,72
8	11,73	11,51	11,82	11,50
12	11,68	11,46	11,75	11,43
24	11,88	11,78	11,87	11,66
48	12,38	12,18	12,34	12,15
72	12,44	12,23	12,43	12,23
96	12,24	12,15	12,33	12,17
120	12,53	12,41	12,69	12,47
144	12,29	12,20	12,45	12,22
168	12,11	12,04	12,21	12,11
720	11,63	11,33	12,07	11,75
Média ± dp	**11,96 ± 0,31 a**	**11,80 ± 0,33 a**	**12,07 ±0,33 a**	**11,77 ± 0,39 a**

Letras iguais significam não haver diferenças significantes (p>0,05).

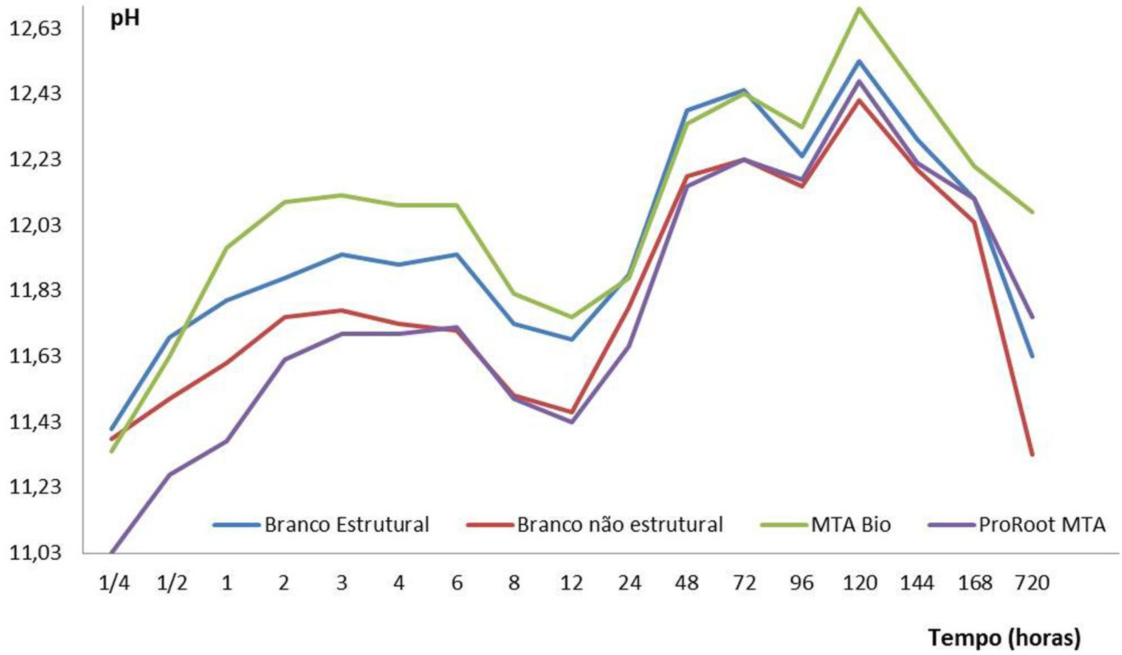

Figura 9. Variação do pH dos cimentos em função do tempo.

Teste de Radiopacidade

A análise estatística mostrou que o ProRoot MTA® e MTA BIO® apresentaram valores de densidade radiográfica superiores ao degrau 3 do penetrômetro (157,95 ± 8,33 mmAl e 156,83 ± 12,98 mmAl, respectivamente) ($p<0,05$), porém estatisticamente semelhantes entre si ($p>0,05$). Os cimentos Portland brancos estrutural (109,76 ± 10,40 mmAl) e não estrutural (118,75 ± 15,44 mmAl) apresentaram valores de densidade radiográfica inferiores ao degrau 2 do penetrômetro de alumínio, o que está demonstrado na Tabela 6.

Tabela 6- Médias e desvio padrão da radiopacidade (em milímetros de alumínio) dos cimentos.

Cimento / Placa	Branco Estrutural	Branco não estrutural	MTA BIO®	ProRoot MTA®	Degrau 3
1	107,45	109,17	164,74	155,81	138,60
2	104,31	106,60	140,02	153,76	131,22
3	95,04	102,04	143,17	146,31	129,91
4	119,82	137,77	168,23	166,35	148,83
5	122,05	132,00	167,97	167,33	155,19
Média ± dp	**109,76 ± 10,40 a**	**118,75 ± 15,44 a**	**156,83 ± 12,98 b**	**157,95 ± 8,33 b**	**140,75 ± 10,40 c**

5. DISCUSSÃO

As propriedades físico-químicas dos materiais odontológicos devem ser constantemente avaliadas, especialmente frente à necessidade de manter o controle de qualidade. Em condições clínicas, materiais retrobturadores e obturadores de canal radicular ficam em contato íntimo com os tecidos periodontais e periapicais. Por essa razão, bem como pelo fato de até o presente momento não existirem critérios específicos para testar as propriedades físico-químicas dos materiais retrobturadores, as pesquisas com os cimentos à base de MTA e Portland, seguem a especificação n. 57 da ANSI/ADA (2000), para materiais obturadores do canal radicular (Kogan *et al.,* 2006; Duarte *et al.,* 2012), seguindo as modificações propostas por Carvalho-Júnior *et al.* (2007).

O MTA desde sua introdução na endodontia foi eleito um material de eficácia no selamento de perfurações radiculares, o que estimulou várias pesquisas. O MTA associado à água destilada como veículo tem apresentado curto tempo de trabalho, longo tempo de endurecimento e deficiente consistência (Camilleri *et al.,* 2005; Islam *et al.,* 2006a; Kogan *et al.,* 2006; Oliveira *et al.,* 2007). Essas caracerísticas estimularam a busca de veículos alternativos com vistas a suprir possíveis limitações nas propriedades físico-químicas destes materiais (Kogan *et al.,* 2006; Holland *et al.,* 2007; Wiltbank *et al.,* 2007; AlAnezi *et al.,* 2011; Duarte *et al.,* 2012). O efeito do *Aloe vera* na proporção pó/líquido, tempo de endurecimento, solubilidade, alteração dimensional, potencial hidrogeniônico e radiopacidade dos cimentos à base de MTA (ProRoot MTA® e MTA BIO®) e Portland brancos, estrutural e não estrutural, foi alvo do presente estudo.

Os cimentos à base de MTA e Portland brancos, manipulados com solução glicólica de *Aloe vera* a 2%, atenderam as recomendações da ANSI/ADA (2000) no que se refere ao tempo de endurecimento, solubilidade e alteração dimensional. Os cimentos Portland brancos associados à solução glicólica de *Aloe vera* a 2% evidenciaram valores de radiopacidade abaixo da recomendação mínima da especificação n. 57 da ANSI/ADA (2000). Os cimentos á base de MTA (ProRoot MTA® e MTA BIO®), quanto à radiopacidade, apresentaram valores dentro das exigências mínimas.

As características físico-químicas dos cimentos à base de MTA são influenciadas por diversos fatores tais como o tamanho das partículas, temperatura ambiente, umidade da cavidade no momento da aplicação, umidade relativa do ar, incorporação do pó na solução e proporção pó/líquido (Torabinejad *et al.,* 1995b). Portanto, é fundamental determinar a quantidade exata de pó a ser incorporado em um volume específico de solução. Os materiais à base de MTA são manipulados com água destilada na proporção 3:1, de acordo com as instruções do fabricante. O cimento Portland foi projetado para a construção civil e a proporção específica para seu uso na odontologia ainda não foi determinada. Contudo, em alguns estudos o cimento Portland foi utilizado na mesma proporção dos cimentos à base de MTA (Islam *et al.,* 2006a; Song *et al.,* 2006). A proporção de 3:1 resulta em uma mistura muito fluida, dificultando ou impedindo a aplicação (Fridland & Rosado, 2003). Neste estudo, a proporção pó/líquido variou de 3,14 para 3,52 gramas de pó para 1 mL de solução glicólica de *Aloe vera* a 2%, indicando que a quantidade de pó na mesma quantidade de líquido estava próxima para os diferentes materiais. Não foram observadas diferenças significantes entre os cimentos, o que reforça que a composição química do MTA e cimento Portland é semelhante (Estrela *et al.,* 2000*,* 2012; Funteas *et al.,* 2003; Islam *et al.,* 2006a; Song *et al.,* 2006).

Holland *et al.* (2007) avaliaram a influência do tipo de veículo (água destilada ou propilenoglicol) na resposta dos tecidos apicais de cães após o preenchimento do canal radicular com MTA, considerando dois diferentes limites. Os resultados evidenciaram que as pastas de MTA manipuladas com água destilada ou propilenoglicol apresentaram comportamento biológico semelhante. Além disso, os autores relataram maior facilidade no preenchimento dos canais radiculares com a mistura MTA/propilenoglicol. Duarte *et al.* (2012) avaliaram a influência do propilenoglicol nas propriedades químicas do MTA branco. A adição do propilenoglicol como veículo ao MTA aumentou o tempo de endurecimento, melhorou a fluidez, aumentou o pH e a liberação de íons cálcio nos períodos iniciais.

No presente estudo, a manipulação de solução glicólica de *Aloe vera* a 2% ao MTA e cimento Portland resultou em um material com aspecto de massa de vidraceiro, facilitou a manipulação e pode favorecer o seu uso em situações clínicas. A especificação n. 57 da ANSI/ADA (2000) preconiza que a consistência do cimento obturador deve ser tal que, ao levantar a espátula, o cimento a ela aderido demore de 10 a 15 segundos para cair. Além disso, quando a superfície plana da espátula for colocada sobre a mistura e levantada lentamente da placa de vidro, deverá formar um fio de cimento de pelo menos 2,5cm, que une a espátula à massa de cimento que está sobre essa placa. Contudo, as características dos materiais retrobturadores à base de MTA, bem como as condições clínicas de uso são diferentes dos cimentos obturadores de canal. Bernabé & Holland (2004) esclarecem que o índice de consistência normal de uma pasta de cimento Portland é utilizado para verificação de duas características importantes do cimento: os tempos de pega e a instabilidade do volume. Para que a execução dos ensaios seja uniforme e os resultados sejam comparáveis, é necessário que as pastas de cimento apresentem as mesmas características. No caso de pastas e cimentos, se forem igualadas as viscosidades, todos os ensaios acontecerão dentro das mesmas condições. Segundo os mesmos autores, a consistência normal é característica de cada cimento e sua determinação é feita por tentativas (Bernabé & Holland, 2004).

O emprego do *Aloe vera* como veículo não afetou as propriedades físico-químicas desses cimentos. O *Aloe vera*, além de possuir ação terapêutica (Wynn, 2005; Bhat *et al.,* 2011; Fani & Kohanteb, 2012; Babaee *et al.,* 2012) é um veículo com características viscosas e solúvel em água.

A principal desvantagem quando do uso clínico dos cimentos à base de MTA, como material retrobturador, está no seu tempo de endurecimento prolongado. A hidratação dos aluminatos tem relação direta com o gesso, que quando ausente promove uma reação instantânea, levando ao endurecimento imediato do cimento (Camilleri *et al*., 2007).

Com o objetivo de diminuir o tempo de endurecimento dos cimentos à base de MTA, Kogan *et al.* (2006) investigaram a influência da solução salina, lidocaína a 2%, gel de hipoclorito de sódio a 3%, gel de gluconato de clorexidina, gel K-Y e cloreto de cálcio a 3% e 5%, nas propriedades do MTA. Foram observadas a diminuição do tempo de endurecimento e melhora na manipulação com gel de hipoclorito de sódio. Enfatizam a possibilidade de sua utilização na manipulação com o MTA em casos de sessão única, quando não é necessária uma barreira adicional para proteger o MTA. Wiltbank *et al.* (2007) adicionaram aceleradores do cimento Portland (cloreto de cálcio, nitrito/nitrato de cálcio e formato de cálcio) ao MTA, cinza e branco, e cimento Portland. Os três aditivos aceleraram significativamente a reação de presa dos materiais testados. AlAnezi *et al.* (2011) relataram que a manipulação de KY líquido, cloreto de cálcio e hipoclorito de sódio ao MTA cinza melhorou a manipulação, diminuiu o tempo de endurecimento e apresentou biocompatibilidade semelhante ao MTA cinza misturado com água.

Torabinejad *et al.* (1995b) reportaram que o tempo de endurecimento para o ProRoot MTA é menor que 4 h, enquanto que Kogan *et al.* (2006) relataram tempo de endurecimento de 50 min, quando da mistura com água destilada. Essas diferenças podem ser atribuídas aos diferentes métodos utilizados, como aqueles que utilizam as agulhas de Gillmore (ANSI/ADA, 2000) e a técnica de Vicat (ISO- 6876/ 2001). Chng *et al.* (2005) determinaram que os tempos de endurecimento, inicial e final do ProRoot MTA, pelos métodos descritos pela ISO 6876 (2001), são de aproximadamente 70 e 175 min, respectivamente. Essas diferenças também poderiam ser explicadas pelas alterações na composição do pó do MTA ocorridas desde a sua introdução na odontologia (Asgary *et al.,* 2005; Kogan *et al.,* 2006).

No presente estudo não houve diferença estatisticamente significante para o tempo de endurecimento entre os cimentos Portland branco estrutural (85,75 min) e o não estrutural (84,00 min). Entre os demais cimentos, a diferença observada foi significante em 5% ($p<0,05$). O cimento Portland é constituído de gesso (3%) e clínquer (97%), que por sua vez, apresenta o calcário e a argila como matéria prima. O gesso age fundamentalmente como retardador de presa, prolongando o tempo de endurecimento do material. Em contato com a água e sem a presença de gesso, o cimento endureceria quase que instantaneamente (Associação Brasileira de Cimento Portland, 2002). O cimento MTA BIO® apresentou o menor tempo de endurecimento (66,75 min) e o ProRoot MTA® o maior (116,50 min). Apesar de ambos serem à base de MTA, o cimento MTA BIO® é constituído de 80% de cimento Portland e 20% de óxido de bismuto e não contém quantidade adicional de sulfato de cálcio (Duarte *et al.*, 2003; Song *et al.*, 2006; Oliveira *et al.,* 2007). Oliveira *et al.* (2007) afirmaram que a ausência de sulfato de cálcio reduz o tempo da reação de presa do cimento.

O cimento ProRoot MTA®, além dos dois componentes anteriores possui acréscimo de 5% de sulfato e cálcio na sua composição (Torabinejad & White, 1995; Ferris & Baumgartner, 2004), o que aumenta significantemente o tempo de endurecimento em relação ao MTA BIO®. Os resultados observados neste estudo são condizentes com aqueles obtidos por Ferris & Baumgartner (2004) e Song *et al.* (2006) que argumentaram que apesar da concentração de cimento Portland ser maior para o MTA BIO® (80%) em relação ao ProRoot MTA® (75%), este último possui maior adição de sulfato de cálcio.

Vale salientar que, devido ao tempo de endurecimento prolongado do ProRoot MTA® tem-se adicionado a este cimento, hidróxidos ou aluminatos, visando acelerar o endurecimento do material (Islam *et al.*, 2006b; Bortoluzzi *et al.*, 2006).

Borges (2008), manipulando cimentos à base de MTA com água destilada, relataram tempos de endurecimento menores do que os observados neste estudo (MTA BIO® 33,10 min e ProRoot MTA® 90,90 min), o que poderia ser explicado pelos diferentes veículos utilizados. Os resultados do presente trabalho estão de acordo com Duarte *et al*. (2012), que empregaram diferentes concentrações de propilenoglicol/água destilada como veículo para manipulação do MTA BIO®. Relataram aumento do tempo de endurecimento proporcional ao aumento da porcentagem de propilenoglicol na solução propilenoglicol/água destilada. O tempo de endurecimento variou de 85 min (solução composta de 20% de propilenoglicol e 80% de água destilada) a 661 min (solução composta de 80% de propilenoglicol e 20% de água destilada).

Na cirurgia apical, além do contato do material retrobturador com os tecidos periapicais, este também está sujeito à dissolução pelo fluxo sanguíneo (Wiltbank *et al.,* 2007). Também deve ser considerado que de maneira ideal, um material que tenha curto tempo de endurecimento é desejável. De acordo com a ANSI/ADA (2000), o tempo de endurecimento do cimento não deve exceder a 10% do tempo informado pelo fabricante.

Os materiais retrobturadores devem ser resistentes à solubilidade em meio aquoso. De acordo com a ANSI/ADA (2000), a solubilidade dos cimentos obturadores não pode exceder a 3% de sua massa. Os resultados da solubilidade para todos os cimentos testados atenderam as recomendações da ANSI/ADA (cimento Portland branco não estrutural, 2,81%; cimento Portland branco estrutural, 2,30%; ProRoot MTA® 1,74%; MTA BIO® 1,45%). O MTA BIO® apresentou o menor percentual de solubilidade, fato este já relatado em estudos anteriores (Funteas *et al.,* 2003; Asgary *et al.,* 2005; Chng *et al.,* 2005; Song *et al.,* 2006). A diferença entre o MTA BIO® e outros cimentos pode estar relacionada com a composição química, que apresenta diferentes estruturas após a reação de presa (Camilleri, 2007).

A alteração dimensional é outra propriedade importante que necessita ser considerada juntamente com o tempo de endurecimento. Uma alteração nessa propriedade, possivelmente levando à contração, poderia ter impacto negativo sobre a capacidade do MTA em selar o ápice radicular ou reparar a perfuração (Wiltbank *et al.,* 2007). A padronização ANSI/ADA (2000) indica que o cimento não deve exceder a 1% de retração linear média ou a 0,1% de expansão.

No presente estudo, todos os cimentos testados apresentaram valores de alteração dimensional em torno de 1%, não havendo diferenças significantes entre os cimentos. A ligeira expansão observada durante a presa pode ser explicada pela absorção de água pelos cimentos (Camilleri, 2007). Este fato é útil para garantir que o selamento está presente durante a presa do material e, consequentemente, reduz a infiltração posterior (Chng *et al.,* 2005; Camilleri, 2007).

A biocompatibilidade do MTA está relacionada ao seu elevado pH (Kogan *et al.,* 2006). Portanto, o veículo não pode afetar negativamente essa propriedade. No presente estudo, todos os cimentos testados apresentaram pH elevado quando manipulados com *Aloe vera*. O MTA BIO ® apresentou valores médios ligeiramente maiores (12,07). Os resultados obtidos neste estudo estão próximos aos encontrados por Torabinejad *et al*. (1993), que constataram, logo após a hidratação do material com água destilada, pH em torno de 10,2 para o ProRoot MTA®, passando para 12,5, após 3 horas, mantendo-se, então, estável. Os valores obtidos por Duarte *et al*. (2003) foram menores aos observados neste trabalho. A diferença deve-se, provavelmente, à metodologia utilizada, uma vez que trocavam o meio aquoso a cada mensuração realizada.

Dessa forma, logo após uma leitura, o pH da solução retornava próximo ao da água destilada, requerendo um intervalo de tempo maior para atingir um pH mais elevado.

Os cimentos à base de MTA e os cimentos Portland apresentam elevada quantidade de óxido de cálcio (Estrela *et al.,* 2012*).* Em contato com o fluido tecidual ou água, o óxido de cálcio transforma-se em hidróxido de cálcio que se dissocia em íons cálcio e íons hidroxila, resultando em aumento do pH e liberação de íons cálcio. O mecanismo de ação entre o hidróxido de cálcio e o MTA mostrou ser similar (Holland *et al*., 1999). Diversos estudos foram realizados para avaliar o pH do MTA (Torabinejad *et al.,*1995b; Duarte *et al.,* 2003, 2012).Os valores de pH observados para o MTA misturado com água destilada (Torabinejad *et al.,*1995b; Duarte *et al.,* 2003) foram menores que os encontrados neste estudo. O mesmo foi observado quando o MTA foi misturado com propilenoglicol (Duarte *et al.,* 2012). Esta discrepância poderia ser explicada pela diferença na metodologia utilizada entre os estudos e pela diferença na composição dos cimentos.

Os materiais retrobturadores devem apresentar radiopacidade suficiente para serem distinguidos das estruturas anatômicas adjacentes, como o dente e osso alveolar, além de evidenciar espaços vazios e contornos inadequados (Torabinejad *et al.,* 1995b; Laghios *et al.,* 2000; Rasimick *et al*, 2007). De acordo com a especificação n. 57 da ANSI/ADA (2000), o material obturador de canal deve apresentar radiopacidade correspondente a pelo menos 3mm de alumínio. Os dados da Tabela 6 mostraram que o ProRoot MTA® foi o material com maior radiopacidade (157,95mmAl), seguido pelo MTA BIO® (156,83mmAl), não havendo diferenças entre eles. A despeito dos cimentos à base de MTA conterem óxido de bismuto em sua composição, responsável pela radiopacidade (Estrela *et al.,* 2000; Funteas *et al.,* 2003; Camilleri *et al.,* 2005; Danesh *et al.,* 2006) menor quantidade de óxido de bismuto foi detectada no MTA BIO® quando em comparação ao ProRoot MTA® (Danesh *et al.,* 2006; Islam *et al.,* 2006a). Este fato poderia explicar a menor radiopacidade do MTA BIO® em comparação com o ProRoot MTA®. Além disso, estudo recente usando energia dispersiva com raios-X, demonstrou a existência de pequena diferença na concentração de óxido de bismuto entre os cimentos MTA cinza (15,19%) e branco (14,61%) (Estrela *et al.,* 2012). A fórmula original do cimento Portland não apresenta óxido de bismuto (Estrela *et al.,* 2000), conferindo menor radiopacidade e tornando difícil distinguí-lo dos tecidos duros (Duarte *et al.,* 2009).

No corrente estudo, os valores médios obtidos para os cimentos Portland brancos foram mais baixos que 2mmAl, não alcançando o valor mínimo exigido pela ANSI/ADA (2000), resultados de acordo com a literatura (Borges *et al*., 2011). Para suprir a deficiência de radiopacidade, diferentes agentes radiopacificadores foram adicionados ao cimento Portland, com resultados satisfatórios (Duarte *et al.,* 2009).

Novos estudos devem ser desenvolvidos em busca de avaliar possíveis infuências do *Aloe vera* no mecanismo de ação químico e biológico quando associados aos materiais analisados. Portanto, mais estudos, *in vitro* e *in vivo,* são necessários para verificação da biocompatibilidade, propriedades antimicrobianas e selamento do MTA associado ao *Aloe vera*, anterior à sua recomendação para uso clínico.

6. CONCLUSÕES

Com base na metodologia empregada e nos resultados obtidos, pode-se concluir que:

1) O cimento ProRoot MTA® evidenciou o maior tempo de endurecimento, enquanto o MTA BIO® o menor;

2) O cimento MTA BIO® apresentou a menor solubilidade. A maior solubilidade foi verificada com o cimento Portland branco não estrutural. Nenhum cimento estudado mostrou solubilidade maior que 3%;

3) Os cimentos testados apresentaram valores de alteração dimensional semelhantes entre si;

4) Todos os cimentos testados tiveram o mesmo comportamento em relação a análise de pH, mantendo-se elevado por todo o período de teste;

5) Apenas os cimentos ProRoot MTA® e MTA BIO apresentaram valores de radiopacidade acima do degrau 3 do penetrômetro de alumínio

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PUBLICAÇÃO

Artigo: Physicochemical properties of MTA-based and Portland cements associated to *Aloe vera*

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Revista (Submetido): International Endodontic Journal

Running title: MTA-based and Portland cements associated to *Aloe vera*

Key Words: Mineral trioxide aggregate, portland cement, root-end filling materials, physicochemical properties, aloe vera

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Abstract:

Aim: The aim of this study was to determine the solution-powder ration and to evaluate the setting time, solubility, dimensional change, pH and radiopacity of White structural and non-structural Portland cement, ProRoot MTA® and MTA BIO®associated to 2% glycolic solution of *aloe vera*. ***Material and Methods:*** Five samples of each material were used for each test according to American National Standards Institute/American Dental Association specification 57. Statistical analyses were carried out using ANOVA and Tukey's test at 5% level of significance. When sample distribution was non-normal, nonparametric analysis of variance were performed with Kruskal-Wallis test (α=0.05).***Results:*** The results revealed no statistical difference in the water-powder ratio among the tested materials. ProRoot MTA® had the longest setting time (116.50 ± 4.08) ($P<0.05$). White structural and non-structural Portland cements did not have means values on the radiopacity test (109.76 ± 10.40 and 118.75 ± 15.44, respectively) in accordance with ANSI/ADA, being significantly different from the MTA-based cements ($P<0.05$), and demonstrated higher mean solubility values (2.30 ± 0.36 and 2.81 ± 0.40, respectively) ($P<0.05$). The values for dimensional change of all tested materials were considered acceptable by ANSI/ADA. There were no significant differences between the mean pH values recorded for the materials ($p>0.05$). It was alkaline from the beginning to the end of the tests. ***Conclusion:*** The physiochemical properties of the tested materials in association with *aloe vera* conformed to ANSI/ADA's requirements, except white Portland cements, which did not fulfill the ANSI/ADA specification for radiopacity.

Introduction

The Mineral Trioxide Aggregate (MTA) was developed by Professor Mahmoud Torabinejad, in the 1990s, at Loma Linda University, for to seal lateral root perforations (Lee *et al.*, 1993) and as a root-end filling material (Torabinejad *et al.*, 1995ab, 1997). Since its introduction and its approval by the US Federal Drug Administration, MTA has rapidly gained popularity due its biocompatibility and superior sealing ability (Torabinejad *et al.*, 1995ab). MTA can also be used for a variety of other clinical applications including direct pulp caps, repair of furcal perforations, as a dressing over dental pulp after pulpotomy, during the treatment of teeth with open apices and repair of resorptive defects (Darvell & Wu, 2011).

The principal compounds of MTA are tricalcium silicate, tricalcium aluminate, tricalcium oxide and silicate oxide (Lee *et al.*, 1993; Torabinejad *et al.*, 1995b). According to MTA patent, its, basically, composed by ordinary type 1 Portland cement (PC), a material used in civil engineering, and bismuth oxide to improve the radiopacity properties (Estrela *et al.*, 2000; Funteas *et al.*, 2003; Camilleri *et al.*, 2005; Danesh *et al.*, 2006). Currently, two MTA forms are commercially available: ProRoot MTA® (Dentsply Tulsa Dental, OK, USA) and MTA BIO® (Angelus Indústria de Produtos Odontológicos Ltda, Londrina, PR, Brazil). The composition of ProRoot MTA® is reported to be 50-65% calcium oxide and 15-25% silicium dioxide (Dammaschke *et al.*, 2005; Camilleri *et al.*, 2005), while MTA BIO® is composed by 80% of PC and 20% of bismuth oxide (Duarte *et al.*, 2003; Song *et al.*, 2006; Oliveira *et al.*, 2007). Both materials, MTA and PC, are available in white and gray formulations, according to the presence of iron oxide (Asgary *et al*., 2005; Camilleri *et al*., 2005; Islam *et al*., 2006). Considering the presence of carbonatic material, substance related to the material resistance, white Portland cement are also classified as structural and non structural (ABCP, 2002).

The chemical similarity between MTA and PC has been investigated and with the exception of bismuth oxide, MTA and PC have components in common (Camilleri *et al*., 2005; Dammasche *et al*., 2005; Islam *et al*., 2006; Oliveira *et al*., 2007; Estrela *et al*., 2012). Futhermore, MTA and PC have similar antimicrobial activity (Estrela *et al*., 2000), biocompatibility (Camilleri *et al*., 2005), sealing ability, marginal adaptation (Pereira *et al.,* 2004), dimensional stability (Duarte *et al*., 2003) and moisture tolerance (Chong & Pitt Ford, 2005). In addition, MTA and PC stimulate healing of the periradicular tissues (Bernabé *et al*., 2007) and dentine barrier formation (Briso *et al*., 2006). These similarities have been generated interest in the evaluation of PC as a substitute for MTA (Danesh *et al*., 2006).

Although MTA has many favorable properties, there have been concerns about its elevated cost, which makes it impossible to be used in all levels of health attention (Oliveira *et al*., 2007). The others clinical problems with MTA are it extended setting time and difficult handling characteristic (Camilleri *et al*., 2005; Islam *et al*., 2006; Kogan *et al*., 2006; Oliveira *et al*., 2007). The manufacturer recommends mixing MTA with sterile water. This produces a grainly, sandy mixture, which takes 2h and 45 min to completely set (Torabinejad *et al*., 1995b) and is typically difficult to deliver to the required site and hard to compact adequately (Kogan *et al*., 2006). Holland *et al*. (2007) stated that would be of interest to investigate another vehicle, rather than water and saline, which would facilitate the use of MTA.

Aloe vera (AV), a cactus plant that belongs to the Liliaceae family, has been extensively used in pharmaceutical industry due its anti-inflammatory (Vijayalakshmi *et al.*, 2012), antibacterial (Fani & Kohanteb, 2012), antioxidant (Asadi-Shahmirzadi *et al.*, 2012), antiviral (Lowther *et al.*, 2012) and antifungal actions (Bernardes *et al.*, 2012) and due it hypoglycemic effects (Huseini *et al.*, 2012). Potential dental utilities have been explored such as in cases of denture stomatitis (Wynn, 2005; Babaee *et al.*, 2012) apthous ulcers, cracked and split corners of the mouth (Babaee *et al.*, 2012), reducing bleeding, inflammation and swelling in periodontal diseases (Bhart *et al.*, 2011; Fani & Kohanteb, 2012). In endodontic therapy, it is used as intracanal dressing, file lubrication during biomechanical preparation and also for decontaminating gutta-percha cones (Athiban *et al.*, 2012). The effects of the AV on physicochemical properties of MTA and PC have no been evaluated.

The purpose of this in vitro study was to determine the solution-powder ration, setting time, solubility, hydrogenionic potential (pH) and radiopacity of white structural and non-structural Portland cement, ProRoot MTA® and MTA BIO® associated to glycolic solution of aloe vera, according to American National Standards Institute and American Dental Association (ANSI/ADA) standards (2000).

Materials and methods

Tested Materials

The materials used in this study are described in Table 1.

Table 1- Tested materials and compositions.

Cement	Composition*	Manufacturers
White structural Portland	White clinker (100-75%), gypsum (3%) and carbonate material (0-25%)	Votorantin, SP, Brazil
White non-structural Portland	White clinker (74-50%), gypsum (3%) and carbonate material (26-50%)	Votorantin, SP, Brazil
MTA BIO®	Portland cement (80%) and bismuth oxide (20%)	Angelus Ind. Prod., PR, Brazil
ProRoot MTA®	Portland cement (75%), bismuth oxide (20%) and gypsum (5%)	Dentsply Tulsa Dental, OK, USA

*information according to manufactures

Initially, different concentrations of the glycolic solution of aloe vera were tested to observe which one presented better conditions to use. At the end of the test, 2% glycolic solution was chosen. The aloe vera was obtained from a manipulation pharmacy The solution/powder ratio was initially established by weighing 3 g of the tested cement and after that mixing it with 0.20 mL of 2% glycolic solution of *Aloe vera*. This procedure was repeated 5 times for each material.

The setting time of the cements was determined according to methods prescribed by Specification 57 of the American National Standard Institute/American Dental Association (ANSI/ADA) for endodontic sealing materials (2000). The solubility, pH and dimensional changing tests were realized as suggested by Carvalho-Júnior et al. (2007), allowing the reduction of 80% in volume of material for conducting tests, without involvement or interference in results. One evaluator blinded to the identification of the materials made all the analysis.

Setting time

Five stainless steel molds, with 10 mm-inner diameters and 2 mm-uniform thicknesses were made for each tested material. The cement was mixed and insert into the metallic molds. After 120 ± 10 seconds from the beginning of the mixture, the set formed by the glass plaque/metallic mold/cement was left in a plastic recipient with hermetic sealing and maintained at a constant temperature of 37 ± 2° C and 95 ± 5% air humidity, inside an incubator (Olidef, Ind. E Com. Aparelhos Hospitalares, Ribeirão Preto, SP, Brazil), until the end of the test. After 150 ± 10 seconds from the beginning of the mixture, a Gilmore needle (100 ± 0.5 g and 2 ± 0.1 mm active tip) was vertically lowered into the horizontal surface of the material. The needle was inserted at regular intervals of 60 seconds until the indentations could not be observed on the cement surface. The time was determined as the time from the beginning of the mix until the time at which the indentations were not visible on the cement surface.

Solubility

Five samples (1.5 mm thickness and 7.75 mm-inner diameter) were used for each material. The cement was prepared and inserted into the mold. In sequence, a 0.5 mm-diameter waterproof nylon was inserted in the softened cement. After three times the setting time, the sample was removed from the mold and weighed on a precision scale of 0.0001 g (Ohaus Corporation, New Jersey, NJ, USA). The sample suspended by the nylon was placed in a wide-mouthed plastic recipient containing 7.5 mL of distilled water, avoiding the contact with the internal wall. This container was maintained hermetically closed and placed in an incubator at a constant temperature of 37 ± 2°C for 24 hours. After this time, the sample was removed and the excess water removed with absorbent paper. The sample was maintained in dehumidifier for 24 hours, after which it was weighed a second time. The material's solubility was considered as the percentage of lost mass compared to the initial mass. Five repetitions were considered for each material.

Dimensional change

Five Teflon® (polytetrafluroethylene, DuPont, HABIA, Knivsta, Sweden) molds were prepared with 3.58 mm of height and 3.0 mm of diameter. The wells of the Teflon molds were filled with the material, stored, and had their ends ground with a wet 600-grit SiC paper to obtain a regular surface. The samples were removed from the mold, the length was measured, and they were stored in a vessel containing 2.24 mL of distilled water at 37°C for 30 days. The sample was removed from the container, dried, and measured again for length. The percentage of the dimensional alterations was calculated by using the following formula: [(L30-L)/L] X 100, where L30 = length of the sample after 30 days and L= initial length of the sample. The arithmetic mean of 5 replicates for each sealer was recorded as the dimensional alteration of the cement tested.

Hydrogenionic potential (pH)

Five samples (1.5 mm thickness and 7.75 mm inner diameter) were used for each material. Each cylinder was sealed in a flask containing 7.5 mL of distilled water. Distilled water pH measurements (PH30 SensorCorning; Corning Inc, New York, NY, USA) were taken with a pH meter at 1/2, 1, 2, 3, 4, 6, 8, 12, 24, 48, 72, 96, 120, 144, 168 hours and 30 days after spatulation. During the experiment, pH was analyzed for each sample in the same plastic recipient without liquid substitution. It was measured five times for each material. Mean values and standard deviations were recorded for all measurements.

Radiopacity test

Five acrylic plates (2.2 cm x 4.5 cm x 1 mm) with 6 holes measuring 1 mm in depth and 5 mm of internal diameter were fabricated. The acrylic plates were placed onto a glass plate covered by cellophane paper and each orifice was filled with one of the tested cements. For the radiographic exposure, each acrylic plate containing the cements was positioned together with another acrylic plate (1.3 cm x 4.5 cm x 1 mm), which contained a graduated aluminum stepwedge varying from 1 to 10 mm in thickness, and uniform steps of 1 mm each. The set of plates was built with standardized measurements in a way that they would correspond exactly to the sensor size (phosphor plate), from DigoraTM® system (Soredex, Orion Corporation, Helsink, Finland), used for data collection. A 70 kVp and 8 mA radiograph machine, Spectro 70X (Dabi Atlante, Dabi Atlante Indústrias Médico Odontológicas Ltda, Ribeirão Preto, SP, Brazil), was used. The focus-object distance was 30 cm (ANSI/ ADA 2000) and exposure time at 0.2 s, as instructed for digital radiography of phosphor plates, by the manufacturer. An acrylic positioning device with metallic fastener held sensors and provided an adequate and standardized focus-object distance. The radiograph machine head was fixed on the same position with central beam presenting 90° angle of incidence with the acrylic/sensor surface plates set. A rectangular collimator (Dabi Atlante, Dabi Atlante Indústrias Médico Odontológicas Ltda) presenting 3x4 cm aperture reduced possible secondary radiation by being attached to the end of cylinder. The sensor, after being exposed, was inserted into the laser optical reader of DigoraTM® for Windows 5.1 software. As soon as the first image was revealed on screen, parameters suggested by the system were established, allowing to image standardization. The same phosphor plate was used for all exposures to avoid possible differences between plates. The system performed a radiographic density reading over images of each cement revealed on screen, and also a reading of steps on an aluminum stepwedge, resulting in a numeric value for each reading. This value was written down by the evaluator. After evaluating the 5 acrylic set of plates, 5 measurements for each type of cement and for each step of the aluminum scale were obtained. Mean values of radiographic density and graduated aluminum stepwedge were determined for each material. Mean values were taken by a single evaluator previously trained and blinded with regard to the different groups.

Statistical analysis

Statistical analyses were carried out for solution/powder ratio, setting time, solubility, dimensional changing, pH and radiopacity using ANOVA and Tukey's test at 5% level of significance. When sample distribution was non-normal, nonparametric analysis of variance were performed with Kruskal-Wallis test (α=0.05).

Results

The results are presented on Table 2. No statistical difference in the water/powder ratio was observed among the cements tested. The mean amount of powder needed, in grams, when mixed into 2% glycolic solution of AV was presented in Table 2.

Table 2. Mean values and standard deviations of each tested material.

Test	White Structural	White non-structural	MTA BIO	ProRoot MTA
Water/powder	3,14 ± 0,21ª	3,48 ± 0,23ª	3,52 ± 0,14ª	3,41 ± 0,18ª
Setting time (min)	85,75 ± 2,87ª	84,00 ± 3,16[a]	66,75 ± 1,71[b]	116,50 ± 4,08[c]
Solubility	2,30 ± 0,36[a]	2,81 ± 0,40[a]	1,45 ± 0,07[b]	1,74 ± 0,13[c]
Dimensional change	1,00 ± 0,00[a]	1,01 ± 0,01[a]	1,00 ± 0,00[a]	1,00 ± 0,00[a]
pH	11,96 ± 0,31[a]	11,80 ± 0,33[a]	12,07 ±0,33[a]	11,77 ± 0,39[a]
Radiopacity	109,76 ± 10,40[a]	118,75 ± 15,44[a]	156,83 ± 12,98[b]	157,95 ± 8,33[b]

*The same superscript letters represents no statistically significant difference (P<0.05).

Setting time

There were statistically significant differences among the cements ($p<0.05$). MTA BIO had the lowest value, while ProRoot MTA® had the highest. No difference was observed between white structural and non-structural Portland cements ($p>0.05$), although they were different from the others (p<0.05). In summary, ProRoot MTA®> White structural Portland cement > White non-structural Portland cement > MTA BIO®.

Solubility

Statistical analysis showed higher solubility for White structural Portland and White non-structural Portland cements, which were statistically similar among themselves (p>0.05). ProRoot MTA® showed intermediate values, followed by MTA BIO® (p<0.05), which showed the inferior solubility (p<0.05). In summary, White non-structural Portland cement > White structural Portland cement > ProRoot MTA®> MTA BIO®.

Dimensional change

ANSI/ADA specification 57 (2000) states that maximum limits are 1% for linear shrinkage and 0.1% for expansion. The values for dimensional change of all tested materials are considered acceptable by ANSI/ADA (Table 2).

pH

There were no significant differences between the mean pH values recorded for the materials (p>0.05). It was alkaline from the beginning to the end of the tests.

Radiopacity test

All cements except White Portland cements showed radiopacity above the 3 mm of aluminum recommended by ANSI/ADA Specification 57 (2000). Statistical analysis showed that ProRoot MTA® and MTA BIO® presented the superior results, which were statistically similar between themselves. In summary, ProRoot MTA®> MTA BIO®

> White non-structural Portland cement > White structural Portland cement.

Discussion

The physicochemical properties of MTA and PC have been exhaustively studied. Previous studies have demonstrated that MTA mixed with water presents a short working time, delayed setting and poor consistency (Camilleri *et al.,* 2005; Islam *et al.,* 2006; Kogan *et al*., 2006; Oliveira *et al*., 2007). Since the hydration behavior of MTA is significantly influenced by the surrounding environment (Wang *et al*., 2010), different materials have been used in an attempt to improve the handling and set time properties of MTA and to facilitate its clinical use (Kogan *et al*., 2006; Holland *et al*., 2007; Wiltbank *et al*., 2007; AlAnezi *et al*., 2011 Duarte *et al*., 2012). The present study evaluated the effect of AV on the solution-powder ration, setting time, solubility, hydrogenionic potential (pH) and radiopacity of MTA-based and Portland cements.

Kogan *et al*. (2006) investigated the influence of various additives (saline, 2% lidocaine, 3% NaOCl gel, chlorhexidine gluconate gel, K-Y Jelly, 3% and 5% CaCl2) on setting properties of MTA and observed decreased setting time and the superior handling properties with NaOCl gel. For the authors, it could be potentially used to mix with MTA in a single visit scenario where an additional barrier to protect the MTA is not required. Wiltbank *et al*. (2007) added classic PC accelerators (calcium chloride, calcium nitrite/nitrate and calcium formate) to gray and white MTA and PC and observed that all 3 additives significantly accelerated the setting reaction of the tested materials. AlAnezi *et al*. (2011) found that to mixture KY liquid, CaCl2, and NaOCl to gray MTA improved the handling properties, decreased setting time and allowed osteoblasts and fibroblast attachment and spread similar to gray MTA mixed with water.

Holland *et al*., (2007) investigated the influence of the type of vehicle (distilled water or propyleneglycol) on the response of the apical tissues of dog's teeth after root canal filling with MTA at two different limits. The results showed that MTA pastes prepared with either distilled water or propyleneglycol as vehicles had similar biological behavior. Duarte *et al*. (2012) evaluated the influence of propylene glycol on chemical properties of white MTA. The results revealed that mixing propyleneglycol as the liquid vehicle for MTA increased the setting time, improved the flowability, increased the pH and the calcium ion release of the cement in the initial periods. In the present study, the mixture of 2% of glycolic solution of AV to MTA and PC cements resulted in a material with putty aspect. It was easier to manipulate and could favor its use in clinical situations. In addition, AV presents therapeutic actions (Wynn, 2005; Bhart *et al*., 2011; Fani & Kohanteb, 2012; Babaee *et al*., 2012) and is a water-soluble vehicle with viscosity characteristics. Although the biocompatibility of this vehicle in combination with MTA and PC has not yet been investigated, the results of the present study demonstrated that the use of AV as a vehicle did not affected the physicochemical properties of MTA and PC.

Due to the lack of specific standards to test the physical properties of retrofilling materials, published studies have followed the ANSI/ADA specification number 57 for endodontic sealing materials (Kogan *et al.*, 2006; Duarte *et al.*, 2012) and the ISO 6876 specification for zinc oxide and eugenol endodontic sealing materials (Torabinejad *et al.*, 1995b), to support and to reference studies on analysis of physicochemical properties of MTA and PC. Under clinical conditions, both retrofilling and root filling materials remain in close contact with the periodontal tissues, thus, the ANSI/ADA standard was assumed to be applicable to the materials under investigation (Danesh *et al.*, 2006; Martins *et al.*, 2009) following the modifications proposed by Carvalho-Júnior *et al.* (2007).

MTA is a powder that consists of fine hydrophilic particles that harden when they come in contact with water (Lee *et al.*, 2003). The physicochemical characteristics of MTA are influenced by several factors: such as, the type of MTA, the type of storage media, the type of vehicle, the size of the particles, the temperature and humidity at the application, amount of air trapped in the mixture, the mixing procedure itself and the powder-to-water ratio (Torabinejad *et al.*, 1995b; Dammaschke *et al.*, 2005; Kogan *et al.*, 2006; Islam *et al.*, 2006, Danesh *et al.*, 2006; Nekoofar *et al.*, 2007). Fridland & Rosado (2003) stated that the standardization of methods to study the physicochemical properties of MTA is hampered by the lack of control of several of the above mentioned factors, thus, different results might be obtained.

Foremost, is critical to determine the exact amount of powder to be incorporated into a specific volume of the solution. MTA-based cements are mixed with sterile water in a 3:1 ratio according to the manufacturer´s instruction. PC is designed for civil engineering, and a specific ratio for its use in dentistry has not been determined. Thus, in many studies PC is often used in a same ratio as MTA-based cements (Islam *et al*., 2006; Song *et al*., 2006). In some applications, the powder-water ratio proportion of 3:1 results in a very fluid mix that has a consistency similar of soup, hindering application (Fridland &Rosado, 2003). Thus, has been suggested the addition of extra amounts of powder to the mixture or placing the cement under gauze until it acquires suitable consistency for use (Nekoofar *et al*., 2009). The effect of these additional maneuvers on the physicochemical properties of the cements is unknown. In this study, the powder-water ratio was determined to verify the exact quantity of powder that should be incorporated in to a specific volume of the vehicle. The mean powder-water ratio varied from 3.14 to 3.52 g of powder to 1 mL of 2% AV solution. The results indicated that cements with different powder-liquid ratio were not statistically different ($P<0.05$), reinforcing that the chemical composition of MTA and PC are similar (Funteas *et al*., 2003; Song *et al.,* 2006; Islam *et al*., 2006; Estrela *et al*., 2012).

In apical surgery, root-end filling material is placed in contact with periapical tissues and is subject to washout by blood flow (Wiltbank *et al*., 2007). Therefore, a material with a shorter setting time is desirable. According to de ADA, the setting time of the sealer should be within 10% of the stated by the manufacturer. The setting time test showed significant difference between all tested materials. ProRoot MTA® showed the highest value (116.50 minutes); followed by white structural PC (85.75 minutes) and white nonstructural PC cements (84.00 minutes). ProRoot MTA®

’s long setting is one of the major drawbacks of the material and might be explained by its lower amount of gypsum and aluminum species (Dammaschke *et al*., 2005). Torabinejad *et al*. (1995b) investigated the physical and chemical properties of MTA and reported a setting time higher than that observed in the present study (165 minutes), this difference might be attributed to changes in the composition of MTA powder since it was first introduced (Asgary *et al.,* 2005; Kogan *et al*., 2006). The lowest setting time value was observed in MTA BIO® (66.75 minutes). This material is composed of 80% PC and 20% bismuth oxide and no calcium sulphate (gypsum) (Duarte *et al*., 2003; Song *et al*., 2006; Oliveira *et al*., 2007). Oliveira *et al*. (2007) stated that the absence of calcium sulphate is advertised to reduce the setting time reaction.

The dimensional change is another important property that needs to be considered in addition to setting time. A change in that property, possibly leading to contraction, would likely have a negative impact on MTA's ability to seal a root end or perforation site (Wiltbank *et al*., 2007). ANSI/ADA standardization (2000) states that the mean linear shrinkage of the sealer shall not exceed 1% or 0.1% in expansion. In the present study, all dimensional change values were in accordance with ANSI/ADA specification, with no significant difference between the cements. The slight expansion on the setting observed may be explained by the water absorption by the cements (Camilleri, 2007). This fact is helpful in ensuring that the seal is present upon setting and consequently reducing subsequent leakage (Chng *et al*., 2005; Camilleri, 2007).

Root-end filling materials should have resistance to solubility and disintegration in an aqueous environment. ANSI/ADA (2000) establishes that solubility of sealers should not exceed 3% by mass. Solubility results of all tested materials were within ANSI/ADA recommendations (white non-structural PC, 2.81%; white structural PC, 2.30%; ProRoot MTA® 1.74%; MTA BIO® 1.45%). MTA BIO® was less soluble than others, which is consistent with results of previous studies (Funteas *et al.*, 2003; Asgary *et al.*, 2005; Chng *et al.*, 2005; Song *et al.*, 2006). This difference between this cement and others ones may be related to the chemical composition, which showed different structures after hardening reaction (Camilleri, 2007). Lower setting time may be one of the reasons for the greater solubility of PC (Danesh *et al.*, 2006).

The biocompatibility of MTA is related to its elevated pH (Kogan *et al.*, 2006). Thus, a vehicle must not adversely affect this property. In the present study, all tested materials presented alkaline pH levels when mixed with AV. Slightly higher mean value were observed for MTA BIO®. MTA-based and PC cements present high amount of calcium oxide (Estrela *et al.*, 2012), which in contact to tissue fluid or water is converted to calcium hydroxide. This is then dissociated into calcium and hydroxide ions, resulting in increased pH and calcium ion release (Duarte *et al.*, 2003; Holland *et al.*, 2007). A series of studies has been conducted to evaluate the pH of MTA (Torabinejad *et al.* 1995b; Duarte *et al.* 2003; Duarte *et al.*, 2012). The pH values observed for MTA mixed with distilled water (Torabinejad *et al.* 1995b; Duarte *et al.* 2003) were lower than those found in the present study. The same was observed when MTA was mixed with propyleneglycol (Duarte *et al.*, 2012). This discrepancy could be explained by the difference in the experimental design between the studies and by the difference in the materials composition.

Retrofilling materials should present enough radiopacity to be radiographically distinguished from surrounding structures, such as tooth and alveolar bone, and to reveal empty spaces and inappropriate contours (Torabinejad *et al.*, 1995b; Laghios *et al.*, 2000). According to the ANSI/ADA specification number 57 (2000), an endodontic sealer material should present radiopacity correspondent to at least 3 mm Al. Data in Table 2 show that ProRoot MTA® was the most radiopaque material (157.95 ± 8.33) followed by MTA BIO® (156.83 ± 12.98), with no significant difference between them. Despite both MTA-based cements contain in their composition bismuth oxide, which is responsible for radiopacity (Estrela *et al.*, 2000; Funteas *et al.*, 2003; Camilleri *et al.*, 2005; Danesh *et al.,* 2006), lower quantity of bismuth oxide have been observed in MTA BIO (Danesh *et al.*, 2006; Islam *et al.*, 2006). This fact could justify the lower radiopacity of MTA BIO® in comparison to ProRoot MTA®. In addition, a recent study using energy-dispersive X-ray microanalysis observed slight difference in the concentration of bismuth oxide between gray (15.19%) and white (14.61%) MTA cements (Estrela *et al.*, 2012). The original formulation of PC does not present bismuth oxide (Estrela *et al.*, 2000), determining its low radiopacity and making it difficult to be distinguished from hard tissues (Húngaro Duarte *et al.*, 2009). This is a major disadvantage of PC if it is to be employed clinically. In the current study, the mean values obtained for this cement were lower than 2 mm Al, not reaching the minimum requirements of ANSI/ADA (2000). The inadequate radiopacity of PC has been overcome by the addition of different radiopacifying agents with satisfactory results (Húngaro Duarte *et al.*, 2009).

The results obtained with MTA associated with AV are promising. However, it is unknown, whether, the use of AV as a vehicle would interfere with the mechanism of action of MTA and which histological events would occur in the periapical region. Thus, further standardized in vitro and in vivo studies are indicated to check the biocompatibility, antimicrobial properties, and sealing abilities of MTA associated to AV before any recommendation for clinical use.

Conclusion

Based on the employed methodology and obtained results, the following conclusion may be drawn: MTA and PC mixed with AV as a vehicle were in accordance with ANSI/ADA's requirements. Except white Portland cements that did not fulfill the ANSI/ADA's specification for radiopacity.

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Anexo 1. Normas de publicação do periódico

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International Endodontic Journal

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Author Guidelines

Content of Author Guidelines: 1. General, 2. Ethical Guidelines, 3. Manuscript Submission Procedure, 4. Manuscript Types Accepted, 5. Manuscript Format and Structure, 6. After Acceptance

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1. GENERAL

International Endodontic Journal publishes original scientific articles, reviews, clinical articles and case reports in the field of Endodontology; the branch of dental sciences dealing with health, injuries to and diseases of the pulp and periradicular region, and their relationship with systemic well-being and health. Original scientific articles are published in the areas of biomedical science, applied materials science, bioengineering, epidemiology and social science relevant to endodontic disease and its management, and to the restoration of root-treated teeth. In addition, review articles, reports of clinical cases, book reviews, summaries and abstracts of scientific meetings and news items are accepted.

Please read the instructions below carefully for details on the submission of manuscripts, the journal's requirements and standards as well as information concerning the procedure after a manuscript has been accepted for publication in *International Endodontic Journal*. Authors are encouraged to visit Wiley Blackwell Author Services (http://authorservices.wiley.com/bauthor/) for further information on the preparation and submission of articles and figures.

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Authors submitting a paper do so on the understanding that the manuscript has been read and approved by all authors and that all authors agree to the submission of the manuscript to the Journal.

International Endodontic Journal adheres to the definition of authorship set up by The International Committee of Medical Journal Editors (ICMJE). According to the ICMJE, authorship criteria should be based on 1) substantial contributions to conception and design of, or acquisiation of data or analysis and interpretation of data, 2) drafting the article or revising it critically for important intellectual content and 3) final approval of the version to be published. Authors should meet conditions 1, 2 and 3.

Acknowledgements: Under acknowledgements please specify contributors to the article other than the authors accredited. Please also include specifications of the source of funding for the study and any potential conflict of interests if appropriate.

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Experimentation involving human subjects will only be published if such research has been conducted in full accordance with ethical principles, including the World Medical Association Declaration of Helsinki (http://www.wma.net/en/20activities/10ethics/10helsinki/index.html) (version 2008) and the additional requirements, if any, of the country where the research has been carried out. Manuscripts must be accompanied by a statement that the experiments were undertaken with the understanding and written consent of each subject and according to the above mentioned principles. A statement regarding the fact that the study has been independently reviewed and approved by an ethical board should also be included. Editors reserve the right to reject papers if there are doubts as to whether appropriate procedures have been used.

When experimental animals are used the methods section must clearly indicate that adequate measures were taken to minimize pain or discomfort. Experiments should be carried out in accordance with the Guidelines laid down by the National Institute of Health (NIH) in the USA regarding the care and use of animals for experimental procedures or with the European Communities Council Directive of 24 November 1986 (86/609/EEC) and in accordance with local laws and regulations.

All studies using human or animal subjects should include an explicit statement in the Material and Methods section identifying the review and ethics committee approval for each study. The authors MUST upload a copy of the ethical approval letter when submitting their manuscript. Editors reserve the right to reject papers if there is doubt as to whether appropriate procedures have been used.

2.3 Clinical Trials

Clinical trials should be reported using the guidelines available at www.consort-statement.org (http://www.consort-statement.org). A CONSORT checklist (http://www.consort statement.org/mod_product/uploads/CONSORT%202001%20checklist.doc) and flow diagram (as a Figure) should also be included in the submission material.

The International Endodontic Journal encourages authors submitting manuscripts reporting from a clinical trial to register the trials in any of the following free, public clinical trials registries: www.clinicaltrials.gov (http://www.clinicaltrials.gov), http://clinicaltrials.ifpma.org/clinicaltrials/ (http://clinicaltrials.ifpma.org/clinicaltrials/), http://isrctn.org/ (http://isrctn.org/). The clinical trial registration number and name of the trial register will then be published with the paper.

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International Endodontic Journal requires that all sources of institutional, private and corporate financial support for the work within the manuscript must be fully acknowledged, and any potential conflicts of interest noted. Grant or contribution numbers may be acknowledged, and principal grant holders should be listed. Please include the information under Acknowledgements.

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Manuscript that do not conform to the general aims and scope of the journal will be returned immediately without review. All other manuscripts will be reviewed by experts in the field (generally two referees). International Endodontic Journal aims to forward referees´ comments and to inform the corresponding author of the result of the review process. Manuscripts will be considered for fast-track publication under special circumstances after consultation with the Editor.

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Supporting Information: *International Endodontic Journal* encourages submission of adjuncts to printed papers via the supporting information website (see submission of supporting information below). It is encouraged that authors wishing to describe novel procedures or illustrate cases more fully with figures and/or video may wish to utilise this facility.

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Abbreviations: International Endodontic Journal adheres to the conventions outlined in Units, Symbols and Abbreviations: A Guide for Medical and Scientific Editors and Authors. When non-standard terms appearing 3 or more times in the manuscript are to be abbreviated, they should be written out completely in the text when first used with the abbreviation in parenthesis.

5.2. Structure

All manuscripts submitted to *International Endodontic Journal* should include Title Page, Abstract, Main Text, References and Acknowledgements, Tables, Figures and Figure Legends as appropriate

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(i) Clinical Trials should be reported using the CONSORT guidelines available at www.consort-statement.org (http://www.consort-statement.org). A CONSORT checklist (http://www.consort-statement.org/mod_product/uploads/CONSORT%202001%20checklist.doc) and flow diagram (as a Figure) should also be included in the submission material.

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When experimental animals are used the methods section must clearly indicate that adequate measures were taken to minimize pain or discomfort. Experiments should be carried out in accordance with the Guidelines laid down by the National Institute of Health (NIH) in the USA regarding the care and use of animals for experimental procedures or with the European Communities Council Directive of 24 November 1986 (86/609/EEC) and in accordance with local laws and regulations.

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Discussion: may usefully start with a brief summary of the major findings, but repetition of parts of the abstract or of the results section should be avoided. The Discussion section should progress with a review of the methodology before discussing the results in light of previous work in the field. The Discussion should end with a brief conclusion and a comment on the potential clinical relevance of the findings. Statements and interpretation of the data should be appropriately supported by original references.

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Main Text of Review Articles should be divided into Introduction, Review and Conclusions. The Introduction section should be focused to place the subject matter in context and to justify the need for the review. The Review section should be divided into logical sub-sections in order to improve readability and enhance understanding. Search strategies must be described and the use of state-of-the-art evidence-based systematic approaches is expected. The use of tabulated and illustrative material is encouraged. The Conclusion section should reach clear conclusions and/or recommendations on the basis of the evidence presented.

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It is the policy of the Journal to encourage reference to the original papers rather than to literature reviews. Authors should therefore keep citations of reviews to the absolute minimum.

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Examples of correct forms of reference follow:

Standard journal article

Bergenholtz G, Nagaoka S, Jontell M (1991) Class II antigen-expressing cells in experimentally induced pulpitis. *International Endodontic Journal* **24**, 8-14.

Corporate author

British Endodontic Society (1983) Guidelines for root canal treatment. *International Endodontic Journal* **16**, 192-5.

Journal supplement

Frumin AM, Nussbaum J, Esposito M (1979) Functional asplenia: demonstration of splenic activity by bone marrow scan (Abstract). *Blood* **54** (Suppl. 1), 26a.

Books and other monographs

Personal author(s)

Gutmann J, Harrison JW (1991) *Surgical Endodontics*, 1st edn Boston, MA, USA: Blackwell Scientific Publications.

Chapter in a book

Wesselink P (1990) Conventional root-canal therapy III: root filling. In: Harty FJ, ed. *Endodontics in Clinical Practice*, 3rd edn; pp. 186-223. London, UK: Butterworth.

Published proceedings paper

DuPont B (1974) Bone marrow transplantation in severe combined immunodeficiency with an unrelated MLC compatible donor. In: White HJ, Smith R, eds. Proceedings of the Third Annual Meeting of the International Society for Experimental Rematology; pp. 44-46. Houston, TX, USA: International Society for Experimental Hematology.

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Ranofsky AL (1978) Surgical Operations in Short-Stay Hospitals: United States-1975. DHEW publication no. (PHS) 78-1785 (Vital and Health Statistics; Series 13; no. 34.) Hyattsville, MD, USA: National Centre for Health Statistics.8

Dissertation or thesis

Saunders EM (1988) In vitro and in vivo investigations into root-canal obturation using thermally softened gutta-percha techniques (PhD Thesis). Dundee, UK: University of Dundee.

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Tables: Tables should be double-spaced with no vertical rulings, with a single bold ruling beneath the column titles. Units of measurements must be included in the column title.

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7 Guidelines for reporting of DNA microarray data

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